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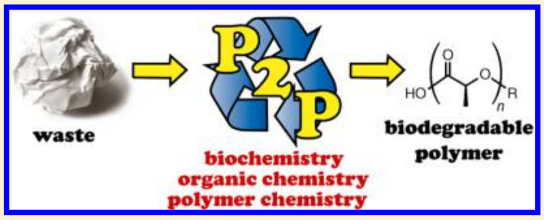
Paper to Plastics: An Interdisciplinary Summer Outreach Project in Sustainability

Fiona Tamburini, Thomas Kelly, Eranthie Weerapana,* and Jeffery A. Byers*

Department of Chemistry, Boston College, Chestnut Hill, Massachusetts 02467, United States

Supporting Information

ABSTRACT: Paper to Plastics (P2P) is an interdisciplinary program that combines chemistry and biology in a research setting. The goal of this project is 2-fold: to engage students in scientific research and to educate them about sustainability and biodegradable materials. The scientific aim of the project is to recycle unwanted office paper to the useful biodegradable polymer poly(lactic acid) (PLA). Through this program, students learn firsthand how chemistry and biology interact to form useful materials from waste. Students combine biological techniques, such as enzymatic digestion and fermentation, with chemical techniques, such as distillation and catalysis, to accomplish the conversion of waste paper into PLA. Through this summer program, students ultimately become familiar with diverse laboratory techniques, while learning how their scientific interests can be used to address important social problems.



KEYWORDS: *High School/Introductory Chemistry, Biochemistry, Organic Chemistry, Polymer Chemistry, Interdisciplinary/Multidisciplinary, Hands-On Learning/Manipulatives, Bioorganic Chemistry, Catalysis, Green Chemistry, Polymerization*

In response to low graduation rates among American students in science, technology, engineering, and mathematics (STEM) disciplines, The National Academy of Science (NAS) recently published suggestions for modifying STEM education in grades K−12.[1] The recommendations stressed a three-dimensional model for a successful educational program: (i) describe scientific and engineering practices; (ii) explore crosscutting concepts that have applicability across multiple scientific disciplines; and (iii) describe core ideas. These recommendations represent a dramatic paradigm shift in pedagogy away from representing science as an insurmountable collection of facts, to representing it as a creative and malleable discipline whose primary objective is to uncover the goings on of the natural world.

In line with the educational framework set forth by the NAS are educational outreach programs whose aim is to demonstrate scientific principles and thinking in the context of topics that are relevant and interesting to students. Engaging students in topics that they find interesting is particularly important, because research has shown that personal interests, experiences, and enthusiasm are a primary factor linked to later educational and career choices.[2−5] With these factors in mind, we set out to develop an outreach program designed to engage high-school-aged students in a project that best mimicked a genuine research environment. We intended to create a project that could be carried out in the summer months by colleges and universities that are equipped with standard instrumentation used in modern research environments.

In order to maximally engage the students, a socially significant and multidisciplinary topic was needed as the subject matter for the project. An ideal candidate that met these criteria is the synthesis of biodegradable polymers from waste. In the past few decades, petroleum-based plastics have fallen out of favor because they are not environmentally sustainable.[6] Consequently, there is an increasing demand for environmentally friendly alternatives to conventional plastics. Poly(lactic acid) (PLA) is a biodegradable polymer that is suited to many of the applications in which petroleum-based polymers are typically employed, such as grocery bags and food storage containers.[7] Currently, PLA is produced on an industrial scale from renewable starch sources, chiefly corn.[8,9] In theory, similar syntheses of PLA can be conducted on a smaller scale using any source of starch that is inexpensive and abundantly available. Used office paper not only is widely available at any college, university, or high school but also is an abundant and convenient source of starch. Through a series of biochemical and chemical transformations, we hypothesized that the cellulose derived from used office paper could be converted into PLA in the laboratory. Such a research project would be ideally suited to encourage high school students to participate in STEM disciplines as it addresses all three dimensions of the educational framework set forth by the NAS: (1) the students learn practices and procedures common to any scientific discipline, (2) the project uses a multidisciplinary approach to answer scientific inquiry, and (3) the project addresses many of the core ideas targeted by the NAS (e.g., matter and its interactions, the dynamic interaction of energy and ecosystems, earth and human activity, etc.).[1] Previous educational and outreach projects that incorporate some or all of these aspects have been reported as highly successful.[10−12]

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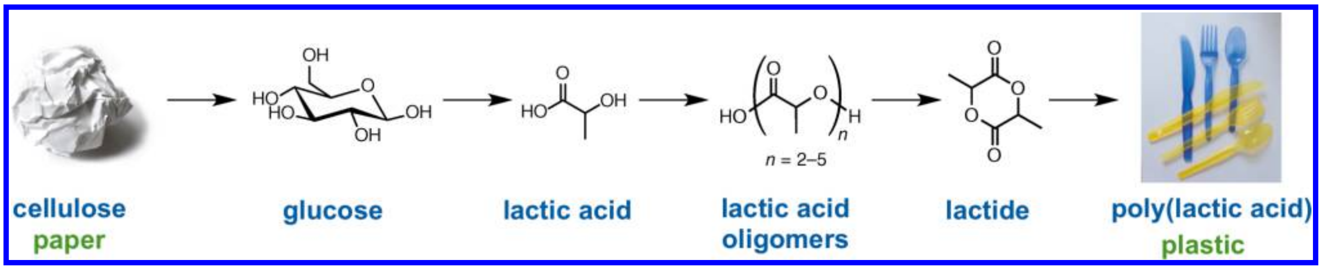
Scheme 1. Outline of Biochemical and Chemical Transformations Involved in the Conversion of Cellulose from Paper into Poly(lactic acid), a Biodegradable Plastic



In this publication, we describe the "Paper to Plastics" program (P2P) that has been developed in the chemistry department at Boston College. Through a series of laboratory experiments based on existing literature and industrial techniques,[13−18] we have created a sequence of modules that harness biological catalysts as well as traditional chemistry techniques to transform used office paper into biodegradable plastic (Scheme 1). Each experiment introduces students to salient topics in biology and chemistry, such as biological and chemical catalysis, chemical reactions, and organic synthesis. Though a broad range of topics is addressed, the techniques detailed are designed to be comprehensible and fully hands-on. At every stage, there is opportunity for inquiry and adaptation. As such, the P2P program can be readily tailored to a variety of academic settings. Our aim through P2P is to cultivate excitement for scientific research in students by allowing them to work on a process that is both stimulating and socially relevant. Under the guidance of two undergraduate mentors, six high-school students carried out this suite of experiments in the summer of 2013. In the summer of 2014, the program will be extended to 16 high school students.

■ RATIONALE

In keeping with the interdisciplinary aim of the program, the six experimental modules shown in Table 1 encompass multiple areas within chemistry and biology. The first three modules are more biology-oriented, while the latter three employ organic synthesis techniques.

Table 1. Summary of the Experimental Modules

Module	Experiment	Discipline[a]
1	Paper pulping and de-inking	G
2	Cellulase digest	BC
3	Lactic acid fermentation	B, BC
4	Lactic acid oligomerization	OC, PC
5	Lactide formation	OC
6	Lactide polymerization	PC

[a]The disciplines are general chemistry (G), biology (B), biological chemistry (BC), organic chemistry (OC), and polymer chemistry (PC).

■ THE EXPERIMENTS

The following modules detail the biological and chemical transformations necessary to convert office paper into poly(lactic acid) (Scheme 1). Detailed protocols for each of the steps are provided in the Supporting Information. To ensure a sufficient yield for each step, each module can be supplemented with purchased reagents. A more advanced group of students may wish to attempt to carry the original paper pulp through the entire series of transformations.

Module 1: Paper Pulping and Floatation De-Inking

Students use a home blender to create a homogeneous suspension of paper pulp. The process of removing ink contaminants makes use of the hydrophobic properties of ink pigments. To accomplish the de-inking step, the pulp is added to a large flask with deionized water. Upon addition of dish soap, the mixture is aerated through a tube connected to an air outlet so that bubbles form in the solution. The hydrophobic soap bubble attracts the ink particles and carries them out of the flask into a waste basin (Figure S1 in the Supporting Information). Students then vacuum filter and oven-dry the de-inked pulp, and obtain a yield from the original paper source.[16]

Module 2: Enzymatic Digestion

Cellulases are a family of enzymes produced by bacteria and fungi that catalyze the hydrolysis of cellulose.[19] Cellulase is used to break down cellulose in the paper pulp into glucose monomers (Figure 1A). To achieve this, a mixture of commercially available cellulase enzymes (from *Trichoderma reesei*) and cellulose is incubated at 37 °C, and students take aliquots at regular intervals to determine the most effective incubation time for the reaction.[15] The glucose yield is calculated using a commercially available Glucose (GO) Assay Kit (Sigma-Aldrich) that enzymatically oxidizes the glucose to gluconic acid and hydrogen peroxide. The resulting hydrogen peroxide then reacts with *o*-dianisidine in the presence of peroxidase to form a colored product. Product formation is monitored by absorbance at 450 nm. The percent yield can be calculated to determine the efficiency of conversion, as well as the most efficient length of incubation. To expand this module, students can opt to vary the ratio of cellulase enzyme to cellulose to determine the optimal concentration for catalysis. This module introduces students to enzyme catalysis and the effects of parameters such as temperature, time, and concentration on the amount of product afforded by an enzymatic reaction. Furthermore, the students learn to use a spectrophotometer and a coupled assay to monitor glucose formation.

Module 3: Fermentation

Students utilize the microorganism *Lactobacillus casei* to convert glucose to lactic acid. In this module, students learn about the nutritional and environmental requirements for bacterial growth. In a similar manner to the cellulase digestion, students inoculate glucose-free growth medium with bacteria and add varying amounts of glucose.[14] Aliquots of the fermentation broth are taken at daily intervals, and the lactic acid content of each sample is assayed with a Lactate Assay Kit (Sigma-

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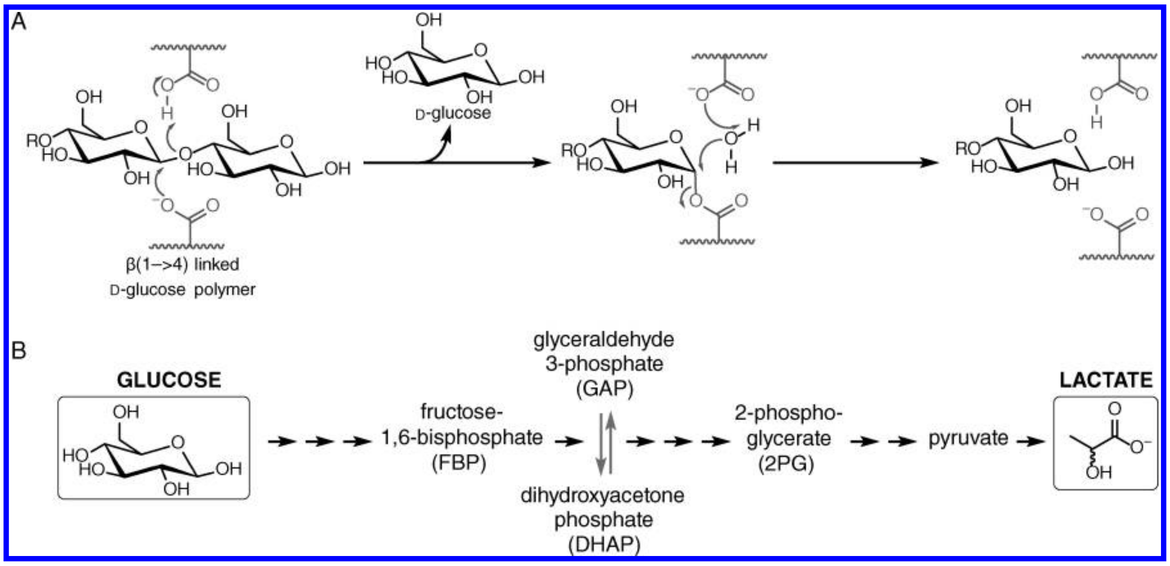


Figure 1. Biological transformations involved in the conversion of cellulose to lactate: (A) the general catalytic mechanism of cellulase enzymes; (B) the enzymatic transformations in *Lactobacillus casei* that convert glucose to lactate.

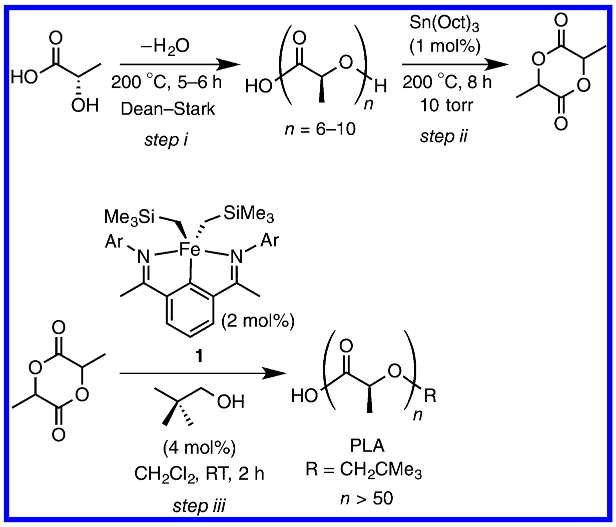
Aldrich), which utilizes a colorimetric readout to measure lactate concentrations.

This module exposes students to standard sterile techniques, by teaching them to culture *L. casei* in both solid agar plates and liquid media. They also learn about the concomitant action of a series of enzymes in a metabolic pathway that converts glucose into lactate (Figure 1B). These pathways are conserved in higher organisms and form a significant component of college level biochemistry courses, thereby providing students an early introduction to the topic of metabolism.

Module 4: Oligomerization

While self-condensation of lactic acid to PLA is a feasible route to PLA, in practice the high molecular weight polymer that is most useful for engineering polymers is unattainable through this route. Instead, lactic acid must be converted to the cyclic monomer lactide prior to polymerization. The first step in this process is a lactic acid oligomerization reaction, which is the first of three chemical transformations in the conversion of waste paper to PLA (step i, Scheme 2). It is also the students' first introduction to a chemical reaction, along with the prerequisite glassware and setup. During this procedure the students are introduced to the theory and design of a complex reaction setup including use of a heat source and specialized glassware such as a Dean–Stark trap (Figure S2 in the Supporting Information). In the absence of significant quantities of lactic acid from the fermentation step, reagent-grade lactic acid can be heated in a round-bottom flask to 200 °C for ~4.5 h. During this time the water produced from the condensation reaction of lactic acid collects in a Dean–Stark trap. The volume of water collected is proportional to the progress of the reaction and can be used to calculate the average oligomer chain length.[17] By monitoring the production of water over time, the students have the opportunity to learn about chemical kinetics. Additionally, this module introduces the students to modern spectroscopic techniques such as nuclear magnetic resonance (NMR) spectroscopy, which can be used to monitor the progress of the reaction and to evaluate its purity (Figure S3 in the Supporting Information).

Scheme 2. Chemical Transformations Involved in the Conversion of Lactate to Poly(lactic acid)



Module 5: Lactide Formation

The cyclization of oligomeric PLA used to produce lactide (step ii, Scheme 2) reinforces many of the skills that the students learned during the oligomerization reaction, while adding a new level of complexity. Prior research has found that higher yields of lactide are achievable by oligomerization of lactic acid prior to cyclization rather than from direct dimerization of lactic acid. The advantage of this procedure is presumably due to entropic reasons. In order to obtain high yields of lactide, the cyclization relies on a short-path distillation under reduced pressures that removes the lactide product from the reaction mixture and drives the equilibrium toward the desired product.[18] Under these conditions, the students learn how to design and plan a reaction setup to maximize control of parameters such as temperature and pressure (Figure S4 in the Supporting Information). The students also learn how to carry out distillation under reduced pressures. After distillation, the lactide product is obtained as a mixture of diastereomers (*rac* and *meso*) and is often contaminated with small amounts of

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linear lactic acid dimer. In order to carry out effective lactide polymerization, the lactide monomer must be very pure. Moreover, the *rac* lactide diastereomer leads to polymer with more desirable mechanical properties. Therefore, the students learn how to purify the *rac* lactide by crystallization of the crude distillate. The progress of this reaction is monitored by the volume of the distillate and later verified through ^{1}H NMR spectroscopy (Figure S5 in the Supporting Information). This module is particularly amenable for experimentation as the yield of the lactide is very sensitive to the reaction conditions. An instructive use of this module would be to have different groups carry out the lactide forming reaction under different pressures and temperatures. Yield and purity of the lactide can be used as parameters for optimization thereby illustrating to the students how systematic experimentation is used to find ideal conditions for a chemical reaction.

Module 6: Polymerization

The polymerization reaction is the final step in the conversion of used office paper to PLA (step iii, Scheme 2). New catalysts for the ring-opening polymerization of lactide are currently an active area of research.[20,21] Therefore, to emphasize the research aspect of the project, we decided to carry out the polymerization of lactide using an iron catalyst that was recently reported by one of us.[13] The polymerization reaction also allowed for the demonstration of many common and useful techniques in polymer and organometallic chemistry (Figure 2). The catalyst precursor is air and moisture sensitive, so the students were able to learn how to handle air sensitive reagents using equipment such as inert atmosphere glove boxes, solvent purification systems, and high vacuum lines. Formation of poly(lactic acid) was monitored by ^{1}H NMR spectroscopy (Figure S6 in the Supporting Information). Additionally, analysis of the reaction was carried out using gel permeation chromatography (GPC) to monitor polymer molecular weights and molecular weight distributions. For laboratories that are not equipped with such specialized equipment, there are numerous other air and moisture stable catalysts that have been developed for the polymerization of lactide.[20,21] Many of these catalysts, such as tin(II) 2-ethylhexanoate[22] or 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU),[23] are inexpensive and are commercially available. In the absence of gel permeation chromatography or other types of size exclusion chromatography, the progress of the polymerization reaction can easily be monitored by measuring the viscosity of the reaction mixture. Precipitation of the polymer in methanol solution also allows the students to analyze the macroscopic physical properties of the new material (e.g., T_m, T_g, etc.), which is very similar to many of the industrial plastics that they are more familiar with.

■ EXPERIMENTAL TIMELINE

This program was run over the course of 8 weeks during the 2013 summer (June–July) meeting twice weekly. By splitting into two sections, one meeting on Mondays and Wednesdays and the other meeting on Tuesdays and Thursdays, the schedule provided adequate time for fermentations, digests, and similar procedures to progress significantly between each class period (Table 2). Further streamlining these procedures is possible, which will likely allow for the program to be carried out over the course of 4 weeks instead of eight. Experiments range from 2 to 5 h.

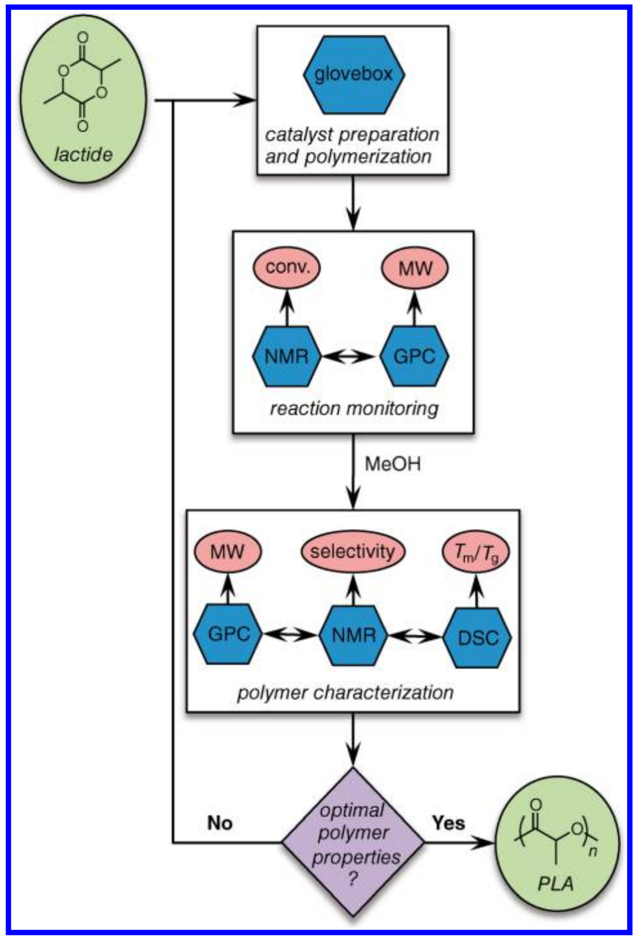

Figure 2. Flowchart highlighting the work flow and techniques in polymer and organometallic chemistry introduced during the polymerization step.

Table 2. Experimental Timeline

Session	Experiment
1	Safety Training and Paper Pulping
2, 3	Cellulase Digest[a]
4	Glucose Assay and Culture Preparation
5, 6	Fermentation[a]
7	Lactic Acid Assay
8	Lactic Acid Oligomerization
9	Lactide Formation Reaction
10	Poly(lactic acid) Formation

[a]Procedure takes more than 48 h to complete incubation steps.

■ EQUIPMENT

To carry out these experiments, the laboratory should contain basic organic glassware (round-bottom flasks, condensers, etc.) and biochemical equipment (e.g., pipets, culture plates, etc.), a Dean–Stark trap, a standard vacuum manifold with a vacuum pump and air outlets, a fume hood, an analytical balance, a drying oven, a shaking incubator, and a UV–visible spectrophotometer. An inexpensive kitchen blender can be purchased from any home goods store for module 1. Modules 4 and 5 require an NMR spectrometer for optional product analysis, but product identification can also be carried out using more classical techniques such as infrared spectroscopy, viscometry, and/or melting point determinations. However, a major goal of the project is to mimic genuine research

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conditions; using modern analytical techniques such as NMR spectroscopy emphasizes this goal and in our experience generates excitement among the high school students.

■ INTEGRATION INTO CURRICULUM

The P2P program is amenable to modification to suit a range of purposes. The modular nature of the program allows for much flexibility in the duration of the program.

High School

The primary goal of the P2P program is to expose high school students to multidisciplinary research at a higher-level research institution. This program gives the students access to instrumentation and resources that are not routinely available in high-school laboratories. To highlight the benefits of this exposure, we quote some of the student feedback we received from the program: "I thought that it was really wonderful to be able to work in a real lab, instead of doing uninteresting experiments at school", and "It was a great experience to use the high-tech equipment". These quotes attest to the value of the opportunity that we are providing these high school students and our successes at generating interest in scientific research at an early age.

In addition to being replicated as a summer program at a research university, P2P can be extended to an entire semester project for a high-school biology or chemistry class. While some of the techniques and instruments used are outside the means of many high school facilities, many of the procedures can be adapted to the available methods. These modules could also be applied individually as an after-school club or an independent project for an advanced student. To make this a more comprehensive project, students can focus on optimizing the procedures and varying the conditions to obtain the best yield in each step or investigating the synthesis and polymerization of similar cyclic diesters.

Undergraduate

The P2P program, as designed, provides undergraduate students with an ideal setting in which to mentor younger students, while honing their own research skills in a variety of research arenas. We recruited two junior undergraduate students with aspirations to pursue careers in teaching and scientific research to mentor the high school students over the course of the summer. This opportunity enabled the undergraduates to independently optimize the steps of each module prior to the arrival of the high school students, thereby providing them the opportunity to perform research and troubleshoot experiments with minimal supervision. Furthermore, the mentoring of the high school students involved dissecting fundamental concepts in biology and chemistry to terms that were approachable to the high school students, thereby providing a forum to practice their teaching and mentoring skills.

Outside of the mentoring benefits provided to undergraduates interested in a career in science education, the experiments presented herein would also be suitable for an undergraduate organized laboratory setting. This project could be integrated into the curriculum of an introductory biology or chemistry lab. Similarly, this could be implemented as an independent research project for an undergraduate student.

■ PROGRAM STATISTICS AND EVALUATION

The P2P program was implemented at Boston College over the course of 8 weeks during the summer of 2013 with 6 high school students attending, under the mentorship of 2 undergraduate students (a pilot program involving two high school students and one graduate student was also carried out in the summer of 2012). In order to maintain a research environment, we feel it important that the ratio of high school students to undergraduate students does not exceed 4:1. In 8 weeks, the high school students had ample time to successfully complete all modules of the program. This experience suggested that a condensed four-week schedule would be possible. With two one-month sessions meeting every other day during the summer, the program can theoretically accommodate 16 high school students with the aid of two undergraduate mentors.

The success of the program was evaluated with the use of an exit questionnaire (Supporting Information, Appendix I). This evaluation was designed to provide feedback on the following aspects of the program: (i) the effectiveness of the program in increasing the current knowledge base of the students in topical areas of chemistry and biology; (ii) the effectiveness of the program to create enthusiasm about careers in science; (iii) the efficacy of the program for promoting the value of interdisciplinary research. The feedback we received was overwhelmingly positive (Supporting Information, Appendix II), and the students stated that the program was enjoyable and educational and, importantly, encouraged them to pursue science in college.

We also used this questionnaire as an opportunity to obtain feedback to improve the program in subsequent years. Based on the comments we received, we aim to provide students with related side experiments to keep them occupied during any lag times in the main experimental workflow. These side experiments can include other polymer-related demonstrations, such as the synthesis of nylon.[24] As we expand the P2P program and increase annual student participation, we will continue to obtain feedback and continuously improve upon the general organization, workflow, and educational value of this outreach effort.

In addition to the program evaluation questionnaire, the long-term efficacy of the program will be evaluated by contacting past participants five years after their participation in the program (Supporting Information, Appendix III). The focus of this brief survey will be to evaluate the students' choice in major, their career directions, and the impact that the paper to plastics program has had on these decisions. At this stage it is too early to assess the long-term efficacy of the program, but we fully intend to publish these data as they become available along with a more comprehensive evaluation of the program's short-term efficacy in due course. To facilitate this effort, we encourage other institutions that adopt similar programs to utilize our program evaluation questionnaire (Supporting Information, Appendix I) and long-term evaluation survey (Supporting Information, Appendix III) so that a uniform evaluation mechanism is maintained. Completed evaluation forms can be sent to either of us via e-mail or regular mail so that they may be included with our own data. If desired, we guarantee full anonymity or full credit by inclusion in future publications.

■ CONCLUSIONS

In summary, the P2P program is a unique opportunity that intersects science, education, and society. It not only encourages students to think about scientific problems from a multidisciplinary perspective but also provides them with hands-on experience in a research environment. This first-hand research experience, coupled with its social implications, provides an exciting environment for the students to learn and grow. The P2P program is ideally suited to achieve the following goals: (i) introduce high school students to a genuine scientific research environment to encourage careers in science, engineering, and technology; (ii) demonstrate how scientific problems can be addressed effectively from a multidisciplinary approach; (iii) provide a concrete example of how science and technology can be used to address problems with social significance; (iv) provide an opportunity for high school students to interact with undergraduate students, graduate students, and professors in order to foster open discourse about pursuing higher education and the value of science degrees and careers in science and technology; and (v) develop leadership and mentoring skills among undergraduate students who are considering careers in science, engineering, and technology. We believe that the experimental modules described herein are highly versatile and easily incorporated into similar outreach programs, as well as current high school and undergraduate curriculums. By bridging the fields of chemistry and biology in a socially relevant research endeavor, we are confident that high school students will develop an early appreciation of the scientific discipline, which will serve to increase student participation in STEM fields as outlined in the objectives of the NAS.

■ ASSOCIATED CONTENT

Supporting Information

Detailed experimental protocols for each of the modules; project evaluation forms. This material is available via the Internet at http://pubs.acs.org.

■ AUTHOR INFORMATION

Corresponding Authors

*E-mail: eranthie@bc.edu.
*E-mail: jeffery.byers@bc.edu.

Notes

The authors declare no competing financial interest.

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