Monitoring Ion Exchange Chromatography with Affordable Flame Emission Spectroscopy

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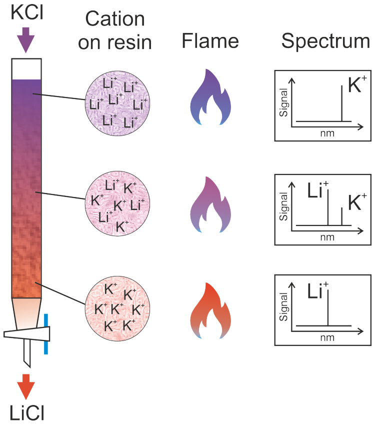
ABSTRACT

Highlighting the interdisciplinary nature of research, we present a series of experiments for undergraduate lab courses that teach the principles of preparative ion exchange chromatography and flame emission spectroscopy. Through these inquiry-based experiments, the students learn about experimental design and instrument limitations, naturally connecting general chemistry principles with analytical and physical chemistry and materials science. The goal is to devise experimental parameters that allow quantitative ion exchange on a small scale, and to monitor the exchange with optical spectroscopy. The small-footprint, inexpensive flame emission spectroscopy setup we developed can be used to accurately determine relative concentrations of alkali metal ions in a sample, while also being suitable for simpler experiments, which allow it to be employed in a variety of classes. In demonstration for energy research, we then perform the presented experiments on redox flow battery electrolytes to investigate the influence of the cation choice on battery performance.

GRAPHICAL ABSTRACT



KEYWORDS

First-Year Undergraduate/General; Second-Year Undergraduate; Upper-Division Undergraduate; Analytical Chemistry; Demonstrations; Inorganic Chemistry; Physical Chemistry; Hands-On Learning /

Manipulatives; Inquiry-Based / Discovery Learning; Aqueous Solution Chemistry; Atomic Spectroscopy; Chromatography; Gravimetric Analysis; Ion Exchange; Materials Science; Qualitative Analysis

INTRODUCTION

Laboratory experiments in chemistry curricula generally follow exactly defined protocols to discuss certain learning goals. This approach, however, rarely gives students an insight how research is conducted and does not foster competence with practices such as experimental design, formulation of scientific questions, and troubleshooting.[1,2] Among the numerous criticisms of traditional chemistry curricula is the lack of interdisciplinarity and segmentation along subdisciplinary lines of chemistry.[3] In this manuscript we present a series of experiments discussing preparative ion exchange chromatography (IEC) as well as flame emission spectroscopy (FES) to monitor the degree of ion exchange. The experiments connect general, analytical, and physical chemistry with materials science and do not follow a precisely determined procedure. This makes them well suited to be implemented in an inquiry-based laboratory or course-based undergraduate research experience that emulates authentic research in a teaching laboratory. Optimizing the experimental setup and devising ways to improve the ion exchange efficiency are excellent opportunities to increase student ownership of laboratory work and to highlight the interdisciplinary nature of research. Meanwhile, the presented FES setup is simple enough to be implemented in existing classes, for example during the first semester of general chemistry courses when atomic structure, atomic emissions, and flame colorations are covered.

IEC is a well-known technique for separation, purification, and desalination in fields such as water treatment, protein and biochemical analyses, pharmaceutical trace material analysis, and recently in energy research involving batteries and catalysis.[4–7] While IEC is generally taught in the frame of analytical chemistry courses, for example for purification of biochemical compounds or analysis of trace inorganic ions,[8–12] it is also a powerful synthetic method, an aspect that is frequently not considered in undergraduate education. With often time-consuming or complicated synthetic pathways, having the ability to easily exchange an ion, e.g., a cation, of a synthesized material allows researchers to study cation effects very efficiently, and preparative IEC is an excellent approach for such endeavors. Ion exchange resins are available in various forms, including macroporous or gel-type beads, and contain fixed ionic functional groups in the polymer structure that are neutralized with a counterion. The functional group can be anionic, commonly a sulfate group, for cation exchange, or cationic, typically a quaternary ammonium, for anion exchange.[13] The resin is packed in a column or tube and a sample solution is flowed through the resin-bed to replace cations in the sample with the cations present in the resin. The resin can then be regenerated for multiple uses, improving the cost-effectiveness of this method. In commercial applications, ion exchange columns generally have a narrow diameter, a pathlength greater than one meter to ensure a large concentration gradient, and slow flow rates to ensure sufficient time for interaction between the resin and the sample. These conditions are, however, difficult to replicate on a small scale, such as in a teaching or research laboratory, which often results in incomplete ion exchange. Devising experimental parameters that enable quantitative ion exchange on small scale is hence a great opportunity for students to optimize an experimental setup and parameters in an inquiry-based experience.

Optical emission spectroscopy (OES) is perfectly suited to assess the cation exchange efficiency (η_{CEX}), the percentage of a product containing the exchanged cation and not the initial cation. Typically, this method uses a heat source (a plasma for ICP-OES and MP-OES or simply a flame in FES) to excite atoms in the gas phase to a higher state of energy according to the Boltzmann equation.[14,15] As the atoms in the excited state relax to the ground state a characteristic light emission, often in the visible range, can be detected with a spectrometer. The intensity of a measured signal is proportional to the number of transitions from excited to lower energy states and is hence concentration dependent. The number of atoms that are excited is further dependent on the temperature of the excitation source and exactly controllable heat sources like high temperature plasmas are generally employed for quantitative analyses. Usually, OES is financially prohibitive for teaching labs as instrumentation can cost tens of thousands of USD. Simplified setups have been reported in the literature, but still required commercial air/acetylene burners,[16,17] intricate 3D printed nebulizers,[18] or were limited to the analysis of only one element.[19–21] We introduce an even simpler setup that is easily implemented in teaching or small-scale laboratories by removing the need for a nebulizer and pumps, and instead relying on rapid heating of liquid samples under direct exposure to a flame. The rapid evaporation of the liquid pseudo-nebulizes the sample into an open flame which is monitored with a fiber-coupled spectrometer. For the application presented here, deducing the ratio of cations in a sample rather than exact concentrations, the flame temperature does not need to be known since references and samples are measured under the same conditions. Furthermore, the spectrometer can easily be detached from the FES setup to be used in other experiments such as UV-Vis spectrophotometry to establish Beer's law or to observe spectra of different light sources.

Undergraduate students were tasked to optimize the FES setup and IEC parameters for quantitative ion exchange, and the experiments described here are designed to span several lab periods. Through these experiments, students are exposed to the concepts of IEC and how ion size, charge density, and resin affinity can affect the process, while also being introduced to FES as an analytical method, learning about principles of optical spectroscopy, emission lines, the Boltzmann equation, and excited states. The experimental design aspect further promotes critical thinking and independence and gives the students a real sense of achievement when quantitative ion exchange is, eventually, realized. Note that short FES experiments using this simple setup could also be implemented in first semester or high school classes when first discussing flame colorations of alkali metals. Select students continued into the next semester to explore the effect the choice of cation had in redox flow batteries and the results of these experiments are included in the Supporting Information to provide instructors with a real-world application of the presented methods in energy research.

EXPERIMENTAL

Chemicals

Chemicals were used as received from Alfa Aesar and Acros Organics unless otherwise noted. Chemicals required for ion exchange are LiCl, KCl, Amberlyst 15® H cation exchange resin, 1 M HCl solution, LiOH, and distilled water.

Hazards

Amberlyst cation exchange resin and HCl are corrosive. LiCl and KCl are mild skin and eye irritants. Properly fitting latex or nitrile gloves and goggles should be worn during experiments and laboratory coats are recommended to prevent accidental contact. Experiments, particularly FES, should be performed in a fume hood.

Ion Exchange Resin Functionalization

Amberlyst cation exchange resin is a strongly acidic sulfonic acid resin designed for cation exchange and has a capacity of 4.7 mmol of cation per gram of resin. The resin (10 g) was soaked in distilled water for ten minutes and added to a glass column (1 cm diameter, 30 cm length) and water was eluted through, maintaining a water level at or above the top of the resin, until the pH of the eluate was neutral by litmus paper. The extremely slow flow rates used in industry were not feasible with a glass stoppered column, but flow rates of 20-30 drops per minute were achieved. Resin was initially lithium-functionalized by eluting two equivalents of 1 M LiCl (10 mL, 10 mmol). The protons in the resin were replaced by lithium ions and were flushed out of the column, resulting in an acidic eluate. The column was then flushed with water (100 mL) until the pH was again neutral by litmus paper. Following this method, preparation of a lithium functionalized column ready to be used for ion exchange experiments takes approximately 40 minutes. A near saturated solution of potassium chloride was prepared and eluted through the column to perform the ion exchange. To ensure that all the active material was collected, two aliquots of water were also eluted and collected. This step only takes ca. 10 minutes.

Flame Emission Spectroscopy Setup

The FES setup was easily constructed with inexpensive materials including a butane torch, a clean metal spatula, a ring stand, some clamps, and utilized a glass fiber coupled UV-Vis spectrometer, which many university and college teaching laboratories already have. The setup is shown in **Figure 1**. A ring stand was assembled with an upper and lower clamp. The upper clamp held the spatula parallel to the bench surface, and the torch was fixed to the lower clamp so that the flame emitted would be directly under the edge of the spatula with a fixed distance between the torch and the sample, ensuring consistent heating of the samples between experiments. The glass fiber was connected to the spectrometer and set level with the spatula, 2 cm away. The spectrometer used was an Avantes AvaSpec-ULS2048L with a spectral resolution of 1.4 nm, wavelength range of 200-1100 nm, and a ILX511 2048-pixel detector. Samples were measured in scope mode with a 16 ms integration time, measuring from 220 to 900 nm. A blank sample was recorded by torching the spatula without sample material for background subtraction before each set of experiments.

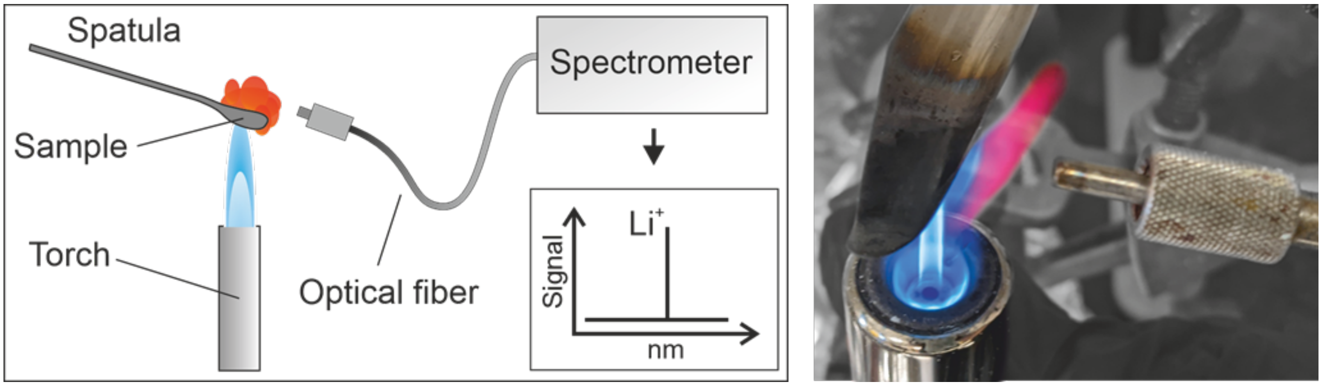


Figure 1. Schematic and photo of the setup used for FES experiments. The photo shows the characteristic carmine red color of a lithium-containing sample.

To record FES spectra, the following protocol proved most efficient by the students: The spectrometer is set to continuously record scans. Once one scan is almost complete, the torch is started and kept on for the subsequent scan, which allows enough time for students to manipulate both the computer and the torch. Once the second scan is complete, the flame is extinguished, and the spectrometer measurement is stopped. This sequence ensures that the sample is exposed to the flame for the duration of one whole scan, significantly improving reproducibility between experiments. The unoptimized setup can be installed within five minutes and recording data takes less than a minute per sample.

RESULTS AND DISCUSSION

Setup Utility

The FES setup was tested by undergraduate chemistry students to determine the ratio of Li:K in chloride salt samples. They were tasked to optimize the setup towards improved reproducibility by testing variables such as sample amount, flame strength, flame-sample distance, and sample-detector distance. Burning solid samples led to inconsistent results between experiments, so a small amount of sample was dissolved in water, and students pipetted a drop of the solution onto the spatula for vaporization. One drop consistently produced reliable results while larger volumes resulted in poor reproducibility, likely due to incomplete vaporization. Following this approach, the students can experiment with the FES setup and explore how the peak ratios of LiCl/KCl samples are affected by sample composition and experimental parameters such as the sample-torch distance, the angle at which the flame hits the spatula, and the distance between the spatula and spectrometer. This setup is capable of obtaining signals for all alkali cations consistent with literature, a sodium singlet at 589 nm, a lithium singlet at 670 nm, a potassium doublet at 766/769 nm, a rubidium doublet at 779/794 nm, and a caesium doublet at 852/894 nm (see **Figure 2**).[22] Note that increasing the flame strength increased the intensity of the signals but decreased the resolution of the potassium doublet, likely due to increased self-absorption (see **Figure S1**).

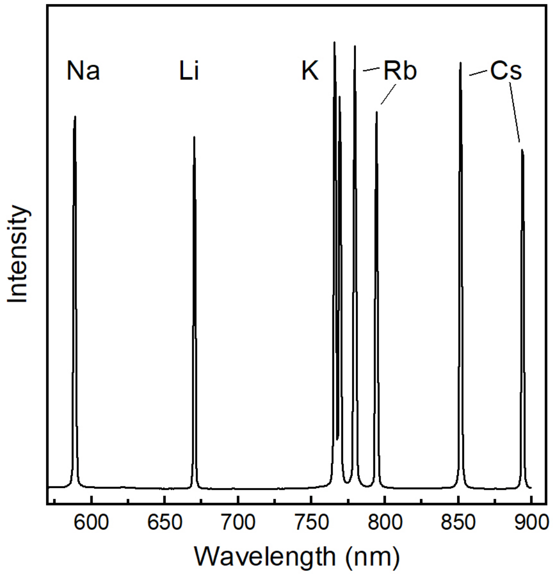


Figure 2. Flame emission spectrum of a solution containing alkali chloride salts.

Sample Referencing

The students were then encouraged to think about how to prepare a reference curve for assessing η_{CEX} in the samples prepared via IEC using reference samples with KCl and LiCl in various ratios. While weighing samples to prepare reference solutions with well-defined Li:K ratios is the easiest approach, weighing small amounts increases the uncertainty of the measurement, while preparing large amounts of reference mixtures to compensate for weighing errors is wasteful, as only ~50 µL of solution is needed per FES experiment. Students thus prepared solutions with cation ratios ranging from 999:1 Li:K to 1:999 Li:K from standard (1 M) solutions and performed FES (**Table S1 and Figure 3a**). The peak intensity ratios were then calculated for each sample to prepare a reference curve relating peak ratios to cation ratios (**Figure 3b**). This curve was then used to assess the η_{CEX} in materials prepared via IEC. To monitor reproducibility of results, each reference sample was measured twice in triplicate, on multiple days, and by different students. Error bars in the figure represent standard deviations for experiments with n=6. Depending on the number of students and how many FES setups are available, preparing such a reference curve will likely occupy an entire lab period.

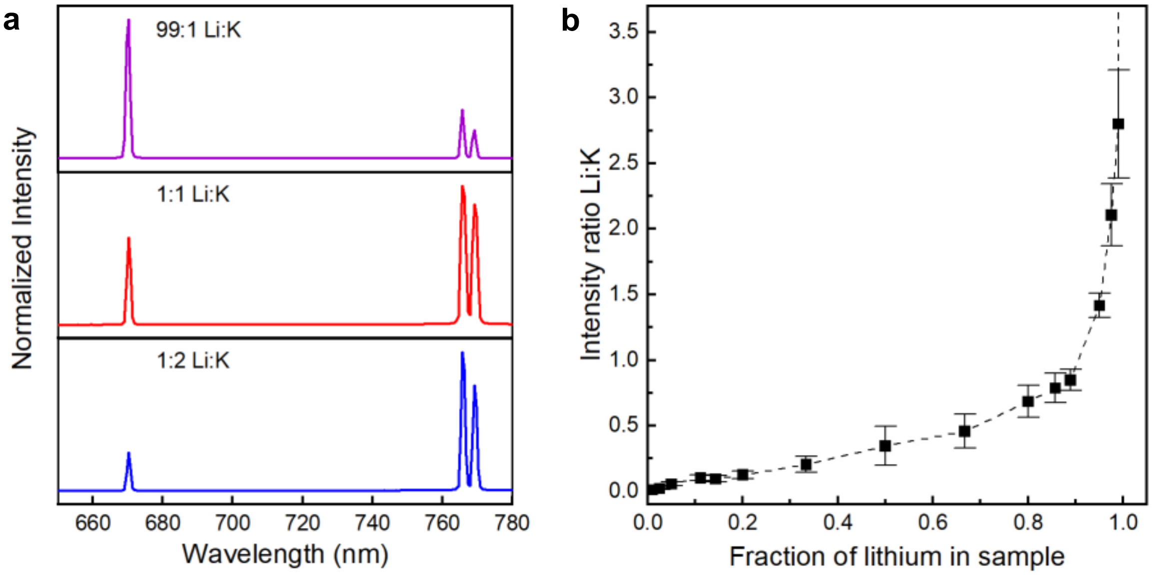


Figure 3. (a) Representative flame emission spectra of three different reference samples with Li:K ratios of 99:1, 1:1, and 1:2, respectively. (b) Reference curve relating peak ratios to cation ratios for samples with Li:K ratios of 999:1 to 1:999.

Ineffective Ion Exchange through Functionalized Resin

Preparative IEC was first performed by eluting KCl solutions through the lithium-functionalized resin utilizing 100% resin capacity and η_{CEX} was determined via gravimetric and FES analysis. The experimental product mass after drying exceeded the expected yield, indicating it still contained KCl, therefore suggesting a low η_{CEX}. This was confirmed by the presence of both lithium and potassium signals in FES. This ineffectiveness was surprising to most students, and they were noticeably self-motivated to come up with conditions to make the experiment more efficient using the available equipment. To explore the impact of flow rate on η_{CEX}, the students eluted samples at various flow rates; ca. two drops per second (fast), ca. one drop per second (medium), or one drop every 2-3 seconds (slow). Representative FES data for this experiment is shown in **Figure S2**. The slower flow Rate resulted in better ion exchange, η_{CEX} of 89%, compared to the medium (81%) and fast (68%) trials. Flow rates were subsequently minimized, and students experimented with using less resin capacity to increase n_{CEX}. In IEC trials utilizing less resin capacity, the gravimetric analysis suggested that a quantitative conversion from KCl to LiCl occurred when a 60% or lower resin capacity fraction was utilized (see **Table S2**). However, when the ratio of Li:K was measured by FES, the percentage of lithium in the product only increased slightly from 89% at full resin capacity to 92% at half resin capacity. These results show that gravimetric analysis is insufficient on its own and that, in contrast to industry applications, the cation exchange resin was unable to quantitatively exchange a smaller cation for a larger cation with a lab-scale column setup.

Resin Regeneration

Each ion exchange trial utilizes 10 g of resin, which would lead to a large amount of waste in a teaching lab without reusing the resin. Two methods were explored to regenerate used resin, either by following the initial functionalization procedure, eluting LiCl through the used resin, or eluting HCl to first regenerate the resin to its acidic form and then eluting LiCl. To compare the efficiency of the refunctionalization, ion exchange trials were performed using collected eluates repeatedly until a Li:K ratio corresponding to >99% Li was achieved. As shown in **Figure 4**, the first trial resulted in a Li-content of ca. 89%. By initially restoring the resin to the acid form, conversion to >99% is achieved with only one additional elution. Following the initial functionalization procedure of just eluting a LiCl solution required four additional ion exchange trials to achieve a comparable result. This observation was counterintuitive to most of the students because protons have a lower resin affinity than lithium cations but were more efficient at replacing potassium cations in used resins. This enabled a discussion about charge density, ion mobility, and concentrations gradients: While the affinity of protons to the resin is in fact lower than for alkali cations, displacement is more efficient by flushing with HCl due to the high mobility of protons, the steep concentration gradient, and large proton excess economically achievable when eluting with HCl. Achieving the same concentration and gradient with LiCl would be excessively wasteful and cost prohibitive.

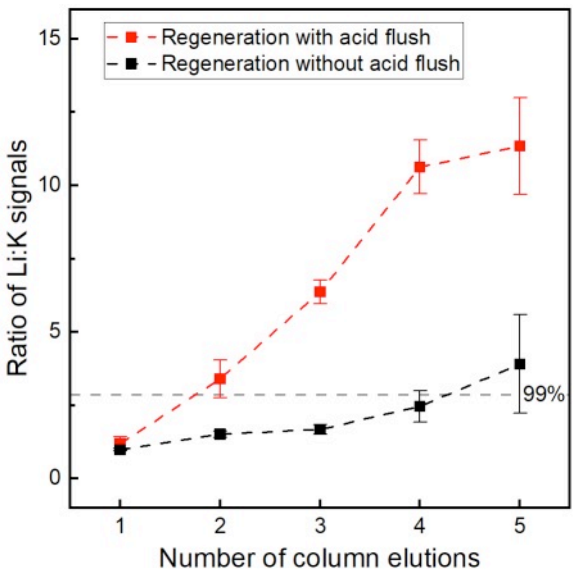


Figure 4. Resin regeneration following two methods, either by directly eluting LiCl through used resin, or by first flushing HCl through the column, followed by eluting LiCl. The eluate in each case was collected to perform FES, the resin was refunctionalized, and the eluate was then again eluted through the freshly refunctionalized column. By flushing HCl through the column prior to LiCl functionalization, a lithium content of >99% is achieved after two trials, while the direct refunctionalization with LiCl required five trials to yield a comparable result.

Direct Ion Exchange Method

Since functionalization of the resin with LiCl resulted in low η_{CEX} even when using half the capacity of the resin, a direct method for exchanging cations was explored to circumvent the necessity of multiple elutions. By eluting KCl solutions through ion exchange resin in its acidic form into a solution containing an equimolar amount of LiOH, the potassium ion was exchanged with a proton in the column, temporarily forming HCl, then underwent an acid-base reaction to form LiCl in the LiOH solution. This method excluded the possibility of inefficient resin functionalization with lithium cations and enabled cation exchange efficiencies >99.9% with one elution using resin that was previously regenerated to the acidic form (**Figure S3**). After many unsuccessful elutions with functionalized resins, achieving quantitative ion exchange by implementing this direct method sparked a real sense of achievement with the students. In post-lab discussions it was pointed out that, while the method of FES was “cool” enough on its own, successful ion exchange was highly motivating and that without achieving it the IEC experiments would have felt “irrelevant” and “pointless”.

Ion Exchange in Energy Research

Electrochemistry presents an opportunity to elegantly connect preparative IEC and FES with real-world applications, e.g., rechargeable batteries, as the nature of the cation in battery electrolytes directly affects physicochemical properties such as conductivity, viscosity, and density.[23] In our experience, battery research is a particularly exciting topic to explore with undergraduate students since media coverage on battery breakthroughs and storage of renewable energy is currently ubiquitous.[24,25] In the Supporting Information we discuss results obtained from a series of experiments where select students explored the impact of cation choice on the performance of a redox flow battery. This type of battery was selected because ion exchange membranes, a crucial component of flow batteries, follow the same operating principles as ion exchange resins used in IEC. Tasked to characterize the cation-dependent performance of such a battery, the students combined IEC and FES with electrochemistry and were particularly excited to use their materials in a research-setting and to observe the impact of something as small as the cation choice and η_{CEX} when exchanging cations, on flow battery operation.

Implementation in a Teaching Lab

The experiments described here are modular so that instructors can design a laboratory experiment based on time allotments. For simple demonstrations of flame colorations, the FES setup can be prepared within five minutes. For a full lab period experiment teaching the principles of IEC and FES, instructors can provide students with a well working FES setup and pre-recorded reference curves to then explore the difference in η_{CEX} between a lithium functionalized column and the direct method using the resin in its acidic form. If an instructor wishes to expand on these basic experiments, optimization of the FES setup and recording reference curves approximately occupies one lab period each, while optimization of IEC parameters such as flowrates and resin capacity ratios easily requires one to two lab periods. This can further be expanded upon by implementing additional cations. Initially not achieving quantitative ion exchange naturally leads to discussions about the correlation between cation size and resin affinity, and, for more advanced students, experiments can also cover the concepts of spin-orbit coupling and the Zeeman effect by including a discussion on why the increase in energy difference between the spin-orbit coupled excited states increases doublet peak separation with increasing cation size (**Figure 2**).[26] The entire series of experiments, from first IEC and FES trials to fully optimized parameters and quantitative exchange of ions, easily fulfills the requirements of a course-based undergraduate research experience. Furthermore, there is an opportunity for a semester long thesis project when combined with applied research, such as synthesis or battery experiments.

CONCLUSION

We devised a series of experiments to introduce undergraduate students to the principles of preparative ion exchange chromatography and designed a simple, cost-effective method to characterize reaction products via flame emission spectroscopy. Monitoring the ion exchange efficiency via optical spectroscopy provides an excellent platform to discuss the primary learning objectives of understanding optical spectroscopy and ion exchange, while also teaching students about calibration curves, acid-base reactions, and the Boltzmann equation. Following the inquiry-based approach, the laboratory additionally teaches aspects of experimental design and troubleshooting. The experiments are easily implemented in current undergraduate chemistry curricula since most of the materials needed for the presented experiments are typically already available in general chemistry teaching laboratories and can be utilized for multiple experiments, lab courses, and analytical methods. Importantly, students are exposed not only to preparation and characterization of materials but also aspects of instrumental development, teaching them about the multidisciplinary nature of research. Finally, we demonstrate the utility of the described methods in energy research, an exciting field to many junior researchers.

ASSOCIATED CONTENT

Supporting Information

Additional experimental details on FES optimization and preparative IEC experimentation, comparison of gravimetric and FES analysis, and discussion of flow battery experiments, as well as information and materials given to students prior to any experimentation. The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.XXXXXXX. [ACS will fill this in.]

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