

NEW LABORATORY COURSE FOR SENIOR-LEVEL CHEMICAL ENGINEERING STUDENTS

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The career diversity for chemical engineers has changed dramatically over the last 30 years. For example, more than 75% of chemical engineering positions in 1975 were with companies involved in production of commodity fuels and chemicals.^[1] By 2003, only 25% of the careers for chemical engineers were in these industries,^[1] while biotech and electronics/materials industries employed approximately 15% and 10%, respectively, of chemical engineers.^[2] These trends are expected to continue with a growing emphasis on the development and production of more complex materials including biologically active and nanostructured materials.^[3]

The responsibilities of today's chemical engineer are evolving as a result of the changes in the industries that employ them. Chemical engineers are now more involved in the synthesis and development of new products and devices. Twenty-five years ago, only 15% of the graduating chemical engineers were in product development, whereas more than 50% of recent graduating chemical engineers are working in this area.^[3] Also, the need for chemical engineers to be able to effectively interact with scientists from a range of disciplines such as materials science, biology, and medicine is increasing as a result of evolving employment opportunities.^[4]

Even with the dramatic changes in career diversity and responsibilities for recent graduates, the chemical engineering curriculum has changed little over the last 40 years.^[3, 5] Much of the focus remains with large-scale process equipment such as distillation towers and heat exchangers, and many of the examples used in courses continue to come from the petroleum refining and bulk chemical production industries. A growing number of leaders in chemical engineering believe that

chemical engineers need to be taught more about product and process synthesis rather than large-scale chemical engineering equipment.^[1] Furthermore, it has been argued that more time needs to be spent in chemical engineering education on

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atomic- and molecular-scale phenomena, and on the translation of fundamental science to engineering principles.^[6]

Because of these changes and needs, the Chemical Engineering Department at the University of Virginia (UVa) decided to overhaul its senior-level laboratory course to provide students with experiences and opportunities to learn concepts and develop skills required for success in today's changing world. The objectives of this paper are to communicate the overall concept of the new laboratory course, provide an overview of each experiment, and describe student feedback from the course. Details about each experiment can be obtained by corresponding with the author.

The objectives of the new 3-credit-hour laboratory course, based on a full course load of 15 to 18 credit hours per semester, are to:

- ▶ *Provide students with experiences that are more relevant to the contemporary chemical engineer.*
- ▶ *Engage students in*
 - *integration of process steps.*
 - *relationships between molecular structure and macroscopic properties.*
 - *translation of fundamental science to engineering principles.*
- ▶ *Provide students with an opportunity to develop teamwork skills in an environment similar to industry.*

These objectives were accomplished by first developing three 4-week-long experiments in Bioprocess Engineering (protein synthesis and purification), Catalysis and Energy Conversion (catalytic production of hydrogen coupled to fuel cells), and Polymer Synthesis and Characterization (structure/property relationships of advanced materials). A class structure was then developed to reflect a real-world chemical engineering environment. Sharing of information and ideas was accomplished by having small teams of students work together as part of a larger team on each experiment. Knowledge and experimental results were communicated between the smaller sub-teams using different types of written reports and an oral presentation.

The three experiments were set up in a new laboratory facility in Wilsdorf Hall that opened in the fall of 2006. This state-of-the-art laboratory, dedicated to undergraduate chemical engineering, has over 2,000 square feet of space, walk-in hoods, and abundant natural light (Figure 1). The major investment in the new laboratory space, and the equipment required for the three new experiments, represent a

high-level commitment to undergraduate chemical engineering at UVa.

This space also is used for the junior-year laboratory course for chemical engineering students at UVa during the prior semester. The emphasis of this laboratory course is on more traditional unit operations experiments with heat exchangers, a distillation column, a fluid flow demonstrator, and equipment for agitation and mixing.

Moving into a new laboratory facility made it easier to implement the new senior-level course in a single semester. This also provided the opportunity to benchmark the new course to the old course of more traditional unit operation experiments including a gas absorption column and a fixed-bed reactor.

LABORATORY DESCRIPTION

Each of the three 4-week-long experiments is designed for division into three or four separate parts. Teams of six to eight students divide themselves into three or four sub-teams to work on the different parts of an experiment (two to three students per sub-team). A member of the teaching team is assigned to each experiment and is responsible for supervising the experiment and evaluating the students (teaching team consists of one faculty member and two graduate research assistants). Each teaching-team member spends 15-20 hours per week on the course, which includes two 4-hour lab periods/week, experiment preparation, grading, and office hours to answer questions.

The first week of each 4-week-long experiment is a planning period that is used by each teaching-team member to explain his or her experiment to a student team. The students also use this time to divide themselves into sub-teams and to become familiar with their part of the experiment. The final three weeks are used to run the experiments to accomplish the objectives of the experiment. The schedule of required



Figure 1. New 2,000-square-foot laboratory facility in Wilsdorf Hall for undergraduate chemical engineering at UVa.

reports and oral presentation for each experiment is shown in Table 1.

The total equipment cost for the three experiments was in excess of \$400,000. Major equipment costs for the Bioprocess Engineering experiment included the 5-liter fermenter (\$45,000), liquid chromatography workstation (\$62,000), and the ultra filtration apparatus (\$6,000). The major equipment costs for the Catalysis and Energy Conversion experiment were the plug flow reactor system (\$55,000), gas chromatograph (\$40,000), and fuel cell system (\$20,000). The two big expenditures for the Polymer Synthesis and Characterization experiment were for the dynamic mechanical analyzer (\$65,000) and the differential scanning calorimeter (\$55,000).

The summer prior to the first offering of the new laboratory course was spent by the instructor and five undergraduate students setting up equipment and working out the details of each experiment. Assistance was obtained during this time from other faculty members with expertise in the areas of the particular experiments. General course material, experimental procedures, and background information were prepared. Relevant journal articles and reference materials were placed on a Web site developed for the new course.

BIOPROCESS ENGINEERING

The Bioprocess Engineering experiment involves the production of recombinant green fluorescent protein (GFP) from genetically transformed *E. coli* cells.^[8] GFP is well-suited for use in this type of experiment for several reasons: its fluorescent nature allows students to detect its presence visually; the concentration of GFP can be measured in a protein mixture due to a unique absorbance peak at approximately 304 nm; and the extremely hydrophobic nature of GFP enables a straightforward purification strategy.^[9] Because of this, laboratory experiments for undergraduate chemical engineering students have been developed for the production and purification of GFP.^[10,11]

Information from published experiments with GFP has been used to develop an experiment that can be run in a 4-week time period with 4 hours/week of experimental time. A working cell bank of transformed cells, created by faculty and graduate students in preparation for this course, is used as inoculum. Students determine the effect of different process parameters on the growth of *E. coli* cells and protein expression using a 5-liter fermentation vessel (Figure 2a) in the upstream part of this experiment. Centrifugation, mechanical cell lyses, tangential flow ultra filtration (Figure 2b) and liquid chromatography (Figure 2c) are used in

End of Week	Required Report/Presentation	Comments
1	Planning Report	Team report with separate sub-team grades
2	Oral Presentation	Team presentation to teaching team member
3	Progress Report	Individually prepared report
4	Final Report	Team report that integrates work and results of each sub-team. A peer-evaluation process is used to adjust individual grades. ^[7]

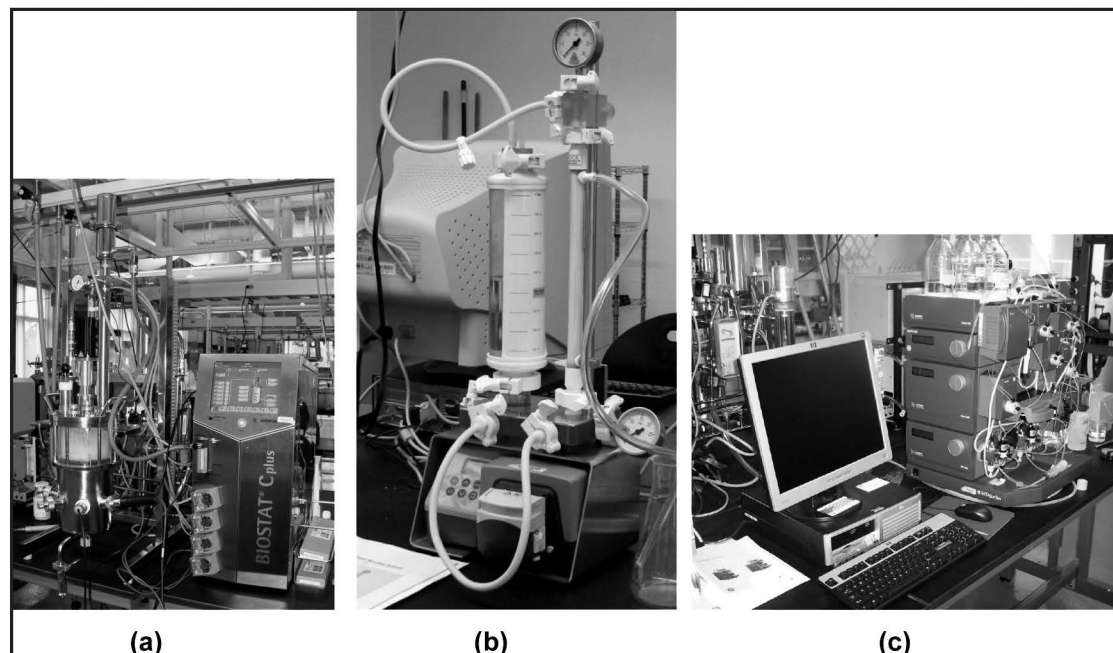
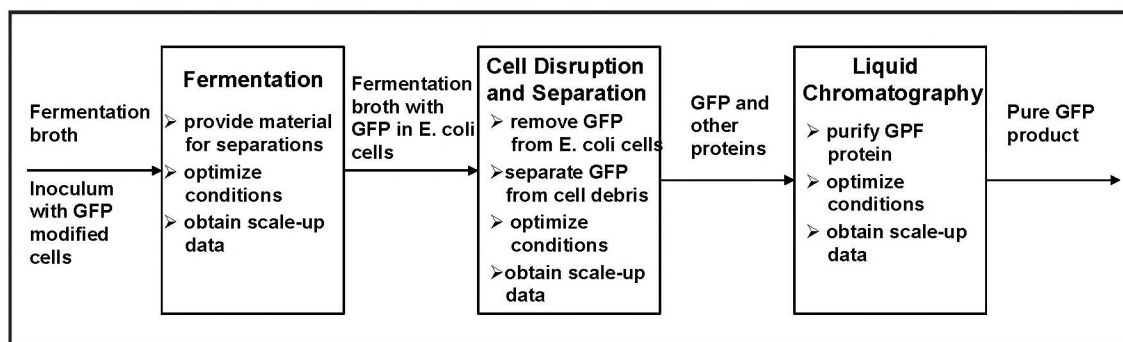


Figure 2. Photographs of the major pieces of equipment in the Bioprocess Engineering experiment including (a) 5-liter Sartorius BIOSTAT CPlus® fermenter, (b) GE Healthcare QuixStand® benchtop ultra filtration apparatus, and (c) AKTA Purifier® liquid chromatography workstation.

Figure 3. Block diagram of the BioProcess Engineering experiment with the flow of materials outside each box and sub-team objectives listed inside each box.



the downstream part to recover and purify the GFP product.

A block diagram of the Bioprocess Engineering experiment (Figure 3) illustrates the flow of material through the experiment and highlights the objectives of each major part of the experiment.

The combination of fermentation, cell disruption and separation, and liquid chromatography enables students to evaluate and understand the overall process used to make a protein product. This helped the students develop an appreciation that a successful process-development team must work both cooperatively and independently to develop an optimized, multi-step manufacturing process.

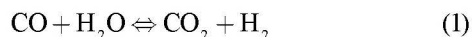
Representative examples of student feedback from this experiment are:

- ▶ “This experiment fit in very well with biotech courses and tied them together.”
- ▶ “Enjoyed seeing the process from fermentation through downstream processing to final purified product.”
- ▶ “Enjoyed applying my coursework to actual experimentation.”
- ▶ “I liked this experiment because it introduced me to the field of biochemical engineering without having to take the biochemical engineering electives.”

CATALYSIS AND ENERGY CONVERSION

The Catalysis and Energy Conversion experiment is motivated by an interest in hydrogen as an alternative fuel source and the potential technical and environmental advantages of a fuel cell to convert hydrogen’s chemical energy into electrical energy.^[12] The block diagram (Figure-4) shows the flow of material and the objectives of the major parts of this experiment.

Pure CO and H₂O are converted to CO₂ and H₂ over a copper alumina catalyst (BASE, Selectra Shift 4P+^[14]) in the reversible water-gas shift (WGS) reaction experiment shown in Eq (1).^[13]



The reaction is conducted in a fixed-bed reactor located in a BTRS-Jr reactor system (Autoclave Engineers). Liquid water

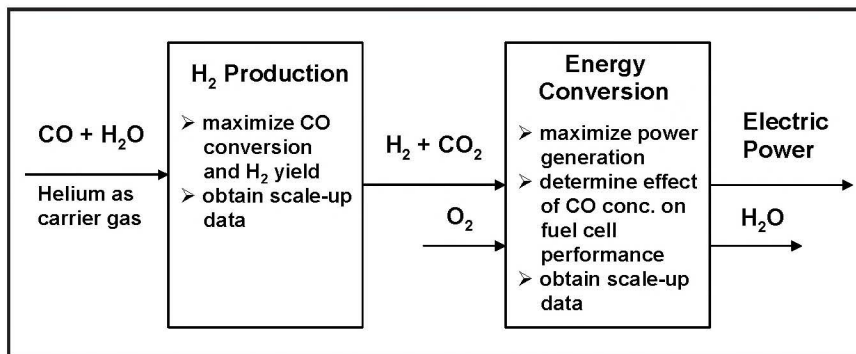
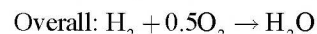
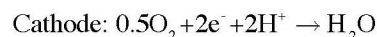
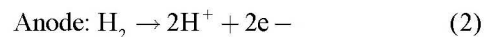


Figure 4. Block diagram of the Catalysis and Energy Conversion experiment with the flow of materials outside each box and sub-team objectives listed inside each box.

enters through an HPLC pump and is vaporized before being fed into the reactor. Helium is used as a carrier gas to minimize the temperature increase from the exothermic WGS reaction.

The concentration of CO and CO₂ in the reactor effluent is determined using an HP 6890 Series gas chromatograph. This information, along with the inlet flow rates to the reactor, is used by the students to determine the CO conversion and H₂ yield for a range of operating conditions. These data are used to identify the process conditions that result in the highest CO conversion and largest H₂ yield. Students also compare their experimental CO conversion values to their calculated equilibrium CO conversion values to determine whether the reaction is kinetically or thermodynamically limited.

A Nafion proton exchange membrane (PEM) fuel cell is used to convert the chemical energy in H₂ to electrical energy.^[15] Fuel (H₂) is fed into the anode side of a PEM fuel cell where it is converted into protons and electrons in the presence of the anode catalyst. The protons diffuse through the membrane and the electrons travel to the cathode through an external circuit. At the cathode catalyst, oxidant (O₂, either from air or pure O₂ gas) reacts with the protons and the electrons to form H₂O and heat. Eq. (2) is a summary of the reactions that occur on the anode and cathode sides of a PEM fuel cell.



The fuel cell part of this experiment is based on published laboratory experiments with PEM fuel cells.^[16,17] The temperature of the fuel cell is varied from room temperature to 80 °C. Pure H₂ or H₂ mixed with a small amount of CO (<100 ppm) is passed through a heated humidifier before being fed to the anode side of the fuel cell. The moisture maintains a high proton conductivity of the electrolyte membrane. Likewise, pure O₂ or air as oxidant is passed through a humidifier before being fed to the cathode side of the fuel cell. The flow rates of both streams to the fuel cell are controlled by digital mass flow controllers (OMEGA, model FMA6500).

An Agilent Electronic Load (model 6060B) is connected between the anode and cathode sides of the fuel cell. This instrument is used to vary the external load on the fuel cell from 0 Ω to 1,000 Ω and to measure the voltage and current of the fuel cell.

Figure 5 is a plot of fuel cell voltage as a function of current density at three different fuel cell temperatures. Each curve in this figure, which is referred to as a polarization curve, was obtained by varying the external load on the fuel cell over the range of 0 Ω to 1,000 Ω.

Students are able to determine the optimum conditions for operating the fuel cell by examining fuel cell performance over a range of temperatures, pressures, and gas flow rates. Fuel cell efficiency, defined as the electric power generated divided by the product of the rate of reactant utilization and its Higher Heating Value, is calculated under the optimum conditions. This information is used by the students to scale-up their results from a single fuel cell data to a fuel cell “stack,” *i.e.*, fuel cells connected in series, which is capable of producing enough energy for an average-size home.

Students observe that a CO concentration in H₂ as low as 10 ppm can significantly affect the performance of a PEM fuel cell, which illustrates why it is important for the WGS reaction to be run with a very high CO conversion value. In addition, they appreciate why the H₂ produced in the upstream part of this experiment as currently configured cannot be fed directly to the PEM fuel cell since the lowest CO concentration in the reactant stream leaving the reactor is approximately 20,000 ppm. A new type of fuel cell made by BASF, that can tolerate a CO concentration as high as 30,000 ppm, is currently being evaluated for use in this experiment.^[18]

Representative student comments from this experiment are:

- ▶ “I learned a great deal in this lab, having no previous experience with fuel cells.”
- ▶ “Enjoyed the real-life application with the fuel cell.”
- ▶ “I really liked the design problem.”
- ▶ “Enjoyed applying what we learned in class to real experimentation.”
- ▶ “Enjoyed learning about fuel cells and seeing the different factors that affect them.”
- ▶ “Reactor experiment was practical and related many of the basic chemical engineering concepts to practice.”

POLYMER SYNTHESIS AND CHARACTERIZATION

Polyurethane is a polymer that contains urethane linkages formed by the reaction of a diisocyanate containing two or more isocyanate groups (NCO) with a glycol molecule or a low-molecular-weight diol containing two or more hydroxyl groups (OH).^[19] Figure 6 includes structural representations of these different chemical groups where R and R' are two different carbon chains (R is usually aromatic, R' is usually aliphatic).

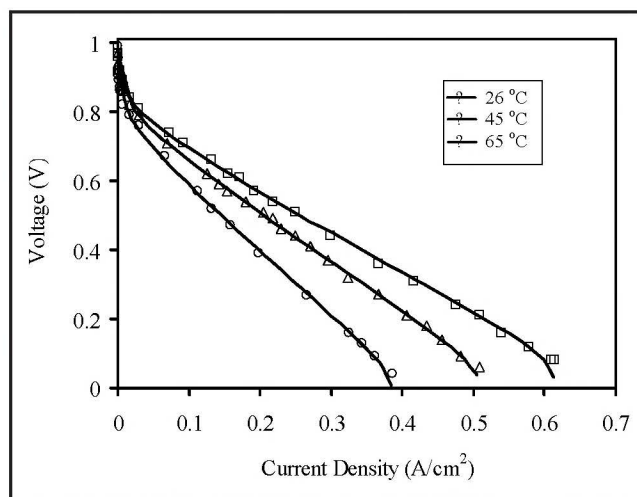
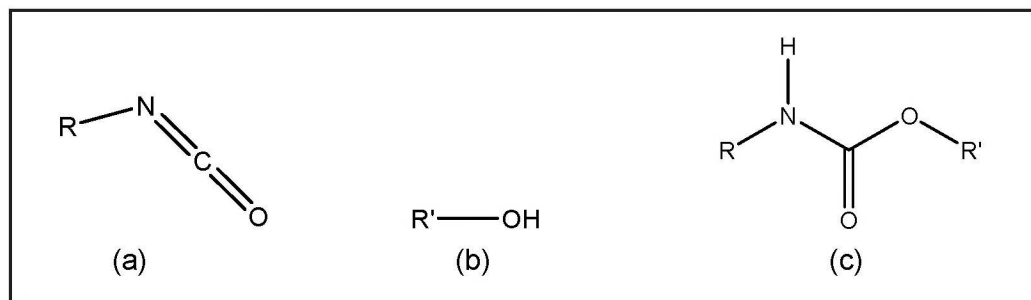


Figure 5. Effect of temperature on fuel cell performance. Experimental conditions: H₂ flow rate 30 ml/min, O₂ flow rate 30 ml/min, anode side pressure 10 psig, cathode side pressure 6 psig. Data obtained from student experimental measurements.

Figure 6. Structural representations of the (a) isocyanate and (b) hydroxyl reactive groups, and (c) urethane linkage.



Polyurethane polymer chains are composed of alternating “soft” segments and rigid “hard” segments. The soft segments are formed by the reaction of high-molecular glycol molecules with diisocyanate molecules. The number average molecular weight (M_n) of the glycol molecules used to make a polyurethane polymer is usually between 1000 and 2000 (*i.e.*, M_n of R' is between 1000 and 2000). A typical soft segment of a polyurethane polymer contains between two and four glycol molecules that are joined together with urethane linkages.

The hard segments are formed from the reaction of diisocyanate molecules with a low molecular weight diol, such as 1,4 butanediol, that is typically referred to as the chain extender. The hard and soft segments are joined end-to-end with urethane linkages. For this reason, polyurethane polymers are usually classified as block copolymers. A schematic representation of a polyurethane polymer chain is shown in Figure 7.

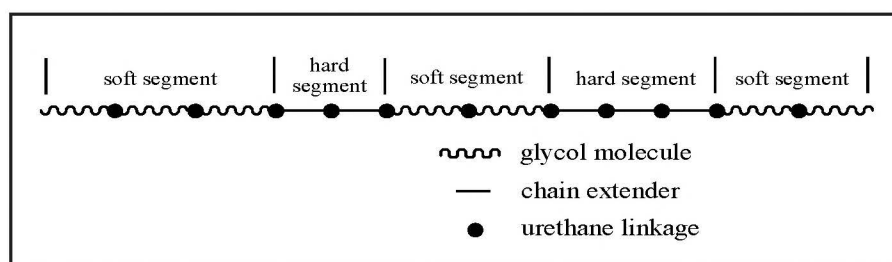


Figure 7. Schematic representation of a polyurethane polymer chain showing the soft segments (long chain glycol molecules connected with urethane groups) and hard segments (chain extender connected with urethane groups).

The students are given the assignment in the Polymer Synthesis and Characterization experiment to produce a soft, energy-absorbing polyurethane polymer for use in applications including the soles of shoes and personal protective equipment. This objective is accomplished by the students synthesizing polymers from several different prepolymers and chain extenders and then measuring the performance properties of the polymers. Figure 8 is a block diagram to illustrate the flow of materials and information through this experiment, and to summarize the objective of each of the four sub-teams.

The students synthesize polyurethane polymers by adding the appropriate amount of chain extender(s) to a prepolymer with a known mass and isocyanate concentration (%NCO) in a plastic cup. After mixing with a disposable stir-stick, the mixture is poured into heated sample molds and allowed to cure at 80 °C for 16 hours. The %NCO value of each prepolymer is measured by the students using a titration technique.

Four different polyurethane prepolymers are obtained from ITWC, Inc., for this experiment.^[20] These prepolymers are prepared by reacting polyether- or polyester-based glycols with an excess of 4,4'-Diphenylmethane diisocyanate (MDI). An excess of MDI is used so that unreacted diisocyanate molecules remain after all the glycol hydroxyl groups have been consumed. The amount of excess diisocyanate is quantified with a parameter referred to as %NCO, which is simply the wt% of NCO in the prepolymer (prepolymers used in this experiment have NCO values between 6 and 12 wt%). Based on the %NCO, the amount of chain

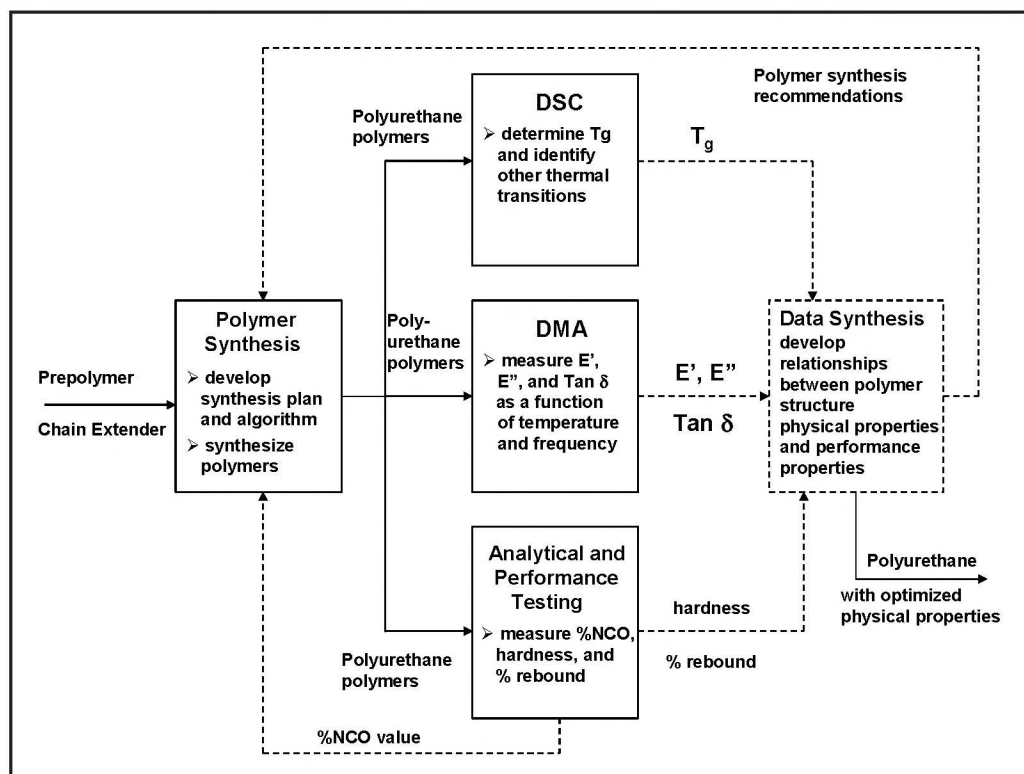


Figure 8. Block diagram of the Polymer Synthesis and Characterization experiment to illustrate the flow of materials (solid lines) and information (dotted lines) through this experiment. The objectives of each of the four sub-teams are listed inside each box. Data synthesis is accomplished by the four sub-teams working together.

extender required to react all the isocyanate groups to form the final polymer can be determined.

Students use two different chain extenders (1,4 butanediol and diethylene glycol) to react the NCO groups of the prepolymer to form the final polyurethane polymers. Synthesizing polyurethane polymers with different types of soft and hard segments, and with different hard-segment concentrations, enables the students to test polymers with a wide range of rheological properties and thermal transitions and to observe a range of performance properties.

A Shore SRI Resilometer, commonly referred to as a Bayshore Resilometer, is used to measure the % rebound of the polyurethane polymers following ASTM procedure D2632. Polymer hardness is measured using hand-held durometers obtained following ASTM procedure D2240. “A” and “D” scale durometers are used so that polymers with a wide range of hardness values can be characterized.

A TA Instruments Q1000 differential scanning calorimeter (DSC) is used to determine the effect of polyurethane composition changes on the glass transition temperature (T_g). DSC also is used to identify any other higher-temperature thermal transitions in these materials. The rheological properties of the polyurethane samples, including elastic (E') and viscous (E'') modulus, are measured as a function of temperature and frequency using a dynamic mechanical analyzer (DMA) manufactured by TA Instruments (Model Q800). The energy damping coefficient, $\tan \delta$, which is the ratio of E'' to E' , is calculated using the measured moduli values.

The students are challenged to use all of their measurements to develop an understanding of the relationships between molecular structure, rheological properties (*i.e.*, E' and $\tan \delta$), and performance properties (*i.e.*, % rebound and hardness) of their polyurethane samples. They are then expected to communicate their understanding to the polymer synthesis team to help decide which prepolymer and chain extender(s) to use in their second batch of polymers to obtain a material with the best combination of energy absorbing and hardness properties for the stated end-use of the material.

Representative student comments from this experiment are:

- ▶ “This lab really allowed for all the sub-teams to come together, and I thought that that was very helpful.”
- ▶ “Learning about the challenges of polymer synthesis was very interesting.”
- ▶ “Enjoyed how the sub-teams relied on feedback from other sub-teams throughout the experiment.”
- ▶ “Learned a great deal including the application of techniques and end-use tests.”
- ▶ “This experiment really gave us a good idea of how polymers are made.”

STUDENT FEEDBACK

Student feedback regarding the new laboratory course was obtained from the online course evaluation that is administered by the school and by a custom questionnaire that was administered at the end of the semester. The online course evaluation provided comparisons of student feedback from the old and new laboratory courses. The questionnaire provided an opportunity to obtain quantitative information about how well the objectives of the new laboratory course were met, how students enjoyed the organization of the new laboratory course, and how students felt about each of the three experiments that make up the new laboratory course.

The online course evaluation consists of 20 general questions about the course and the instructor. Students respond to each of the questions on a scale of 1 to 5 where 5 is the most favorable. Overall ratings for the course and instructor are calculated based on responses to the 20 questions by all the students. The overall course rating for the last two years of the old laboratory course was 4.03 and 4.05 (out of 5) indicating a good course rating. The overall course rating for the new laboratory course was 4.48 indicating a significant increase in course satisfaction by the students for the new course compared to the old course.

For the custom questionnaire, a response scale of 1 to 5 was provided for each statement, where 1 is strongly disagree and

TABLE 2
Summary of student responses to the statements that they were provided on the custom questionnaire about how well the course objectives were met

Statement	Strongly Agree	Agree	Neutral or Disagree
1. Collectively, the three 4-week-long experiments provided me with educational opportunities that are relevant to today's practicing chemical engineer.	68%	32%	0%
2. The three 4-week-long experiments taught me about the:			
a. integration of process steps.	36%	56%	8%
b. relationship between molecular structure and macroscopic properties.	16%	60%	24%
c. translation of fundamental science to engineering principles.	28%	68%	4%
3. Dividing my lab team into smaller sub-teams provided an opportunity to develop teamwork skills in an environment similar to what I might experience working in industry.	48%	44%	8%

5 is strongly agree. Students were also encouraged to add individual comments about each statement. Included in Table 2 is a summary of student responses to the three statements about how well the course objectives were met.

Responses to the first statement clearly indicate that the students felt the new laboratory course taught them about technologies that are relevant to the modern chemical engineer. Individual comments indicated that the students enjoyed discussing the details of the experiments during job interviews.

Responses to the second statement indicate that the students learned something about the integration of process steps and how material learned in basic science classes can be applied to engineering-related problems. The connection between molecular structure and macroscopic properties was not made apparent to a number of students, however. Even so, individual comments indicated that the students appreciated the different learning opportunities they were provided during the course (“*Learned more in this course than any other course that I have taken here.*”). Most students felt that the team/sub-team structure was challenging, but felt that this experience helped prepare them for what they will experience working in industry based on responses to the third statement and on individual comments included with this statement. In particular, students commented that they enjoyed the opportunity to be part of smaller sub-teams that worked together to achieve a common goal.

Feedback from the students indicated that a successful multi-week experiment does not depend on whether or not the students had prior coursework related to the subject of the experiment. For example, students who had not taken any bioengineering electives appreciated the opportunity to learn about the field during the Bioprocess Engineering experiment, while students working toward a bioengineering concentration enjoyed being able to apply their classroom knowledge to hands-on experiences. Furthermore, although an elective course in polymers is currently not offered at UVa, students were able to learn enough about the subject, and the details of polymer rheology and thermal transitions, to have a productive and enjoyable laboratory experience in polymers.

An overall observation by the instructor is that students stayed much more focused and engaged with the new laboratory course throughout the semester compared to students during the old laboratory course. It is the instructor’s belief that the primary reason for the change in student attitude and interest is that the students find the three 4-week experiments that make up the new laboratory course more interesting and relevant to what they might do as chemical engineers after graduation than the traditional unit operations experiments that were used in the old laboratory course.

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