Polystyrene Laboratory Analysis: A Hands-On Experience to Determine the Molecular Weight of Polystyrene Through Spin Casting in A University Laboratory Setting

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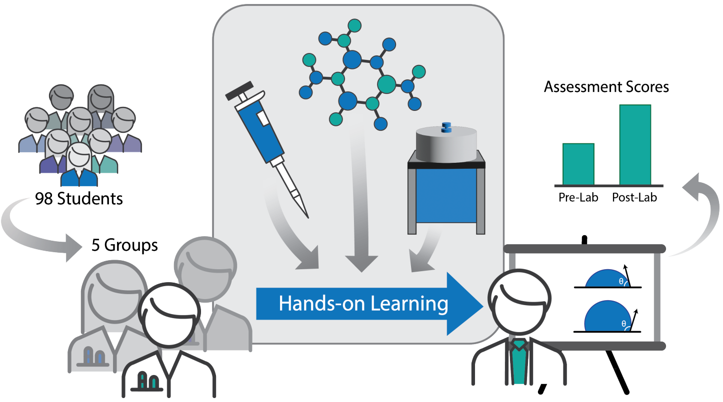
ABSTRACT

Hands-on learning is a staple in high school science education, as it provides students with a fast-learning curve and a great degree of field competency. However, due to the safety risks associated with high school students in university chemistry laboratory settings, high school students rarely engage in authentic hands-on chemical learning. To bridge the gap between the benefits and drawbacks, this study investigates a method to educate high school students (with no previous experience) about standard chemical laboratory practices. 98 high school students experimented throughout two days to determine the molecular weights and characteristics of various polystyrene samples, essential knowledge for polymer recycling. Students were split into 5 groups so that laboratory usage be organized and staggered. After laboratory safety training was administered, students created different types and concentrations of toluene-based samples and spin casted these samples onto silicon wafers, determining thickness through ellipsometry. With the data, each group calculated molecular weight, propagated error, and wrote laboratory reports. In order to evaluate the extent of learning through this process, students were given pre-training and post-experimentation assessments with the same questions pertaining to laboratory safety, equipment usage, and materials science related topics. On average, students displayed scores 63% higher on the post-experiment assessment compared to those of the pre-training assessment. The results suggest the experience not only taught students about the various materials science concepts, but also improved their laboratory logic. Therefore, our method is recommended to be implemented at the university level for motivated high school and first-year undergraduate students.

GRAPHICAL ABSTRACT



KEYWORDS

First-year Undergraduates, Laboratory Instruction, Hands-on Learning, Materials Science and Chemistry, Laboratory Equipment

PART A

Introduction

Numerous studies have been conducted in the comparison between multimedia and hands-on learning to indicate that students are able to understand a topic quicker and more accurately when obtaining hands-on experience. Previous research has shown that emotional intelligence is more accurate in predicting student success compared to IQ[1]. Cooperative group learning, where the interactions between students are courteous and inclusive, has been shown to encourage academic and social growth[2]. In particular, hands-on, extracurricular projects have shown to enhance student interest in science and increase problem[3]. Hand-on learning also allows for longer term retention of information and provides competency-based education, including engagement in learning and collaboration[4].

In 2018, 35.7 million tons of plastic waste was generated in the United States, which was 12.2 percent of municipal solid waste[5]. Only 8.7% of plastic was recycled in 2018, resulting in landfills receiving 27 million tons of plastic. Furthermore, plastic is non-biodegradable, remaining for long periods of time in landfills[6]. Approximately 8 million tons of plastic waste enter oceans annually, affecting many coastal environments and harming wildlife[7]. Thus, it is imperative to emphasize the need to reduce plastic waste and develop a technique to facilitate its recycling. This laboratory exercise not only introduces students to a variety of laboratory techniques, but also emphasizes the need to develop a viable method of recycling plastics.

By determining the molecular weight of a polymer such as polystyrene, the function and maximum capacity of the polymer can be deduced, which has significant implications for recycling and reusing discarded polystyrene waste. Determining a polymer's molecular weight is crucial in utilizing the polymer and its properties, such as viscosity, melting point, and plastic chemical resistance. For example, knowing the molecular weight of polystyrene enables calculating the approximate length of the polymer chains, which relates to numerous properties. Polymers with a higher molecular weight have longer polymer chains, increasing their viscosity and chemical resistance due to their ability to absorb more energy.

There are many ways to determine the molecular weight of a polymer in the lab. General methods include permeation chromatography (GPC), matrix-assisted laser desorption/ionization time-of-flight mass spectroscopy (MALDI-TOF MS), solution viscosity, cryoscopy, light scattering, and boiling point elevation. While other methods of molecular weight of polystyrene determination use a single procedure, this experiment utilizes various methodologies and equipment to find the molecular weight of polystyrene, thus exposing the student to many laboratory techniques while also employing a simpler and low-cost analysis.

A previous experiment had spun-cast polystyrene-toluene solutions onto glass slides, then measured thickness with FTIR and the Beer–Lambert law to generate a thickness vs. concentration curve. However, glass slides were used as opposed to silicon wafers, a fan instead of a research-grade spin casting machine, FTIR rather than ellipsometry for thickness, and did not use the information to determine the molecular weight of the polymer[8]. Another experiment analyzed various materials as substrates for polymer spin casting but did not use silicon wafers[9].

This report strives to answer questions regarding the types of techniques that will most effectively introduce a large group of students without prior laboratory experience to methods for determining the molecular weight of a polymer in a condensed time, emphasizing the importance of determining molecular weight as a key for applications in recycling.

This experiment process was designed as a way for high school students to transition from basic high school laboratory work to advanced hands-on experience in a university setting. It served as a first for many regarding the experimental approach, such as the importance of maintaining experimental controls, accounting for error, and literature review; laboratory safety and the wearing of PPE, keeping a lab notebook, avoiding contamination, and the safe disposal of hazardous waste; and realistic laboratory expectations like collaboration and communication between project participants and the vitality of asking questions to fortify understanding.

This experiment introduces 98 high-school students to basic principles of laboratory safety in practical application, with procedures for operating laboratory equipment like Fourier-transform infrared spectroscopy (FTIR), ultraviolet-visible spectroscopy (UV-Vis), differential scanning calorimetry (DSC), contact angle goniometry, compression molding, optical microscope visualizations, spin casting, and ellipsometry. Through guided instruction and demonstrations, students acquire relevant laboratory techniques to improve their proficiency for future research pursuits in college and beyond. Dividing the students into several research groups consisting of 8-10 students each, the simplicity of the experiment allows them to become familiar with laboratory procedures on a fundamental level, while also improving their skills of collaboration. In addition, while adhering to the familiar structure of the scientific method that they initially learned in middle school, it reinforces the fact that it still applies to all levels of research.

Students first learned about polystyrene – both its benefits as a durable and versatile material and its determinants as a non-biodegradable material. Then, they received a comprehensive lab tour to understand the purpose of each instrument and the significance of each one’s results. They were introduced to the idea of error and the various statistical tools that assess it, including error bars, standard deviation, and error propagation. Finally, they were taught the importance of maintaining a detailed lab notebook that outlined all steps of an experiment and recorded results. Lab protocols were emphasized, including the wearing of PPE, cleaning and preparing lab equipment, and safely transporting and storing samples.

Once students were familiar with the necessary lab concepts, they were divided into groups to tackle the experiment from different angles. Some groups worked with a polystyrene sample of known molecular weight, serving as the control; other groups worked with polystyrene samples from ubiquitous products to determine their unknown molecular weight. Each group formed a hypothesis for their specific sample before beginning the experimental phase. Since each group needed to employ the same equipment, their experimental procedures had to be carefully scheduled so that no two groups were using the same equipment at once, highlighting the value of organization when collaborating with others in a laboratory setting. After groups finished collecting their data, they calculated the molecular weight of their polystyrene samples and accounted for error. Each group composed a detailed lab report outlining the purpose of the experiment, the methods, data, calculations, and the implications of their results. Lastly, groups presented their findings to each other to obtain a clearer picture of the efficacy of their methods and the accuracy of their results.

At the end of the experiment, the students had effectively gained a comprehensive overview of the lab instruments available on campus, how to operate them, how they worked, and the significance of the data they yielded. Due to the thorough instruction provided by mentors and the students' own notetaking and experiment writeup efforts, they were left with the ability to perform this experiment on their own without step-by-step guidance.

PART B

Pre-Laboratory Training

The 98 high school students involved in the experiment were all attendees of a summer research internship at Stony Brook University. All students were above the age of 16, therefore eligible to conduct experimentation in university facilities. In the program's first week, students were introduced to the concepts of materials science research through lectures by researchers and educators at Stony Brook University. They learned about laboratory notebook keeping, scientific research process, and authentic research projects with diverse applications. These lectures allowed the students, some of whom had no previous research experience, to get a sense of how to operate in a materials chemistry laboratory, making them more comfortable in a lab environment and more knowledgeable about the project they would later choose to investigate.

Following this, laboratory safety lessons were presented by representatives from Stony Brook's Department of Environmental Health and Safety. On the first day a mandatory chemical hazards presentation was given to all students, equivalent to two semester-long university-level chemical safety courses. Basic safety concepts such as RAMP (Recognize, Assess, Minimalize, and Prepare), PPE (Personal Protection Equipment), fire plan, emergency eyewash and shower, safety data sheet (SDS), and chemical fume hood usage were presented. Students were also taught how to write a Standard Operating Procedure (SOP) before they commence experimentation. Afterward, students were required to demonstrate their chemical safety knowledge through a ten-question exam via google forms; a score of 80% or higher was required to move on to the next step.

After passing the aforementioned assessment, the students were given an afternoon of laboratory facility tours, where graduate students and postdoctoral researchers described the equipment in each room, such as the ellipsometer, goniometer, differential scanning calorimeter (DSC), Fourier Infrared (FTIR) Spectrometer, and spin caster; in some cases, example demonstrations were given (FIG 1). After the lab tours were completed, students were sent slides regarding the proper use and specifications of each piece of equipment. On the following day, students were tested on the use and specifications of the equipment through a 10-point assessment. After scoring 80% or higher, students were handed their PPE, goggles and lab coat, and a laboratory notebook in preparation for experimentation. In this way, students went through an extensive preparation before embarking on laboratory work.

Figure 1. DSC demonstration. Figure generated by authors.

Samples

Prior to experimentation, students were instructed to collect polystyrene-based consumer goods. The three sources—coffee cups, ramen noodle cups, and plates—became the experimental samples of unknown molecular weight. Smaller pieces were then cut out of these large items to create the samples used in solution making. Two control samples with known molecular weight—Aldrich 35k MW polystyrene and Aldrich 280k MW polystyrene—were obtained from the Stony Brook University Materials Science Department. Semicircular silicon wafers were provided to students to practice cleaving and to examine under an optical microscope. Finally, 99.9% toluene for HPLC was also provided to make the polystyrene solutions needed for spin casting.

Equipment/Techniques

Since the goal of the experiment was to determine the molecular weight of various samples of polystyrene, each piece of equipment was used by students to analyze a different property of polystyrene that was affected by the sample's molecular weight. For example, students used a differential scanning calorimeter (DSC) to measure glass transition temperature of polystyrene samples for compression molding. A compression molder shaped the samples into specific shapes to have their chemical compositions analyzed by the Fourier Transform Infrared (FTIR) spectrometer, based on the wavelengths of light that are absorbed. UV/Vis spectroscopy was also completed to find molecular absorption properties of the samples. Before being cut into 1 cm^2 pieces for spin casting, silicon wafers were placed under an optical microscope with a digital display to allow students to see the structure and orientation of the material they were working with, and then to determine which type of lattice structure would be most suitable. Students cleaved silicon wafers and spin casted the polystyrene samples. Using these coated wafers, the students measured contact angles and hydrophobicity on a goniometer and perform ellipsometry to determine film thickness, which was used in the calculation of the samples' molecular weight.

Experimentation and Data Collection

The student spin casting experiment took two days to complete. Prior to the first day of the experiment, students were split into ten groups of ten students in each. Nine of the groups were sorted based on alphabetical order, and the last group consisted of all the virtual students on Zoom, making the project hybrid in nature with some students researching in the labs themselves while others observed the process online. Each group also had two research experience undergraduates (REUs) who conducted the experiment alongside the high school students. Combined groups 1 and 2, as well as combined groups 5 and 6, served as the controls who worked to confirm the 280k and 35k molecular weight samples of polystyrene, respectively. Then, every other two consecutive groups worked together to obtain the molecular weight of the other polystyrene samples, such as a coffee cup or ramen cup (FIG 2).

Each pair of groups went to their assigned rooms and met with a postdoctoral researcher or graduate student to discuss the proper procedures of the experiment, specific safety measures, and the equipment utilized. On the first day, students prepared polystyrene solutions of each small group's concentration (5-27.5mg/mL) in toluene and cleaved silicon wafers into one cm by one cm squares, all of which was completed in four hours. On the second day, the students spent 3 hours performing the tests mentioned in the equipment section, such as UV/Vis and DSC. After, samples were spin casted onto the silicon wafers at 2500 rpm for 30 seconds each. Ellipsometry was performed on the samples to determine thickness of the spin casted polystyrene concentrations. Based on thickness, molecular weight of the different polystyrene types was calculated, further elaborated on in the results section.

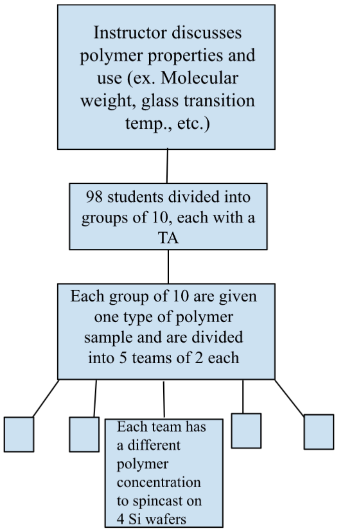


Fig 2: Each team of two students are assigned a different concentration of their polymer and given a silicon wafer to cleave into 4 parts, two for each student. Producing four trials per concentration enables error analysis and promotes collaboration between the five teams of each group to combine data and plot concentration vs. film thickness for their polymer. Figure generated by authors.

Calculating Universal Curve for Prediction of Molecular Weight

To validate the scientific reasoning behind using spin casting in identifying molecular weights of different polystyrene samples, a group of four students tested five more samples of pure polystyrene with known molecular weights of 123k amu, 200k amu, 400k amu, 650k amu, and 900k amu. Using the same procedures as before, the students spun cast toluene-polystyrene solutions of various concentrations onto silicon wafers for analysis.

Once data correlating thickness and concentration was gathered regarding the different Molecular Weights, the data was plotted, and a best fit curve was determined for the data. Using the best fit curve, a critical concentration was determined at each thickness, 1000, 1250, 2000, and 3000 Angstroms. Using this information and the provided equations below, the molecular weight was calculated for each thickness.

For each of the four critical thicknesses below, use the concentration of polystyrene to determine the predicted molecular weight utilizing the respective equations listed below:

Equation for 1000 Å: $ln\, y = \ln(1.207 * 10^{12}) + (-5.766)\, ln\, x$

Equation for 1250 Å: $ln\, y = ln\,(8.965 * 10^{11}) + (-5.358)\, ln\, x$

Equation for 2000 Å: $ln\, y = ln\,(8.742 * 10^{11}) + (-4.802)\, ln\, x$

Equation for 3000 Å: $ln\, y = ln\,(1.415 * 10^{12}) + (-4.509)\, ln\, x$

Note: In the equations above, y stands for predicted molecular weight, and x stands for concentration.

Using the calculated molecular weights, the best predictor of the curve was determined by performing error propagation with the values at each thickness. The error in concentration was calculated via error propagation with the curves of best fit of concentration vs. thickness. This error was then multiplied with the derivative of molecular weight relative to the concentration, which will help us calculate the error in molecular weights from the measured values.

Error Analysis

Having the students reflect on sources of error encouraged questioning aspects of the experiment that they might have otherwise considered infallible, increasing awareness and motivating them to reduce those sources of error in their real lab work. Students were given formal instruction in error analysis. They learned to identify possible sources of error in four different categories: human, environmental, instrumental, and random error; statistical procedures of proficiency testing; error propagation and chi-squared analysis; and the confidence level/level of significance.

Assessment of the Learning Process:

The experiment's hands-on learning efficacy in teaching students proper laboratory safety and experimental procedures was empirically determined by pre-experimentation and post-experimentation assessments conducted on Google Forms. The pre-experimentation and post-experimentation assessments contained the same 20 multiple-choice questions about laboratory safety, experiment-specific information, and materials science-related topics. They served as a way for the instructors to track improvement in students' comprehension. Each question was also put into a subsection to compare sub scores (laboratory safety, equipment usage, experiment-specific, chemical terminology). Students were not provided with a copy of their previous answers or scores to ensure testing integrity. The assessments recorded scores that reflected students' understanding of laboratory techniques and materials science concepts, and their responses were compared to illustrate the extent of the experiment's impact. The results were displayed as mean ± standard error (SEM). We then performed a paired T-test via GraphPad to confirm statistical significance.

Furthermore, students were required to report on their experiment results through a lab report and slideshow presentation, both of which will be elaborated on in the results section.

PART C

Hazards

Before starting experimentation, all students were trained in laboratory safety techniques and emergency situations through the previously mentioned chemical safety lectures. In addition, graduate students and postdoctoral researchers closely supervised the high school experimenters as they engaged in laboratory activities, especially when handling hazardous chemicals such as toluene. Students followed specific protocols for different laboratory procedures, such as disposing silicon wafer pieces in a sharp's disposal container. Diamond cutters were stored in labeled containers for use when cutting silicon wafers. A fume hood was used to protect students from toxic fumes such as those emanating from the toluene solvent. Students were instructed to check the airflow, keep work 6 inches within the fume hood, set sash height (closed when experiment completed), and keep the workspace clean (FIG 3). As a final line of defense, students were all equipped with PPE equipment. Instructors ensured that all chemical waste was discarded in appropriately labeled waste bottles without being mixed with other potentially reactive substances.

Fig 3. Students learning about fume hood operation. Figure generated by authors.

PART D

Assessment of the Learning Process: Pre/Post-Experimentation Assessments

On the pre-experimentation assessment, students scored on average 30.21% (30.21±1.78) of the questions correctly. On the post-experimentation assessment, students scored on average 93.75% (93.75±1.07) of the questions correctly. Therefore, the students scored 63% higher on the post-experimentation knowledge check compared to scores on the pre-experimentation assessment, indicating a 210 percent increase in mean score (FIG 4). With a paired t-test at a sample size of 98 students, these results yielded a p-value less than .0001, indicating high statistical significance. In particular, questions categorized as "experiment-specific" showed an 84% increase in proficiency, highlighting hands-on learning's ability to facilitate knowledge retention of the experiment. The pre-experimentation assessment's data featured outliers with a few students scoring nearly 20% higher than the mean score on the knowledge check. A possible explanation for these outliers is that some students may have possessed previous laboratory experience or a keen knowledge of material science principles, resulting in a skewed right distribution for pre-experimentation responses. However, for the post-experimentation, the entire sample size had no significant outliers and the distribution showed a general positive trend of improvement, illustrating how the experiment was able to take students with and without prior laboratory experience and increase their proficiency in lab safety and material science knowledge to the same level.

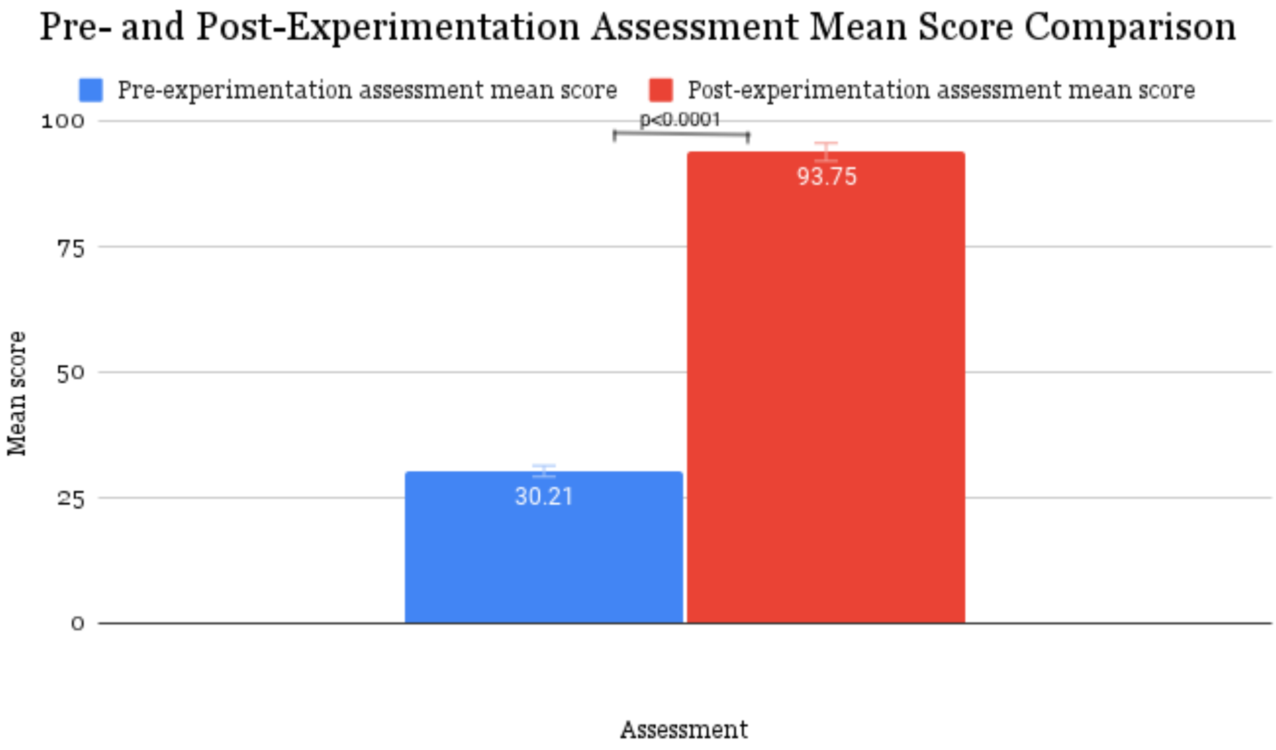


Fig 4. Comparison of mean scores on assessment before and after experimentation. Figure generated by authors.

Assessment of the Learning Process: Student Lab Reports

The lab reports produced by students after the spin-casting experiment included an abstract, introduction, methods and materials section, data analysis and results section, error analysis section, conclusion, and references.

The abstracts of each lab report contained the main reason of the conducted experiment or why the experiment would be impactful, the purposes of the experiment, a brief description of the methods used to obtain the results, and the potential impact of the findings on society as a whole. The introduction includes a more detailed description of polystyrene's chemical properties and industrial applications, in addition to the objectives of the experiment. Students were required to cite at least three references from accredited sources. Students learned the ethics of group work, giving proper credit to previous research and producing their own original work. Next, students recorded in their own words their experimental procedure and the materials and instruments that were required. Based on their data, students produced images, graphs, and curves that were subsequently analyzed to determine the polystyrene samples' molecular weights or experimentally verify the known molecular weight of the control samples. They plotted film thickness versus polymer concentration and then extrapolated from the graph their sample's molecular weight. Students then identified, analyzed and statistically calculated the sources of error in the experiment. They further characterized their polymer with FTIR and DSC and learned the value of recycling by producing thin films from discarded polystyrene containers. After completing their lab reports, each group of ten students could then give an oral presentation of their findings to all.

Limitation of the Experiment (Classroom Setting)

This spin casting experiment was conducted by high school students in university facilities under the careful supervision of graduate students and professors. Due to the large volume of experimentation, careful oversight of every step in the process was not possible. As a result, there were many feasible sources of error. For example, the silicon wafers that were cut by students may not have been uniform in size, resulting in irregular samples when spin casting. Furthermore, the silicon wafers were not cleaned with compressed air before spin casting for some experimental groups, which may have caused dirt or other particles to coat the surface of the sample, affecting the ellipsometer readings. Another plausible source of error could be improper use of the ellipsometer. It may have not been calibrated or focused properly, affecting the accuracy of its readings. A combination of these factors led to inaccurate initial molecular weight determinations.

Additionally, various labs and equipment utilized such as chemical fume hoods, differential scanning calorimeters, and FTIR spectrometers may not be readily available to a substantial number of high schools who may wish to replicate this experiment for their students. A lack of qualified personnel supervising the experiment and resources to provide the required materials could also limit the number of schools that can conduct this experiment. In addition, since toluene was used as a solvent, fume hoods and a means of hazardous waste disposal must be available. Although it is difficult to discuss the feasibility of implementation in schools across the country, given irregular educational funding, there is potential for alternative solutions such as virtual lab experimentation or organized partnerships with local universities, so that students nationwide have the opportunity to perform the experiment and gain valuable experience in science research.

However, by no means is this project limited to high school student instruction. On the contrary, it can also be well-implemented in an undergraduate as well as a graduate level. It spans not only the disciplines of Chemistry and/or Materials Science, but also the fields of Environmental Science and Waste Management Specialists. Through this exercise, the practicality of recycling plastics is brought to light. In addition, it emphasizes collaboration, organization, lab safety, data and error analysis, and drawing conclusions based on results, which are important skills for all science fields and all disciplines in general. Hence, the educational benefits of this laboratory exercise are multifaceted and can prove to be productive to a wide range of student instruction.

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