JOURNAL OF CHEMICAL EDUCATION
Article
Cite This: *J. Chem. Educ.* 2018, 95, 1717−1723
pubs.acs.org/jchemeduc

Designing and Teaching a Course about Characterization Techniques for Solid State Materials in an Undergraduate Institution

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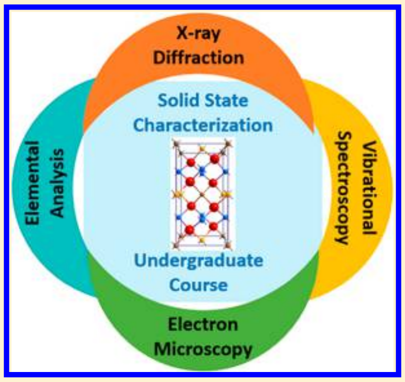
S *Supporting Information*

ABSTRACT: The experience of designing and teaching a lecture-based course about characterization techniques commonly used in solid state chemistry and materials science in an undergraduate institution is described. Among the techniques studied were the following: powder X-ray diffraction, infrared spectroscopy, Raman spectroscopy, scanning electron microscopy, transmission electron microscopy, and techniques for elemental analysis. For each technique, the students were introduced to the physical principles underlying the technique, studied real examples present in the current scientific literature, and discussed how the technique could be applied to other experimental situations involving inorganic, organic, and biological materials.



KEYWORDS: *Upper-Division Undergraduate, Curriculum, Testing/Assessment, Instrumental Methods, Materials Science, Solid State Chemistry*

INTRODUCTION

Solid state chemistry and materials science are two terms frequently used in the scientific literature comprising a broad range of materials, such as metallic materials and alloys, inorganic ceramics, carbon-based materials like fullerenes and graphene, polymers, composites, and biological materials. In view of the importance and interdisciplinarity of solid state chemistry there are many opportunities to approach this topic in an undergraduate course in chemistry.[1] However, these topics are usually briefly studied in general and inorganic chemistry courses in subjects like structure and bonding, and their relationship with properties.[2]

Another important field in solid state chemistry is characterization techniques. In general, in an undergraduate chemistry curriculum, characterization techniques are studied in the context of inorganic and organic chemistry experimental courses and instrumental analysis courses. Although these courses provide a reasonable depth on techniques like UV−vis absorption spectroscopy, infrared spectroscopy, chromatographic techniques, nuclear magnetic resonance spectroscopy (NMR), and mass spectrometry, the application of these techniques is aimed to the molecular level context, such as spectra interpretation and determination of structure of organic molecules.[3−5]

A survey carried out in May 2018, by consulting the detailed course descriptions present in the academic catalogue of 20 graduate school programs in chemistry, in the USA, offering a PhD degree in chemistry with concentration in materials chemistry, revealed that the terms X-ray diffraction, Raman spectroscopy, electron microscopy, nanoscience or nanotechnology, and solid state chemistry are, respectively, present in the course descriptions of 90%, 70%, 75%, 85%, and 80% of the institutions, as shown in Figure 1a. In contrast, this survey was carried out consulting the course descriptions present in the academic catalog of 173 primarily undergraduate institutions, spanned through 45 of the contiguous states in the United States of America, and revealed that the percentage of appearance of these same terms is much lower, for instance, 12.7% for X-ray diffraction, 12.1% for Raman spectroscopy, 11.6% for electron microscopy, 19.7% for nanoscience or nanotechnology, and 40.5% for solid state chemistry, as shown in Figure 1b. More details about the data collection process and higher education institutions consulted to plot the graphs shown in Figure 1 can be found in the Supporting Information.

The discrepancy between the numbers observed for the surveyed terms in graduate school and primarily undergraduate institution setups is worrying and indicates that students in primarily undergraduate institutions may still lack some exposure to many techniques that are commonly applied in materials science and solid state chemistry contexts. For instance, techniques such as powder X-ray diffraction, Raman spectroscopy, and electron microscopies are techniques apparently not properly emphasized or sometimes not even mentioned during undergraduate chemistry courses.

The inclusion of these techniques in some part of the undergraduate chemistry curriculum may represent a signifi-

Received: March 22, 2018
Revised: July 21, 2018
Published: August 9, 2018

DOI: 10.1021/acs.jchemed.8b00207

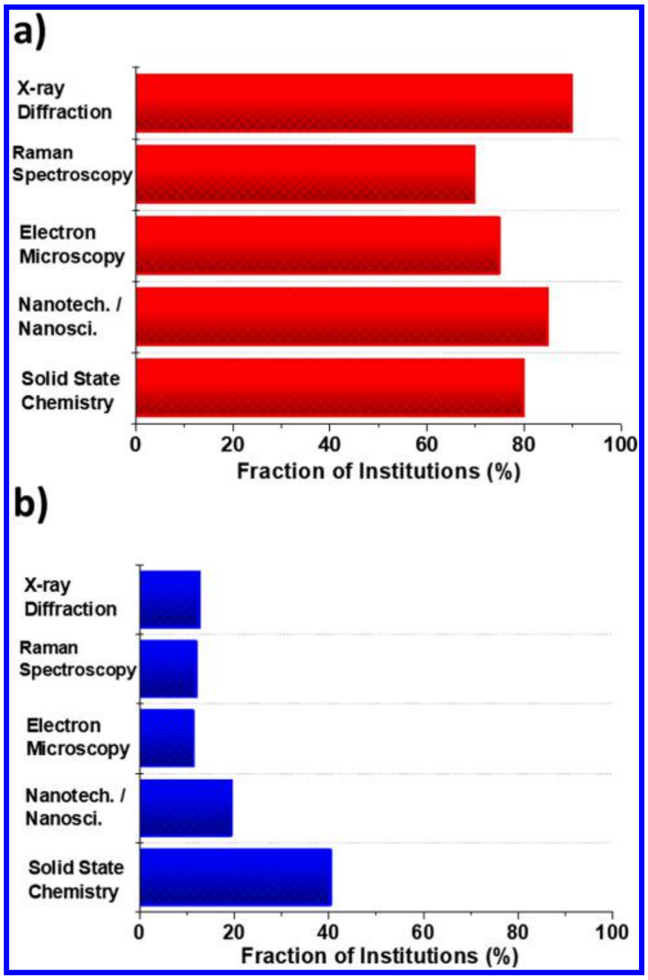


Figure 1. Survey about the appearance of the terms X-ray diffraction, Raman spectroscopy, electron microscopy, nanoscience or nanotechnology, and solid state chemistry: (a) percentage of graduate schools institutions in chemistry with concentration in materials chemistry, in the USA, showing these terms in course descriptions; (b) percentage of primarily undergraduate institutions offering major in chemistry, in the USA, showing these terms in course descriptions.

cant advantage to the students aiming to further their careers in graduate schools in chemistry, chemical engineering, materials science, and professional and allied health schools, and in industrial fields as well. The *Journal of Chemical Education* has published in the past 20 years some interesting experiments using powder X-ray diffraction, for instance, for monitoring the titration of solid acids,[6] and analysis of common household solids;[7] Raman spectroscopy, for instance, for detecting art forgeries;[8] and electron microscopy, for instance, for analysis of archeological materials.[9]

All of those experiments are worthwhile and have contributed for the advancement of solid state characterization techniques in undergraduate teaching. However, generally, they are isolated experiments inserted in other existing experimental courses, which may not give the undergraduate students enough time to understand the underlying physical principles of each technique and correlate them with features of the sample, or to make students able to understand a simple, actual data set present in actual scientific papers.

Due to the importance of the study of characterization techniques, this paper describes the experience of designing and teaching a half-semester, advanced elective undergraduate lecture course at Ithaca College studying techniques commonly applied in solid state chemistry, materials characterization, and nanotechnology, such as powder X-ray diffraction, Raman spectroscopy, scanning and transmission electron microscopies, and techniques for elemental analysis. By gathering all these techniques in a dedicated course, it was possible to offer the students the proper time and knowledge necessary to introduce them to the techniques in a way they could understand more in-depth the physical principle of each technique, how that correlates with the sample features, and how that could be applied for the analysis of different classes of materials.

Finally, it is important to point out that although there are papers describing the implementation of solid state materials characterization courses for upper undergraduate and graduate students in research universities, for example, the course implemented by Izutani and co-workers at Waseda University, in Japan,[10] the experience of implementing a course on these topics is completely different in primarily undergraduate institutions, which usually do not have all the facilities or pieces of equipment necessary, requiring from the instructor creativity to design the classes in a comprehensive way.

■ COURSE DESIGN AND IMPLEMENTATION

Institution Background

Ithaca College is a primarily undergraduate institution located in Ithaca, in the central area of New York state, founded in 1892. Ithaca College has an approximate total enrollment of 6200 undergraduate students divided in different areas such as humanities, natural sciences, business, music, communication, health sciences, and human performance. The college also enrolls around 460 graduate students, in different graduate majors like business, education, music, sport sciences, and physical therapy. Among the natural sciences, Ithaca College offers undergraduate majors in chemistry, biochemistry, biology, physics, and environmental sciences.[11]

The department of chemistry offers an ACS accredited Bachelor of Science (BS) major, a Bachelor of Art (BA) major, and a minor in Chemistry. The BS major in chemistry requires the completion of 6 credits of advanced elective courses; every semester, two or more 1.5 credit elective half-semester courses are offered. As the characterization and physical properties of meso- and nanoscale materials are listed as one of the content requirement areas in the American Chemical Society (ACS) Guidelines and Evaluation Procedures for Bachelor's Degree Program,[12] to fulfill the need for insertion of the characterization techniques for solid state chemistry topic into the chemistry curriculum, an elective course called "Modern Techniques for Solid State Materials Characterization" was created. The course had six junior/senior students from chemistry and biochemistry majors enrolled in its first offering.

Course Structure and Topics Studied

The course was taught in 75 min classes twice per week for 7 weeks, the techniques studied and the sequence in which they were studied are presented in Table 1.

The introduction to solid state chemistry and materials science unit had the goal of familiarizing the students with the potential of each technique to be studied in the course. Each technique was correlated with materials features, for instance, particle size, shape, composition, and crystalline structure, which could be commonly assessed by each technique, as shown on Figure 2.

Table 1. Topics Addressed and Techniques Studied by Week of the Course

Week	Topics and Techniques Studied, and Tasks for Students
1	Introduction to solid state chemistry and materials science
2	Powder X-ray diffraction (XRD)
3	Vibrational spectroscopy (infrared (IR) spectroscopy and Raman spectroscopy)
4	Scanning electron microscopy (SEM)
5	Transmission electron microscopy (TEM)
6	Elemental analysis (energy-dispersive X-ray spectroscopy (EDS), electron energy loss spectroscopy (EELS), X-ray fluorescence (XRF), inductively coupled plasma optical emission spectroscopy (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS))
7	Independent study and final paper first draft due

Furthermore, this unit covered topics like relationship between microstructure features and materials properties; distinction among amorphous, single crystalline and polycrystalline materials; unit cells, crystalline systems and Bravais lattices; crystallographic directions, planes, and Miller indices; and calculation of number of lattice points, lattice parameters, and density for cubic unit cells. Another learning goal of this unit was to provide to the students resources to distinguish an amorphous from a crystalline material. Then, after the comprehension of their differences, many solid state chemistry concepts could be derived for the crystalline materials. For example, the concept of unit cell and how the type and dimensions of the unit cell can lead to different material properties, for instance, the density.

Starting the study of the characterization techniques, the students were introduced to the pictorial concept that every technique in the course would be studied on the basis of the four main steps. They are source of stimulus, interaction with the sample, physical phenomena observed, and data collection and processing, as shown in Figure 3.

For powder X-ray diffraction (XRD) as an example, the source of stimulus for the technique is the X-rays, which in interaction with the sample could be either constructively or destructively diffracted (interaction with the sample and physical phenomenon), and then the constructively diffracted X-ray beams would be translated as peaks in the XRD pattern. Also, the position of the peaks would be dependent on the type and dimensions of the unit cell of the material.

Although the study of the techniques is structured around the main points shown in Figure 3, the order of the content presentation did not need to follow this sequence. For instance, data processing was the first topic presented during the XRD unit, which started with the distinction between amorphous and crystalline materials and how it is translated in a graph showing a powder X-ray diffraction pattern. At this point, it was important to show the students that a certain material can either be amorphous or crystalline, depending on the conditions to which this material was exposed during its synthesis and processing. The example shown in class was silicon dioxide (SiO_2), which can be amorphous, as commonly observed in glass form, or crystalline when processed at temperatures as high as 1400 °C.[13] Next, the instructor presented some equations that would be necessary to understand how the peak position depends on the type of the unit cell, for example, Bragg's Law. After studying the factors affecting peak position, the factors affecting peak intensity were presented, for instance, atomic scattering factor and its relationship with the atomic number of the diffracting atom present in the crystalline structure. The concept of

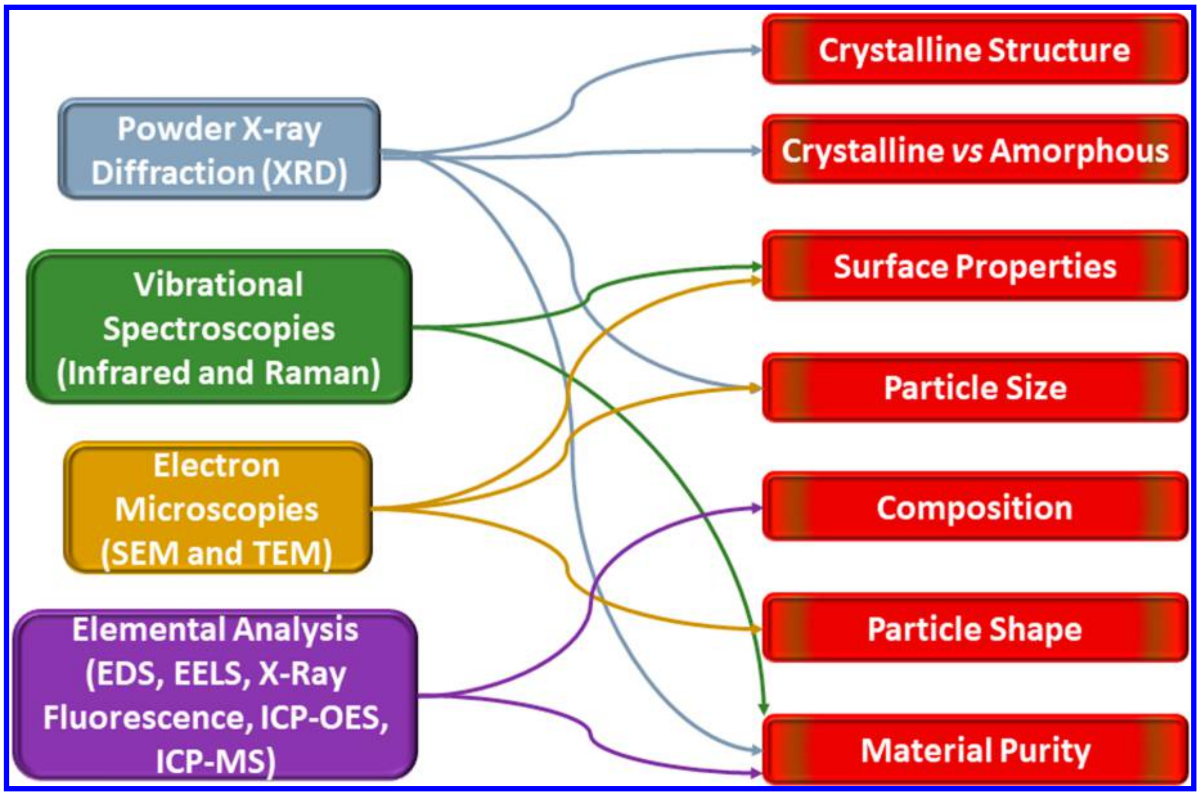


Figure 2. Scheme showing the correlation between different techniques studied in the course and the type of materials information able to be assessed by each of these techniques.

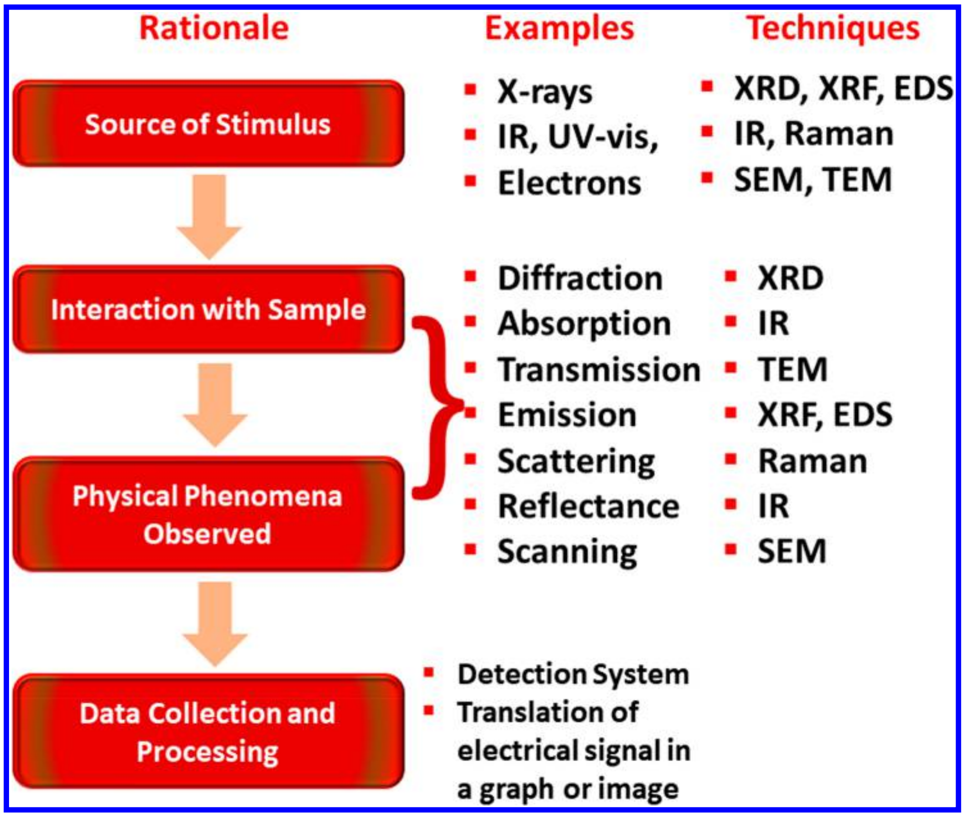


Figure 3. Schematic representation of the class rationale for the techniques studied in the course. The bullet points provide examples for each of the steps involved in the presentation of each technique.

systematic absences for cubic unit cells was presented as well. Finishing the conceptual part of the XRD unit, the process of X-ray generation based on the Rutherford−Bohr atomic model was explained. Regarding the application part of the XRD unit, a paper about the quantification of the polymorphs of the pharmaceutical drug olanzapine was presented showing the importance of the XRD for the pharmaceutical field.[14]

Students were asked to apply the knowledge acquired during this unit by learning how to set up a table in Microsoft Excel to calculate the lattice parameter of a metallic sample with the face-centered cubic (fcc) structure from the data collected by the instructor from an actual aluminum sample.

As the first set of spectroscopic techniques, the vibrational spectroscopy unit introduced the concepts of energy quantization and how different the energy scales involved in the rotational, vibrational, and electronic transitions are. Students had the foundation to understand how vibrational spectroscopy can be explained by the classic harmonic oscillator model. During the infrared (IR) spectroscopy part, the capabilities and differences among the common modes of data collection were explained, such as transmittance, absorbance, diffuse reflectance, and attenuated total reflectance. The differences between the dispersive and Fourier transform IR spectrometers were also studied during the course. The Raman spectroscopy part presented the concepts of Rayleigh, Stokes, and anti-Stokes scatterings, followed by the selection rules comparison between IR and Raman, and how these selection rules differences can be used to obtain complementary information for the same sample by using these two techniques. In the application part of the session, students could see examples from papers related to determination of functional groups attached to the nanoparticle surface,[15] detection of reaction intermediates,[16] isotopic labeling,[16] orientation of molecules adsorbed in surfaces,[17] estimation of the nylon polymer chain size,[18] and determination of amorphous material in zinc selenide nanoparticles[19] using Raman spectroscopy and surface-enhanced Raman spectroscopy (SERS) for identification of dyes in hair.[20]

The scanning electron microscopy (SEM) unit started comparing the capabilities of the visible light microscope and the electron microscopes, giving the students the idea that the possibility to use an electron microscope would enable the researcher to obtain higher magnification than what is possible with a visible light microscope. The next topic followed introducing the different types of electron sources, and different types of phenomena that can happen when electrons interact with matter, for instance, production of secondary electrons and backscattered electrons, which are the two types of electrons responsible for contrast generation in SEM. Also, this topic introduced how the different types of contrast can be used to produce complementary information for the same sample.[21]

The classes about transmission electron microscopy (TEM) presented the system of lenses and apertures present in a transmission electron microscope and the role of those components in image formation. These classes also provided resources to differentiate a SEM from a TEM image by observing differences in the contrast of the image. Additionally, the classes presented other capabilities that are possible to carry out with a transmission electron microscope, such as dark-field images, and selected area electron diffraction. Finally, the students had a demonstration showing how to set up scale bars in TEM images and perform particle size counting using the free software ImageJ.[22]

The last instructional week was dedicated to a variety of techniques suited for compositional analysis such as energy-dispersive X-ray spectroscopy (EDS), electron energy loss spectroscopy (EELS), X-ray fluorescence (XRF), inductively coupled plasma optical emission spectroscopy (ICP-OES), and inductively coupled plasma mass spectrometry (ICP-MS). Due to the large number of techniques to be covered in two 75 min class sessions, this unit was more focused on presenting to the students the differences in the physical principles, sampling, range of elements able to be detected, and limit of detection among all these techniques.[23]

Course Resources and Assignments

Although the Ithaca College instrumental facility is well equipped with pieces of equipment such as single crystal and powder X-ray diffractometers and UV−vis, NMR, and infrared spectrometers, many instruments studied during the course were not available in the College. For instance, electron microscopes are instruments very unlikely to be present in undergraduate institutions. So, in order to make students familiar with those instruments, a strategy used during this course was to use short Youtube videos to present the instruments unavailable in the College to the students; from those, students could have some ideas about how the actual equipment appears, where the pieces are located, and where the sample should be inserted. A list of the videos shown in the course with their respective links can be found in the Supporting Information, followed by the list of the books used in the classes preparation.

Regarding the assignments, students were asked to complete a homework assignment every week. The problems presented in the assignments described real situations that students might be able to find while doing research in the academic or industrial field. As an example, the homework assignment for the XRD unit is presented in the Supporting Information. Besides the regular homework, students were asked to complete a final assignment. For that, the instructor provided a data set taken from an actual scientific paper alongside some background information, for instance, the experimental procedure used to prepare the material. The actual scientific paper where the data set was taken from was not disclosed to the students. Each student received a different data set containing data using two or three techniques studied during the course. The students had to write a scientific paper based on the data set and pieces of information provided by the instructor. The scientific paper had to contain the sections Abstract, Introduction, Experimental Procedure, Results and Discussions, Conclusions, and References, and it was required to be typed into the ACS *Chemistry of Materials* journal template. The final draft submitted was evaluated by the instructor as accepted as is, major revision, or minor revision, and returned to the students with the comments about what was necessary to be changed in order to have the paper accepted and to receive the highest grade possible for the final assignment. Some examples of the paper titles presented by the students with the techniques studied on the paper are shown in the Table 2.

Table 2. Example Student-Written Paper Titles with Techniques Studied and Relevant Literature Sources

Paper Title	Techniques Studied	Data Source (Reference)
One Step Synthesis of Highly Magnetic Iron Oxide Magnetite (Fe_3O_4) Nanorods and Nanoparticles	XRD and TEM	24
Fabrication of a portable sensor based on SERS effect for detection of the explosive trinitrotoluene (TNT)	IR and SERS	25
X-ray Diffraction and Raman Spectroscopy Characterization of the Existing Phases of Y_2O_3−Nb_2O_5	XRD and Raman Spectroscopy	26
Synthesis and Stability of $CsPbI_3$ Quantum Dots	XRD and TEM	27
Characterization of different chronic and acute gallstones by FT-IR, SEM, and EDX	IR, SEM, and EDS	28
Control of crystalline phase and size of Gd^{3+}-doped $NaYF_4$:Yb/Er nanocrystals	XRD and TEM	29

After the feedback in the first draft, all the papers needed either minor or major revisions, and all the students chose to make the necessary changes appointed by the instructor serving as reviewer. This simulated peer-review process was a positive experience for the students due to many reasons. The first one is the fact that students could read actual scientific peer-reviewed papers to help to interpret the data present in their assigned data set, students also had to look for new references to write the Introduction and Results and Discussions sections of their own papers. The second is that students had the opportunity to work with journal templates and citation manager softwares to have their paper and references suitably formatted. Finally, the simulated peer-review process led the students to think more deeply about their results interpretation and paper formatting, giving them the chance to improve their writing and result analysis skills. All these skills practiced in the final assignment are common tasks for graduate students and researchers in any capacity, so, to have students exposed to this type of task might make them better prepared for their careers after graduation, either in graduate school or in an industrial job.

Evaluation of the Course by the Students

The answers received in the anonymous Course Evaluation indicate that the course was positively evaluated by the students. For instance, 100% of the respondents agreed or strongly agreed with the following statement: “This course provided me information that otherwise I would not see in any course of the Chemistry/Biochemistry curriculum”. This confirms that the course attained the goal to provide the students exposure to techniques that are not commonly presented in a chemistry or biochemistry course in a primarily undergraduate institution. About the applicability of the course in student’s career after graduation, 67% of the respondents strongly agreed with the statement “This Course provided me information that will be useful to my academic or professional career after graduation”. Finally, regarding the course recommendation to other students, 100% of the respondents agreed or strongly agreed with the statement: “I recommend other students to take this course”.

■ CONCLUSIONS

The course was, in general, positively evaluated by the students, providing to them information that they probably would not receive in typical chemistry curriculum courses. Some students described that this course made them want to learn more about characterization techniques in graduate school. The homework assignments also received positive comments, being classified by some students as containing many useful applications of the techniques. Regarding the possibility to expand the course for a full semester, 100% of the students who responded the course survey indicated that they would prefer to study the same number of techniques, but more in-depth, instead of seeing a larger number of techniques in a more introductory level.

Although the course had a small number of students registered in its first offering, there are enough reasons to consider that this course was successfully implemented, and even have the possibility to be taught in other primarily undergraduate institutions.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.8b00207.

> Course syllabus, list of Youtube videos used during the course, list of books used for class preparation, sample homework assignment, and details about the survey of materials chemistry related terms (PDF, DOCX)

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Notes

The author declares no competing financial interest.

■ ACKNOWLEDGMENTS

The author would like to thank Prof. Janet Hunting (Ithaca College, Department of Chemistry) for the feedback and suggestions throughout the preparation process of this manuscript. The author also thanks Ithaca College Office of the Provost and Center of Faculty Excellence for financial support. This paper is dedicated to Anthony Barbosa Pinto, on the occasion of his first birthday.

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