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Stimulating Students’ Learning in Analytical Chemistry through an Environmental-Based CURE Project

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ABSTRACT

This article reports on the implementation of a CURE project in upper-level analytical chemistry course to engage a larger number of students in research at a primarily undergraduate institution (PUI). The lab course syllabus was modified to provide students with an opportunity to experience the process of conducting research through a project that was relevant to them personally and the community at large. Students carried out research using microwave plasma atomic emission spectroscopy (MPAES) to correlate lead levels in soil with home location, source, and age. Students’ self-assessment and surveys suggest that implementing a research project that students can relate to, leads to an increased level of mastering analytical skills and demonstration of a higher level of critical-thinking skills. Participation in the CURE project increased a desire and a commitment to engage in independent research and in communicating science through presentations.

KEYWORDS: Upper-level undergraduate, analytical chemistry, environmental, undergraduate research
INTRODUCTION

Research is an important element in an undergraduate curriculum, providing benefits to students, faculty, and the degree program.\textsuperscript{1-8} Students get an opportunity to gain a deeper knowledge of research techniques and processes, and then apply classroom learning in real-world contexts. However, in many undergraduate institutions, only a few students are able to reap the benefits of engaging in undergraduate research due to limited resources and lack of infrastructure to support dynamic and productive research programs.\textsuperscript{8,9} Students personal obligations may also limit availability to engage in research. For example, at Indiana University South Bend (IU South Bend) 37\% of the students enrolled come from first-generation college backgrounds. In addition, the majority of the students commute to school and work part-time jobs to support themselves while others support their families. Besides limited resources and infrastructure, the awareness of research opportunities and benefits of undergraduate research often comes later in a college student’s career, and may be too late for graduating students. The majority of the students go through the undergraduate curriculum without having an opportunity to engage in research. Course-based undergraduate research experiences (CUREs) is a good solution to ensure that undergraduate students graduate with some research experience. CUREs has the potential to reach a large number of undergraduate students and to support and retain undergraduates.\textsuperscript{9-13} In a CURE project, students apply scientific practices to investigate collaborative research topics that are of interest to the broader scientific community with outcomes that are unknown to the students and the instructor(s) alike.\textsuperscript{14-17}

The goals of implementing CURE projects in upper-level science courses are different from other CURE-based laboratories targeting introductory courses.\textsuperscript{10,18-25} Students taking upper-level science courses have already demonstrated their interest and persistence in science.
To this end, the objective was to engage a larger number of students with no prior research experience to help them to understand the research process and develop the ability to integrate theory to a real-life application.

**CURE-Incorporated Lab Development**

At IU South Bend, analytical chemistry is composed of two courses, Analytical Chemistry that is quantitative-based (CHEM 310), and Principles of Chemical Instrumentation (CHEM 410). The courses are taught alternating fall semesters, and are required for chemistry majors and serve as electives for biochemistry majors. The enrollment numbers for both usually range from 9 – 14. The CURE project was introduced in Fall 2019 in CHEM 410. The class consists of two 75 minutes lectures, a one-hour discussion session, a one-hour lab lecture, and a 4-hr lab session per week. The labs in both courses emphasize writing in sciences with lab reports taking the form of a journal. Students taking this course may have taken analytical chemistry, physical chemistry, or biochemistry. CHEM 410 focuses on fundamental principles in spectroscopic, electrochemical, and chromatographic techniques. The labs are designed to help students build practical working knowledge of different instruments and techniques and to develop their laboratory skills beyond the introductory level. They also gain a deeper understanding of the principles and applications of some of the techniques taught in other upper-level courses. Designing good teaching labs for the analytical course is critical in training undergraduate students taking these courses and introducing a CURE project in the laboratory syllabus has unmatched benefits. Research based inquiries help students to articulate a concise research question, and then design and execute an experimental approach to address the question. The laboratory activities carried out before the CURE project helped the students refine their laboratory skills, learn new laboratory techniques, develop skills in data collection
and interpretation, and helped them to be productive in a research setting. In addition, students are introduced to other aspects of experimental work that they have not encountered in other chemistry courses including, statistical data treatment, sample preparation and treatment methods. Students work in groups of two or three to enhance collaboration with their classmates. The CURE research project was introduced during the last five weeks of the semester. Four to five weeks is a typical time frame for chemistry CUREs exercises. Table 1 shows the general outline of the experiments carried out before the CURE project. Therefore, through the CURE project, students were given an opportunity to engage in scientific practices by using the knowledge and the skills that they had accumulated during the semester. In addition, students through the lecture that is associated with the lab, had opportunities to search scientific literature and effectively communicate scientifically through a term paper that is part of the class.

Table 1: Summary of Laboratory Activities

<table>
<thead>
<tr>
<th>Week</th>
<th>Lab Activity: Concepts Explored</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Digital data processing: signal-to-noise ratios</td>
</tr>
<tr>
<td>2</td>
<td>Fundamentals of UV-Vis spectroscopy: Beer’s law, performance of different optical instruments</td>
</tr>
<tr>
<td>3</td>
<td>Simultaneous analysis of a two-component mixture by UV-Vis spectrophotometry: standard calibration curve</td>
</tr>
<tr>
<td>4</td>
<td>Synthesis and characterization of gold nanoparticles (AuNPs) by UV-Vis spectrophotometry: understanding color size-dependent of AuNPs</td>
</tr>
<tr>
<td>5</td>
<td>Vibrational frequencies: A comparison between infrared and Raman spectroscopy: fundamentals of IR and Raman, sample preparation</td>
</tr>
<tr>
<td>6</td>
<td>Fundamentals of gas chromatography (GC) using FID Detection: understanding retention in GC</td>
</tr>
<tr>
<td>7</td>
<td>Fundamentals of cyclic voltammetry at solid electrodes using a well-behaved redox couple: standard calibration curve, statistical data treatment</td>
</tr>
<tr>
<td>8</td>
<td>Electroanalysis of acetaminophen in a pain killer by cyclic voltammetry: standard calibration, sample preparation and treatment, accuracy, precision</td>
</tr>
<tr>
<td>9</td>
<td>Optimization of separation in high performance liquid chromatography: understanding retention in reverse-phased chromatography, precision</td>
</tr>
<tr>
<td>10</td>
<td>Determination of calcium in a dietary supplement using MPAES: standard addition, sample preparation and treatment, accuracy, precision</td>
</tr>
<tr>
<td>11-15</td>
<td>CURE project</td>
</tr>
</tbody>
</table>
CURE Project Description

Lead (Pb) is one of the most toxic heavy metal in the environment.\textsuperscript{34} Its presence is due to past human activities, notably lead in gasoline and lead-based paints.\textsuperscript{34,35} Although Pb is no longer used in paints and gasoline, once it is deposited, it moves very little through soil and can persist for a long time. The legacy of its use is evident in soils along roadways and in the cities.\textsuperscript{35} For example, in places where there is a long history of traffic congestion, such as in the inner cities, Pb accumulation is especially high in the soils.\textsuperscript{35} Exposure to lead has been associated with adverse human health effects especially in the neurological development of children.\textsuperscript{36} South Bend gained attention recently for its high levels of lead in blood in children.\textsuperscript{38} In the early 20\textsuperscript{th} century, the cities of South Bend and Mishawaka (neighboring South Bend) were the leading industrial manufacturing centers of the Midwest. There is no data on lead levels in soils in South Bend and surrounding areas to gauge possible exposure of the human population, (especially children) from contaminated soils. Lead-contaminated soils can pose a risk through direct ingestion, uptake in vegetable gardens, or tracking into homes.\textsuperscript{38} To this end, the goal of the CURE project was to correlate Pb levels in soils from several homes with the location (city, suburb, farms), the year the homes were built, and source (whether the soil sample was obtained from the front yard or the backyard). Students were requested to bring two soil samples from their homes, from the front and backyard, and provide information on location and the year their residence was built. Pb in soil was extracted using a modified EPA method 3050B\textsuperscript{39} and analyzed using MPAES at 405.78 nm.

CURE Project Requirement

A good CURE project should give students opportunities to make critical decisions in the research process and engage them in demonstrating authentic scientific practices. The project engaged students in activities that led to use of scientific practices, discovery, broadly relevant
work, collaboration, and iteration. Students had the opportunity to use scientific practices experience in developing the analytical method for lead detection in soil. They were involved in making decisions in experimental design and execution, data collection and analysis, and in using a current method for analyzing metal ions. By engaging in several lab activities that explored different analytical techniques, students had hands-on experience on several instruments and were able to make decisions on the best instrument to use in designing the analytical method. In labs 8 and 10 students had the opportunity to work with real samples and make decisions regarding sample treatment. They understood what the goal of the sample treatment was before embarking on qualitative and/or quantitative analysis. To prepare solutions for analysis they had to make decisions on the appropriate calibration method given the complexity of the sample. The experiments in labs 2-4, 6, 7, and 9, involved procedures where the analytes were contained in simple matrices that can be duplicated, whereas in labs 5, 8, and 10, analytes were in complex matrices. Finally, the lab reports for different labs prepared the students on how to interpret and analyze data using statistical methods. The CURE project involved discovery of how the lead levels in soil correlate with home location and age in our community. The work has a broad relevance in our community in assessing the exposure of the community to lead through soil. Students worked in groups, and the class shared data for the final report. The CURE project will be iterated to obtain more data on student impacts and on Pb levels from soils samples from other homes in South Bend and surrounding areas.

**Summary of CURE Project Structure**

Weekly pre-laboratory discussions were held during the lab lecture. The instructor acted as a mentor and provided suggestions during the lab lecture and in the lab. Students were involved
in designing the analytical method to determine Pb in soil; the instructor used questions to trigger the discussions and create a research mindset.

*Week 1: Discussion of the research project.*

The instructor had introduced the research problem early in the semester. Students were responsible for identifying the appropriate analytical technique for determining lead and thinking through the steps involved in a chemical analytical problem. The instructor led the discussion on why the detection of lead is important, the toxicity of lead, and its presence in the environment. In the previous weeks before the CURE project, the instructor had tasked the students to find literature on lead toxicity. To identify the analytical method appropriate for lead analysis, the instructor initiated the discussion by asking questions, for example, “among all the techniques we encountered, which one do you think is appropriate and why?” Discussions involved discussing how the properties of the analyte and the principles of the analytical technique help to guide and design the analytical method appropriate for lead detection. Students were able to identify MPAES as the best analytical method to use for the research problem. From previous labs (labs 8 and 10) students were aware that real samples need some form of treatment to transform the analyte to a form that it can be detected by the analytical technique chosen. The instructor tasked the students to search literature on lead extraction from soil samples. In addition, obtaining a representative sample for analysis is critical; therefore, the art of sampling to procure a representative sample for the analysis was also discussed.

Discussion on the calibration method that was appropriate for the lead soil samples was carried out. Students had experience with both the normal standard calibration curve (labs 3 and 7) as well as the standard addition method (lab 10) for analysis of analytes in a complex sample matrix. They understood how matrix effects affect determination of an analyte in a complex matrix, and made
the decision on using the standard addition as the appropriate method for analyzing the soil sample solutions.

**Week 2: Sample preparation.**

The students’ literature search revealed there are several ways to extract lead from soil; the instructor guided the discussion on the choice of the procedure to carry out. The EPA method 3050B was discussed in detail and was chosen as the extraction method to be used. The instructor guided the discussion on modifying the EPA method to simplify it so that the extraction procedure was completed within the lab allocation time. The procedure involved weighing about 1 g of soil sample in a 50-mL beaker followed by adding 10 mL of 50% nitric acid. The mixture was then heated at 200°C for 30 minutes while covered with a watch glass. After heating, the mixture was cooled and then 2 mL of water and 3 mL 30% hydrogen peroxide were added followed by heating gently until effervescence subsided. The mixture was cooled, filtered in a 100-mL volumetric flask, and finally diluted to the mark with distilled water. The performance of the extraction method was evaluated by determining the percentage recovery of lead in a lead certified reference material which was 97.4 ± 2.8% (n = 3).

To ensure safety during the extraction procedures, the instructor established safety rules and guidelines to be followed by the students during the process. Each student carried out sample preparation procedures that included sieving of the soil sample, measurement of the soil moisture content, and extraction of lead using the EPA modified method.

**Week 3: Chemical analysis**

In lab 10 students were instructed on how to calibrate the MPAES instrument, run samples, and obtain data. In week 3 of the CURE project, the instructor guided the students in calibrating the
MPAES instrument using standard lead solutions. Each student prepared multiple solutions for a standard addition procedure followed by the analysis of the solutions using MPAES instrument, which is easy-to-use and is faster than the conventional flame atomic absorption for analyzing metal ions. Therefore, all the students analyzed their soil solutions within the 4-hr lab allocation time.

*Week 4 and 5: Data analysis and report write up.*

Each student prepared a standard addition plot to determine the concentration of Pb in his/her soil samples. Data from each student was collected and combined and was used to prepare the final report; students worked in groups of three for team building purposes to write up the final report. The instructor emphasized the benefit and the value of collaboration. Previous project discussions were held during the prelab lecture and in the lab, but for the report each group decided on its own when to meet and work on it. Students were responsible for analyzing and interpreting the data, making scientific argument about the data, and ensuring that the report followed the format of a journal.

**RESULTS AND DISCUSSION**

**Project Evaluation**

Nine students participated in the CURE project: four students white, three minority, and two Asian; seven were females and two males. As a starting point to evaluate the success of incorporating a CURE project in upper-level analytical chemistry various, Likert-scale self-reported survey data was collected. At the end of the CURE project, the nine students completed a blind attitude survey composed of the questions below. They were asked to rate their perception
on each question and give a rating between 1 and 5, 5 being the best. Table 2 reports the average and standard deviation of all students’ responses to each question.

1. Was the research project interesting and exciting?
2. The project was related to a real-world problem. Did this make the project more interesting and exciting than the other labs you performed during the course of the semester?
3. Do you feel the project gave you an opportunity to apply the concepts you learned in the class?
4. Did the project challenge you to think about the application of science in real life situations?
5. Did the project strengthen teamwork?
6. Would you like to participate in another course-based research during your undergraduate studies?
7. Do you feel the project benefited you for future professional development?

Table 2: Results of the Attitude Survey

<table>
<thead>
<tr>
<th>Question No.</th>
<th>Average Rating ( n = 9)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.5 ± 0.5</td>
</tr>
<tr>
<td>2</td>
<td>4.6 ± 0.7</td>
</tr>
<tr>
<td>3</td>
<td>4.6 ± 0.5</td>
</tr>
<tr>
<td>4</td>
<td>4.9 ± 0.3</td>
</tr>
<tr>
<td>5</td>
<td>4.1 ± 0.8</td>
</tr>
<tr>
<td>6</td>
<td>4.9 ± 0.3</td>
</tr>
<tr>
<td>7</td>
<td>4.8 ± 0.5</td>
</tr>
</tbody>
</table>

The students’ written evaluations had three common themes: science real-life application, collaboration, and a personal connection with the project. The choice of research topic was the key element in the success of the CURE project. A project that is relatable to the students played a big role in the student attitude toward the project. The choice of an appropriate research topic has been shown to play a key role in the success of other CURE projects. Establishing the relevance of
course work to the outside world increases interest, persistence, and a drive to achieve goals. Research has shown that if students value assigned work, understand its relevance to classroom instruction, and its application to real-life situations, they not only invest time and effort in that work, they also recognize that their teachers are providing useful experiences. The majority of the students responded that the CURE project was interesting and exciting. Students’ comments indicated that the main reason they were interested in the project is that it addressed a research question that was not only an interest to them but fitted into a broader scientific endeavor and applied to the community they live in. The positive responses indicate positive students’ experience and outcome. Some of the students’ remarks were:

*I was excited because I wanted to know how much lead was in our soil because we grow vegetables and herbs in our back yard.*

*I do gardening and I care about the environmental issues. It was nice to work on something that has a direct impact on my life.*

*The project was pretty interesting because we used our own soil samples.*

*I did not consider doing research at all, and this has entirely changed my perspective.*

*I had a chance of doing undergraduate research, which I would not have been able to if it was not incorporated in the class.*

*I loved when we had the opportunity to discuss the project with our classmates all by ourselves. Everyone contributed to the conversations and this felt really nice; we felt like real scientists.*

Students showed an increase confidence and in critical thinking abilities. Engaging in the project prompted several scientific discussions during the lab sessions that were not common during the normal lab sessions, giving students an opportunity of an exciting discourse in the lab.
and an improved appreciation of analytical chemistry and science. For example, some of the students discussed the appearance of their soil samples in comparison to other soil samples. Others discussed possible reasons why some soil samples had a high amount of lead and others had low amounts. Many of the students felt the analysis was personal because it affected their lives, and they felt a personal obligation to accurately determine the amount of Pb in their soil samples. The observed benefits to the students agree with other CURE laboratories in upper-level chemistry courses.\textsuperscript{19-25,31} Though publication of research is very important and boosts the self-confidence of the students involved, the benefits that students gain by engaging in research extend beyond articles published. The most important part of research for an undergraduate student is the transformation that takes place during the research process. The benefits they reap go beyond their undergraduate degree and help them succeed in their future science career.

Based on students’ self-assessment the project was a success. However, there were a few obstacles observed by the instructor and reported by students: time management and creating a research environment. Some students felt having a CURE project report and a term paper was a bit overwhelming because they were both due toward at the end of the semester. In the future, the course will be re-organized to separate the two due dates and emphasize the importance of time management and hard work to succeed in research. In the beginning, creating a research environment in the lab required some effort from the instructor to help some of the students who had no prior research experience understand the research process and have a research mind-set to help them understand why the project was included in the course. Students who had engaged in research also shared their experience and these efforts helped in creating a collaborative research environment.
Demonstration of a higher level of critical-thinking skills

The final project reports were marked by a demonstration of a higher level of critical thinking not observed in previous reports. For example, students on their own came up with reasonable explanations on the lead level trends, and reasonable arguments to discuss the data. Some of the students demonstrated an ability to come up with their own research questions and future projects related to lead. For example, one of the students suggested measuring the levels of lead in water and relate that to the age of the homes. Lead can enter drinking water when plumbing materials that contain lead corrode, and the most common sources of lead in drinking water are lead pipes, faucets, and fixtures in homes built before 1986.41 In their reports, some of the students compared the lead levels at different locations with the EPA guidelines,38 demonstrating an ability of using research findings to make informed decisions about a real-life problem.

Data analysis showed a correlation between the amount of the lead in the soil with home location, source, and age. Homes located in the city showed a higher amount of lead compared to homes located in the farms or in newly developed areas. There was also a good correlation between the levels of lead in soil samples collected from the front yard and those obtained from the back yard in homes located in the city, with samples from the front yard showing a slightly higher level. The importance of dissemination of scientific research was discussed; an abstract that summarized the CURE research findings was submitted and accepted in two conferences, our spring Undergraduate Research Conference (URC) and the National Conference for Undergraduate Research (NCUR). Even though the majority of the students had not attended a research conference or presented a poster before this course (only three of the nine students had prepared a scientific poster prior to taking the class), they were motivated to present the research findings in
a conference. One of the students received a travel grant to present a poster during the NCUR conference and the class planned to present a poster jointly during the URC conference.

CONCLUSION AND FUTURE PLANS

This CURE project received a lot appreciation from the students, especially because it gave a larger number of them an opportunity to engage in research when they would not otherwise have had such an opportunity due to their work schedules and lack of enough faculty mentors. The CURE project was expected to motivate students who were not graduating soon to consider seeking research opportunities in the department or apply for research experiences for undergraduate opportunities funded by National Science Foundation in R1 research institutions; two of the students started independent research since taking the course. Incorporating the CURE project in the course was successful measured by the student-reported assessments and surveys feedback. In the future, a CURE project will be part of the C410 syllabus. In the next couple of times of teaching the course, there will be iterations of the project to obtain data on student impacts. One way to measure the effectiveness of the project will be to have initial and final attitude surveys to gauge the success of the project and provide a means for course improvement and direction. Students will be asked to respond to questions including what they understand by engaging in a research project, how well do they feel prepared to undertake the activity, what they hope to gain from the experience, and what challenges they expect to encounter in the research process. After the project they will be asked to provide responses on their experience and how engaging in research has changed their view on research, how the challenges they encountered shaped their attitude toward research and science, and how they think the lessons they learned during the research process will help them in their chemistry career.
The author hopes this work will provide inspiration for other chemistry programs in PUI institutions with fewer opportunities for students to engage in research.

ACKNOWLEDGEMENTS

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Notes

The author declares no competing financial interest.

REFERENCES


