CHEMICAL ENGINEERING SENIOR LABORATORY

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The University of Delaware (UD) Department of Chemical and Biomolecular Engineering (CBE) undergraduate laboratory courses prepare students for future careers in industry. Chemical Engineering Laboratory II, more commonly known as Senior lab, serves as a continuation to the Junior laboratory course at UD.[1] The Senior lab is offered to students in the Fall semester of their senior year and reinforces CBE fundamental theories, safety, technical writing, oral presentations, teamwork, leadership, and time-management skills. The course also emphasizes design, modeling, and cost analysis and exposes students to pilot plant scale equipment. Senior lab consists of three experiments focused on both traditional and novel unit operations such as the distillation of a methanol-water mixture, the production of biodiesel from vegetable oil, and protein fermentation and purification. This article is an overview of the Senior lab at UD and provides sufficient critical details about each of the three experiments that would be needed to implement comparable courses at other universities.

CHEMICAL ENGINEERING LABORATORY II

Students are given three options for completion of the Senior lab requirement, which include (1) participating in chemical engineering research if they meet certain GPA requirements, (2) taking part in a study-abroad program where they take an equivalent Senior lab course in Melbourne, Australia, or (3) completing the Senior lab course at UD. By allowing students to choose research activities in lieu of the planned experiments, the Senior lab helps students to make a decision on whether they want to pursue an advanced degree or a career in industry. About one in seven students select the research option, working with faculty on topics ranging from fuel cells to metabolic engineering, with the option of writing a thesis; these students often go on to attend graduate school. Students who choose to complete Senior lab may fulfill the requirements in a study abroad course at the University of Melbourne in Australia, accompanied by University of Delaware faculty members who teach and run the course on location. The remainder of the students (about 70%) complete the Senior lab course at UD.

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Senior lab at UD consists of three experiments: distillation (DIST), fermentation and purification (FERM), and biodiesel (BIOD). The DIST experiment introduces students to the operation of a pilot plant scale distillation column for separating a methanol-water mixture—reinforcing topics taught in the mass transfer course taken that same semester. The FERM experiment provides students the opportunity to grow green fluorescent protein in a bioreactor and purify the protein—introducing them to fermentation and biochemical separation processes used in the biotechnology industry. The BIOD experiment requires students to optimize an existing protocol for increased yield and purity of biodiesel produced from used vegetable oil for use in UD's biodiesel bus fleet—providing an introduction to alternative energy applications. Several excellent articles have been published describing chemical engineering labs similar in some ways to UD's Senior and Junior labs[1] including programs at the Technical University of Denmark, University of Wisconsin, University of Washington, Cornell University, Clemson University, and the University of Virginia, among others.[2-7] This article written primarily by students describes some unique aspects of UD's Senior lab that enrich their experience, providing as many details as possible for those wishing to create comparable courses at other universities.

The U.S. chemical industry is currently hiring a significant number of undergraduate engineers to work on the construction of new processes and expansion of existing refineries, particularly in the Gulf Coast region due to the availability of low-cost shale gas.[8] UD industry advisors have emphasized the importance for students to learn about both traditional unit operations, such as distillation, and newer processes, such as biodiesel and fermentation, to meet their current hiring needs. For this reason, all students complete the DIST experiment and then choose to run either the FERM or BIOD experiment. The UD CBE department dedicates significant resources to the Senior lab. A professor and graduate teaching assistant (TA) are assigned to each experiment. In addition, two laboratory technicians maintain the equipment and work with the faculty, TAs, and students to continuously improve the experiments. The educational outcomes, which also address the ABET learning objectives,[9] include:

1. *plan an optimum set of experiments that meet well-defined objectives*
2. *recognize and properly use laboratory safety procedures; identify major hazards in the experiment*
3. *collect data, analyze and interpret experimental measurements, and compare to existing theories*
4. *estimate error (uncertainty in measurements) and how it affects the final results,*
5. *design a process using data from experiments and/or theories*
6. *communicate results and conclusions effectively through both written reports and oral presentation*
7. *work effectively in teams by optimal distribution of workload, achieving common objectives within time constraints, and demonstrating leadership skills in a group context*
8. *reinforce chemical engineering fundamentals taught in lecture courses (such as heat and mass transfer, fluid mechanics, thermodynamics, and kinetics) via specific lab experiments*

Each experiment requires six weeks to complete and the students select one of the two experiments to present during the last week of the semester. Students have the option of presenting a traditional oral presentation or they can create an instructional video.

The nationwide trend of increasing enrollment in undergraduate chemical engineering programs presents a challenge for departments, which need to accommodate all students using existing equipment and space. In 2014, students interested in studying one process in depth or exploring new Senior lab projects to be offered in the future were given the opportunity to do so, leaving 12 groups of four students each, which were split such that six groups performed the DIST experiment and six groups performed the FERM experiment during the first cycle. The groups that performed the DIST experiment during the first cycle performed the BIOD experiment during the second cycle. The groups that performed the FERM experiment during the first cycle performed the DIST experiment during the second cycle. The schedule followed during the Fall of 2014 is shown in Table 1.

TABLE 1
Senior Lab Schedule – Fall 2014

Timeline	Group 1	Group 2
Weeks	Cycle 1	
1	Prelab Tour	Prelab Tour
2	Prelab Meeting	Prelab Meeting
3	DIST In-Lab	FERM In-Lab
4	DIST In-Lab	FERM In-Lab
5	Report Drafts	Report Drafts
6	Final Report	Final Report
	Cycle 2	
7	Prelab Tour	Prelab Tour
8	Prelab Meeting	Prelab Meeting
9	BIOD In-Lab	DIST In-Lab
10	BIOD In-Lab	DIST In-Lab
11	Report Drafts	Report Drafts
12	Final Report	Final Report
	Cycle 3	
13	Oral Presentation or Video	

The first week for each experiment features a prelab tour given by the TA, which allows students to become familiar with the laboratory equipment and safety procedures. Videos of the experiments that were created by students in previous years are available online for students to watch and see the equipment in operation.[10] We have found that students particularly enjoy both creating and watching these videos. Faculty present an introductory lecture and provide written documentation for students online about the experiment and the project objectives.[11] Additionally, a Senior lab handbook that outlines what is required for the course is provided online for students. During the second week, each group meets with the faculty member and TA in charge of the experiment for a 90-minute prelab meeting. Prior to the prelab meeting each group prepares a 30-minute oral presentation to demonstrate their familiarity with the experiment. The presentations include safety procedures, equipment diagrams, theory, preliminary models, design calculations, and how the group will conduct the experiment and operate the equipment. The prelab meetings provide plenty of time for students and faculty to ask questions and provide comments. The faculty members meet individually with all groups, which is a significant time commitment, but this is important one-on-one time with the students and ensures that they understand all of the safety precautions and are well prepared for the lab.

During the third and fourth weeks of each experimental cycle, the students perform experiments in the lab. Typically the experiments are scheduled Monday through Friday for four hours; however additional time as necessary is sometimes required. The FERM experiment takes almost an entire day for the fermentation step and several additional lab visits for the separation and purification parts of the experiment. Fortunately, students can work in pairs on the FERM experiment so that they are in lab for about five hours per week. The BIOD experiment takes about 20 lab hours total and students work in pairs for about five hours each week. The DIST experiment typically takes about four hours each week.

Students run the experiments and the TAs are always present during the laboratory experiment for safety and to address questions. The TAs grade each group's in-lab performance, focusing on safety, teamwork, experimental technique, time-management, and overall organization. If a group is unable to complete the experiment they can request and discuss with the faculty and TAs to get additional time in the lab. Faculty and TAs for the Senior lab course are available outside of lab to assist groups with data analysis, modeling, report writing, and other questions.

Labs are scheduled by group and day of the week. The students are given the option of running one of their labs on a weekend if the TA was available. Interestingly, a few of the groups did choose to run their experiment on a weekend. With the growing number of students entering the chemical engineering discipline and finite lab space, we have found that flexibility in scheduling is required.

The fifth and sixth weeks allow students to work together to analyze their data and write their reports. The average report consists of approximately 20 pages with a supplemental appendix. Each professor stresses various aspects of the report during the prelab meeting so students must pay close attention to the details. Additionally, professors provide an example report online for students to use as a guide. Lecture slides are also available from a report-writing training session students were given in the Junior lab course. Students may submit a draft of their report on the fifth week to the TA(s), who can provide comments on data analysis and report writing. The final report is submitted to the respective professor for grading during the sixth week. Professors strive to return the graded lab reports within one week so that the students have their comments before they begin their next experiment.

At the end of the semester, students either create an instructional laboratory video or give a professional oral presentation. The videos are presented during a video night at the end of the semester with the best videos receiving awards as well as being used to help instruct the next year's students on the safety and operation of the equipment.[3] The oral presentation is given in a television studio at UD in front of other students, the Senior lab professors, and the TAs. All presenters have access to an "Oral Fellow," a senior communications-major student, to assist them in the design and delivery of the presentation. Following a period of questions after the presentation, the students are provided a DVD copy of their presentation, allowing an opportunity for student self-assessment. The final assignment for each group is to write a critique of both their oratory skills and overall presentation quality.

An important objective of the Senior lab course is to give students experience in planning and executing experiments and operating pilot plant scale equipment safely. The prelab meeting, in-lab experiments, data analysis, final report, and presentation require students to effectively communicate with each other as well as with their TA and professor. Each of the four students per group performs every aspect of the in-lab experiment. Division of labor of data analysis and report writing is often discussed and managed within each group. Three of the students in each group also get the opportunity to lead one of the experiments or the oral/video presentation. Time management is essential in completing all assignments on time. These experiences are critical for preparing students for a career in industry. The three Senior lab experiments are described in detail in the following sections.

DISTILLATION (DIST)

The distillation experiment teaches students how to apply the theory from coursework with the operation of a pilot plant scale distillation column. Students are taught basic distillation principles and how to run ASPEN Plus® simulations[12] in CHEG325, Chemical Engineering Thermodynamics II, taught in the Spring semester of their sophomore year.

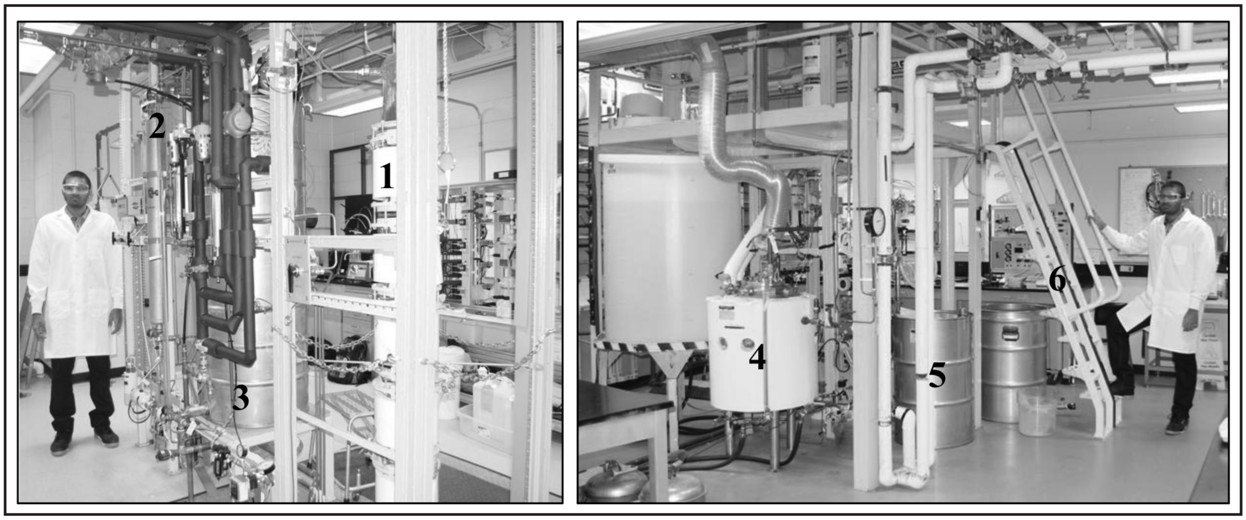


Figure 1. *Distillation experiment: image on left is the top floor where 1 is the distillation column, 2 is the product drum, 3 is the feed drum; image on right is the bottom floor where 4 is the reboiler, 5 is the bottoms drum, and 6 is the ladder for accessing the platform for taking samples from trays 1 to 5. Student pictured is Chirag Mevawala.*

Advanced distillation theory is taught concurrent to Senior lab in CHEG443, Mass Transfer Operations.

In 2014, each group had to determine the proper operating conditions to separate a 30 wt. % methanol (MeOH) and 70 wt. % water mixture into a distillate stream that contained at least 97 wt. % MeOH and a bottoms stream that contained less than 750 parts per million by weight (ppmw) of MeOH. Each year, the feed and product concentrations change making it more difficult to reach either the top or bottom specifications. The problem statement poses that the students work for a pharmaceutical company that has accumulated several drums of MeOH/H_2O waste and the EPA will cite the company if they cannot reduce the inventory of waste in a specified amount of time. The groups work on determining the most economical method for disposal of the waste, which includes using the existing distillation column or modifying the distillation column and comparing the costs versus hiring an outside waste disposal company. Most groups propose to sell the MeOH for a profit in order to recover some of the operating expenses. The bulk selling price for MeOH is a function of purity, so students can make a tradeoff between producing MeOH with a higher purity versus not meeting the bottom specification and incurring additional disposal costs. Students must consider all aspects of operating procedures such as startup, batch versus continuous distillation, and shutdown. In addition, distillation theory, ASPEN Plus® modeling,[12] process hazards analysis (PHA), and operating and capital costs are all critically important and should be included in the final report. This scenario allows the groups to answer the following questions: (1) Will the existing column meet the purity specifications and how long will it take to dispose of the accumulated waste?; (2) Can the column be modified to meet the deadline and what are the associated costs?; (3) What will be the profit from selling the purified MeOH?

During week one, each group tours the glass two-story distillation column shown in Figure 1. The groups develop their own operating procedure, a process flow diagram (PFD), and a piping and instrumentation diagram (P&ID) to help them learn how to safely and efficiently run the DIST experiment. Aspects of control of reboilers and condensers is discussed concurrent to Senior lab in CHEG401, Process Dynamics and Control. Students also conduct a rigorous hazards analysis and as much as half of the 90-minute prelab meeting is dedicated to discussing the hazards associated with operating the DIST experiment and conducting a PHA. Training students how to safely operate laboratory to pilot plant scale equipment in both Junior and Senior lab at UD is one of the most important functions of the lab. For the DIST experiment, students must fully understand the major hazards such as chemical toxicity and flammability of MeOH, the high temperature and pressure of the steam for heating the reboiler, handling sharp materials such as syringes, using electrical and pneumatic control systems, and the potential for slips, trips, and falls. In addition, groups must perform an in-depth "what-if" analysis for the prelab meeting that involves developing procedures for handling unexpected situations that could occur while in the lab. Such situations include, but are not limited to, power outages, methanol leaks in the column, and chemical spills. Only after students completely understand the equipment setup, operating instructions, and safety procedures for this lab are they permitted to begin the actual distillation experiment.

The column contains 12 sieve trays beginning with tray 1 at the bottom of the column and each tray is spaced 0.3048 m (1.0 ft.) apart with 0.3175 cm (1/8 in.) holes in a 0.9525 cm (3/8 in.) triangular pitch. The weir height is 5.080 cm (2 in.) and the column has a 15.25 cm (6 in.) internal diameter. A unique feature of the distillation column is that the sections between each sieve tray are made of Pyrex® glass. Each sec-

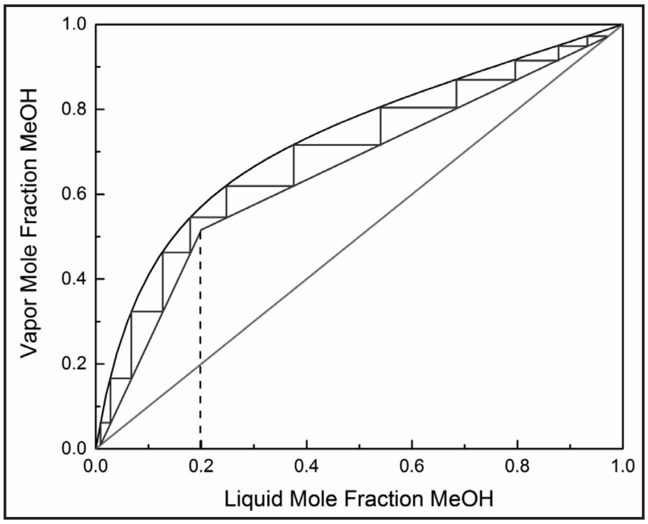


Figure 2. *McCabe-Thiele diagram used for estimating the minimum number of stages (12) and optimal feed stage location (5) required for the separation of a 19.4 mol. % (30.0 wt. %) MeOH and water mixture. Vapor-liquid equilibrium curve generated using ASPEN Plus® database and the NRTL-RK model with pressure = 1.013 bar.*

tion has removable insulation so that students can view and video record the vapor-liquid mixing on each tray. Students are also encouraged to operate the column under conditions that simulate common problems such as weeping, entrainment, and flooding, all of which will affect column efficiency and product purity. The feed stage can enter at tray 4, 5, or 6 and the column contains a total condenser and a partial reboiler. A LabVIEW® data acquisition program is used to control steam flow rate, reflux ratio, and the flow rates for the feed, distillate, reflux, and bottoms pumps.[13] The program also monitors the temperature on each stage, the total pressure inside the column, the differential pressure across the length of the column, the levels in the feed, product, and bottom tanks, and the MeOH concentration in the reflux and product streams using an in-line densitometer. The DIST column can run in either batch mode (partial or total reflux) or continuous mode. The students take samples from each of the trays, reboiler, and collection tanks and use a bench-top densitometer to measure the solution density that can be converted to a MeOH concentration.

Students should start by using the McCabe-Thiele method to estimate the minimum number of stages and the Gilliland correlation to calculate the actual number of stages as shown in Figure 2 for their prelab preparation.[14] Students are also encouraged to begin using ASPEN Plus® during their prelab preparation. For example, students use the ASPEN Plus® DSTWU module for calculating the minimum reflux ratio and RADFRAC to obtain the initial flow rate.[12]

The "in-lab" portion of this experiment can be completed in two labs that are approximately four hours each. During the first in-lab period in the third week of the cycle, students focus on becoming familiar with the operation of a distillation column and running batch experiments in total and partial reflux. Students record a temperature and composition profile for each tray in the column and use the compositions to determine Murphree tray efficiencies and the overall column efficiency.[14] Column pressure and pressure drop as well as steam and cooling flow rates are also monitored. Groups use this information to develop a more rigorous ASPEN Plus® model using the RADFRAC module.[12] This model can be used for optimizing the reflux ratio, feed-to-product flow rates, and steam input necessary to meet the purity specifications. The second in-lab period in week four allows students to validate their ASPEN Plus® model parameters to maximize feed flow rate, while still meeting the purity objectives. Generally, the ASPEN Plus® calculations agree well with the experimental data. For example, nonlinear regression can be used in ASPEN Plus® to calculate the tray efficiencies so that the temperature and composition tray profiles will match the experimental data, as shown in Figure 3. Students also use ASPEN Plus® to make predictions and design commercial-scale columns. A series of tutorial videos is provided online to help students with their ASPEN Plus® simulations.[10]

An accurate energy and material balance around the condenser, column, and reboiler can also be evaluated. Some

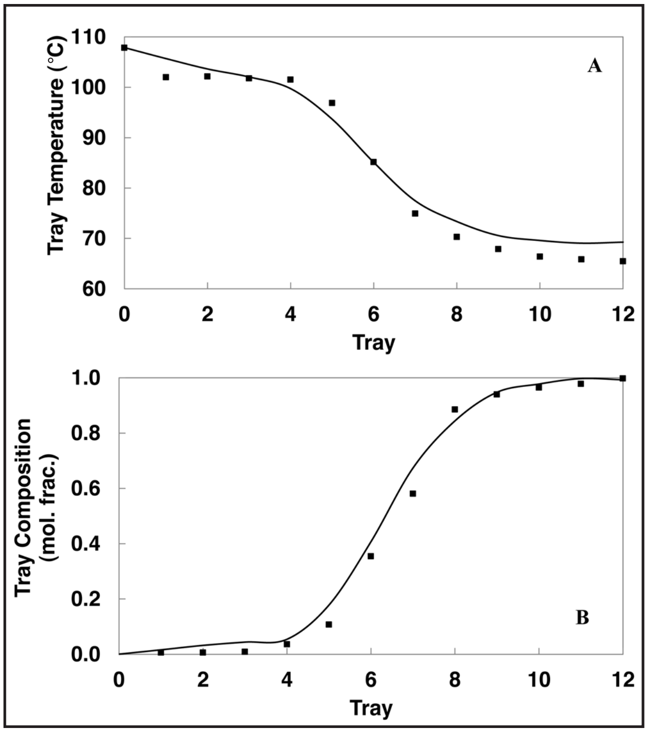


Figure 3. *Temperature (A) and composition (B) profiles across the distillation column with reboiler represented by tray 0 and the top of the column represented by tray 12. Symbols: black squares represent experimental data, solid curve represents ASPEN Plus® RADFRAC model.*

***Figure 4.** Bioreactor (New Brunswick Bioflo reactor, model 115)[16] where 1 is the dual flat blade impeller, 2 is the head plate which holds the pH, temperature and dissolved oxygen probes, 3 is the impeller motor, and 4 is the air sparger.*

groups have been able to achieve an energy balance of almost 100%, which is incredible! This is in part possible because UD provides two technicians to maintain and calibrate the instrumentation used for both the Senior and Junior labs.

Groups are encouraged to be creative when solving the DIST problem and often present several options in their final report and oral/video presentation to meet the product specifications and minimize the disposal cost for the MeOH. Such options have included using the current column, modifying the existing column, building a new column, and hiring an outside company to remove the waste drums. All of these solutions must include a detailed cost analysis, which reinforces for students the reality of their value propositions. Overall, the distillation experiment teaches students about a common industrial process that many students may encounter at some point in their chemical engineering careers.

FERMENTATION (FERM)

As the biotechnology industry rapidly expands, the CBE faculty at UD has recognized the importance of incorporating biomolecular engineering concepts into its curriculum including the introduction of the fermentation (FERM) experiment in the Senior lab course.[7] In the FERM experiment, students produce and purify a green fluorescent protein (GFP).[15] The experiment is comprised of four parts: fermentation of cells, broth filtration, cell lysing, and GFP protein purification. The organism used in this experiment is a strain of Escherichia coli (*E. coli*) genetically modified to produce the GFP.

During week one, the students tour the FERM experiment with the TA and during week two, the students meet with the faculty member and TA for their prelab meeting. Similar to the DIST experiment, the students prepare a 30-minute oral presentation for the faculty member and use the remaining hour to ask and answer questions about the FERM lab. On week three, or the first "in-lab" week, students learn how to operate the bioreactor shown in Figure 4.

The reactor is connected to a control system that can regulate the reactor temperature, O_2 concentration, pH, and agitator speed. The temperature is controlled using a band heater on the outside of the 3 liter glass reactor, the O_2 concentration is regulated by controlling the air flow rate, the pH is regulated by the automatic addition of either hydrochloric acid or sodium hydroxide, and the agitator speed is varied using a variable frequency motor.

Students are provided a variety of techniques for scaling the bioreactor.[17] These techniques range in difficulty from maintaining similar reactor geometry to constant mixing time. Most students find that maintaining a constant volumetric oxygen transfer coefficient, $k_L a$, is the most feasible method for scaling the bioreactor and use the following scaling factor, Φ_R, shown in Eq. (1):

$$\Phi_R = \frac{k}{k_L a} = \left(\frac{P_G}{V_R}\right)^{0.4} (v_s)^{0.5} (N)^{0.5} \tag{1}$$

where k is an empirical constant, P_G is the gassed power, V_R is the reactor volume, v_s is the superficial fluid velocity, and N is the rotational speed of the impeller. This allows students to replicate the commercial scale environment in the laboratory scale reactor. During the first week in lab, students investigate how $k_L a$ is affected by the air flow rate (Q) and the agitator speed (N). The scaled model of the bioreactor is significantly affected by these two operating parameters. Some groups write MATLAB® programs[18] to calculate feasible laboratory scale conditions from the $k_L a$, air flow rate, and agitator speed. A comparison of parameters for the lab-scale bioreactor and a commercial-scale bioreactor determined by a group in 2013 is shown in Table 2.

The goal of the first lab session is to grow the *E. coli* bacterium modified to express the GFP. The bioreactor is charged with 1.5 liters of growth medium (Sigma-Aldrich M9 growth

TABLE 2

Comparison of commercial scale and laboratory scale bioreactor parameters: Φ_R is the scaling parameter, Q is the air flow rate, N is the impeller speed, U is the tip speed, P_G/V_R is power per unit volume

Reactor Parameter	Lab Reactor	Commercial Reactor
Volume (L)	3	3000
Φ_R	292.8	293.2
Q (lpm)	2	5900
N (rpm)	275	75
U (cm/s)	471	1230
P_G/V_R (kW/m^3)	37.5	2.2

medium with minimal salts).[19] The pH, temperature, and dissolved O_2 probes are then inserted into the reactor and the seed culture of *E. coli* is introduced along with a food source (Sigma-Aldrich D-(+)-glucose solution)[20] for the cells.

The experiment runs for eight hours during which students are able to collect pH, temperature, and dissolved O_2 data from the computer at fixed air flow rates and agitator speeds. Students work in pairs during the first lab so that each student is in the lab for only four hours. The bioreactor is also set up so that students can take samples to check the glucose and cell concentrations. Eight samples are collected (approximately one per hour) and analyzed using optical spectroscopy. The optical density (OD_{600}) at 600 nm is measured and used to calculate the biomass concentration using Eq. (2). The remaining broth is stored at -20 °C for the next lab period during the following week.

$$C_{\text{biomass}} = \frac{1}{3} OD_{600} \tag{2}$$

During the second lab period in week four, the students analyze the broth produced from the fermentation for GFP. The *E. coli* cells now contain the GFP and have to be lysed such that the protein can be recovered. First, the broth is filtered to remove any salts and excess water using a Spectrum KrosFlo Research II Tangential Flow Filtration System.[21] The broth is run through the filtration system twice in order to concentrate the *E. coli* cells. After filtration, the solution is passed through a homogenizer operating at 16,000 psig, which generates a shear flow across a letdown nozzle that will lyse the cells and release the proteins. Once the cells are lysed, the solution is filtered again to separate the cell remnants from the released protein. Finally, the GFP molecules are separated from the remaining proteins using ion exchange chromatography (GE AKTA Purifier with a Q Sepharose matrix and fractionator). The solution elutes through the ion exchange chromatogram and is separated into fractions based on the charge. Examining each fraction under an ultraviolet light allows students to determine which samples are rich in GFP due to the fluorescence produced.

By performing the FERM experiment, students are able to effectively study the process of protein synthesis, isolation, and purification. Students also learn how to effectively scale up a laboratory process to a commercial scale. Through the use of computational programs such as MATLAB®[18] students are able to model the fermentation based on Michaelis-Menten kinetics to compare laboratory results with industrial scale data. Parameters such as pH, temperature, oxygen, and glucose concentration must be carefully controlled in order to achieve high GFP protein yield. The FERM lab provides students with an appreciation for the similarities and differences between chemical and biochemical processes and many of the important concepts in the rapidly growing biochemical industry.

BIODIESEL (BIOD)

Biodiesel is a renewable fuel manufactured from vegetable oils, used restaurant oils, animal fats, and greases. The availability and demand for biofuels such as biodiesel is increasing in some world regions with appropriate raw materials available at sufficiently low prices. Biodiesel must be produced to meet industry standards and continual improvement of production processes is necessary for firms to remain competitive. Students should have an understanding of the variety of alternative energy options including biofuel production; therefore, the CBE department at UD has recently introduced the BIOD experiment into the Senior lab course. The BIOD experiment produces high-quality biodiesel in a 200 L pilot plant scale batch reactor using used and recovered cooking oil.

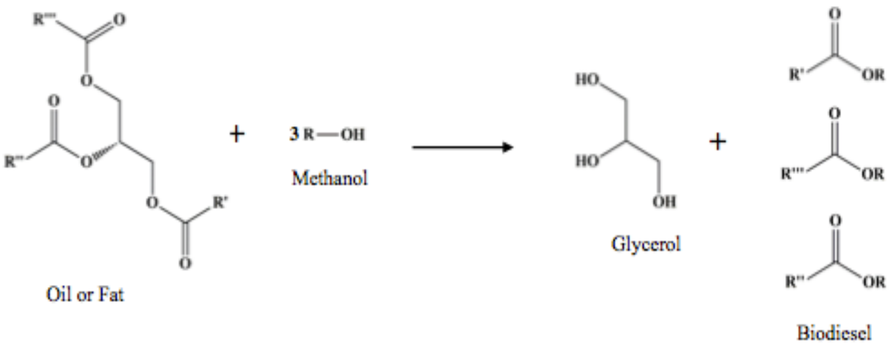
To give the students an opportunity to solve industrially relevant problems, the students are given a set of protocols and process parameters that produce suboptimal yields and purity. Key shortcomings of the existing protocol are insufficient reaction time and the formation of an emulsion during the liquid-liquid extraction step. Students are challenged to analyze the existing process and implement changes to improve yield while maintaining a product quality consistent with industrial standards. Thus the students are introduced to real world, open-ended problems that do not have a predetermined solution. This aspect of the experiment differentiates the UD course from that offered by other universities.[22-25]

During the first week, students are required to meet with the TA and tour the facility. Students are provided with detailed safety procedures and an operating and troubleshooting guide. Training videos are also available for students to watch how students from the previous year operated the equipment.[10] In the second week, the students conduct a PHA, prepare their operational and experimental plans, and give a 30-minute presentation to the faculty member in charge of the experiment during the prelab meeting.

The lab portion of the BIOD experiment is conducted during weeks three and four. During the first lab, students become familiar with how to operate the newly upgraded biodiesel processor, shown in Figure 5 (p. 139). Dr. James Seferis, a UD alumnus from the GloCal Network Corporation, generously donated the biodiesel processor used in this experiment. The equipment is located in a separate building specifically designed to handle flammable solvents with the appropriate electrical classification and ventilation systems.

The general procedure for producing biodiesel in the BIOD experiment starts with preheating approximately 91 kg of pre-treated cooking oil or soybean oil, depending on availability, in a drum to 65 °C using a heating jacket. This procedure is started the evening before the lab because it typically takes 18 hours to reach the desired temperature. The preheating procedure reduces the viscosity of the oil making it easier to pump from the drum into the batch reactor using a reciprocating pump (GPI model 1115s explosion-proof pump). During the first day in lab, students add 0.7 wt. % of potassium hydroxide (KOH) flakes relative to the mass of oil in the catalyst tank. Special handling is required while working with KOH flakes. Students must wear the proper personal protective equipment, which includes a dust mask to prevent inhalation of any KOH powder. KOH is used as the catalyst because it: (1) requires a lower reaction temperature (60 °C) and pressure (1 atm), (2) produces a high purity (98 wt. %) biodiesel product in a short period of time (< 4 hours), (3) it directly produces biodiesel without any intermediate steps, and (4) stainless steel can be used for the materials of construction.

The next step is to pump methanol from 55 gallon storage drums into the methanol tank using a hand pump. A sight glass on the tank is used to determine the proper level based on a 5:1 oil to methanol molar ratio. Next, the methanol is transferred to the catalyst tank and mixed with the KOH. A hand pump is used to circulate the solution until all of the solid KOH has dissolved. Due to risks involved with methanol, all power to the equipment is turned off while manually pumping methanol into the methanol and catalyst tanks. Once the soybean oil has been heated to 65 °C, which is slightly above the reaction temperature of 60 °C, the oil is pumped into the reactor. The methanol/KOH solution is then added to the reactor and the reaction begins. An agitator inside the reactor keeps the reactants well mixed and provides proper heat transfer. The transesterification reaction between soybean oil (triglyceride) and methanol produces methyl esters (biodiesel) and the by-product glycerol as shown in Eq. (3).



(3)

The reaction time can vary from 1 to 8 hours depending on the initial concentrations and temperature. After about 1 hour, samples are analyzed with an Agilent gas chromatograph (GC)[26] to calculate conversion. Dr. James McCurry from Agilent Technologies generously donated his time to set up the GC used in this experiment. In most cases the reaction is complete in 1 to 2 hours. If the desired conversion is achieved, the agitator is turned off and the glycerol is allowed to settle to the bottom of the tank. After about 30 minutes, glycerol is drawn off from the bottom of the reactor tank into a holding vessel and transferred into a vacuum tank using a vacuum pump. Additional methanol and catalyst maybe added if the extent of reaction is insufficient.

Once separated from the glycerol, the biodiesel is transferred to a wash tank using an air-driven pump. The wash tank is equipped with a water sprayer that sprays water at 65 °C at the top of the tank. A flow meter is used to keep track of the total volume of water sprayed into the wash tank. Once the predetermined amount of water has been sprayed, the water sprayer is shut off and the contents of the tank are allowed to separate into two phases. As the hydrophilic layer (water-rich) settles to the bottom, it is drained off by gravity from the bottom of the wash tank and the washing step is repeated until the biodiesel product is at pH of 6 ± 0.5. It typically takes between two to five washes with approximately 50 liters of water to reach the desired pH. If the reaction is carried out as described in the unmodified protocol an emulsion forms, decreasing yield significantly, as product in the emulsion cannot be recovered. Students are warned of this problem and challenged to identify and rectify the cause(s), which may include incomplete reaction of the oil or formation of an emulsifying side-product. Finally, the biodiesel is drained out of the wash tank by gravity and stored in plastic drums. The drums are weighed to calculate the overall yield of the reaction.

The maximum yield achievable for this experiment is 98 mass % and students usually achieve a high yield of more than 85 mass % in the first lab. Students use the GC to analyze the final product for the total quantity of glycerides and free glycerol. A Sandy Brae water kit is used to determine the water content. Students are required to meet the ASTM D6751-07a specifications of ≤ 0.02 mass % of free glycerin, ≤ 0.24 mass % of total glycerin, and ≤ 0.05 vol. % water content in the final product.[27,28] Product of insufficient quality must be blended with the product of another team to reach specifications, if possible, or otherwise returned to the reactor for a polishing run.

The first lab takes approximately 10 hours; therefore students work in groups of two to reduce the workload to about 5 hours per student for the first lab. In the second lab during the fourth week, students use their knowledge and results obtained in the first lab to make changes to their operating procedures in order to maximize the reaction yield.

The BIOD experiment is a great way for students to apply their knowledge of chemical kinetics and liquid-liquid extraction to a practical industrial process. The experiment also provides students the opportunity to understand how a pilot scale data acquisition and control system operates. This experiment is the most time intensive experiment in the Senior lab and requires a total of 20 hours to complete; however, students can work in teams of two over a two-week period so each student spends about 10 hours in lab. This requires students to manage time and workload effectively, which is another important aspect of Senior lab.

CONCLUSION

The Senior lab at UD has continuously improved over the past five years due to recommendations based on student feedback. Each year students are asked to fill out a course evaluation following completion of the course and provide their ideas for improvements. These reviews are read carefully by the faculty and TAs and taken seriously for improving the Senior lab. The reviews have recently led to the ability to run the DIST experiment under continuous flow conditions and the addition of the BIOD experiment to the Senior lab. In addition, the CBE faculty are discussing how to shorten the time required for the FERM and BIOD experiments. One concept is to allow groups to work together. For example in the FERM experiment, the Monday group would produce the GFP and then provide their solution to the Tuesday group who would isolate, lyse, and separate the protein. This would not only shorten the in-lab time, but require groups to work with each other. Additional time in the FERM and BIOD labs would allow other experiments to be considered such as the production and isolation of a second protein or contrasting the differences between producing BIOD from both used and pure vegetable oil. During the winter and summer sessions several students have asked to help the faculty implement new experiments, upgrade the equipment, and modify and improve documentation. The fact that students volunteer to work with faculty each year to improve both the Junior and Senior labs suggests that they are truly interested in what they learn and want to continue to improve the experience for the next class of students.

Figure 5. *Pilot plant scale biodiesel processor, where 1 is the wash tank, 2 is the catalyst recovery tank, 3 is the reactor and 4 is the methanol recovery tank; catalyst tank and methanol tank are located on the back side of the processor (not shown). Student pictured is Chirag Mevawala.*

Chemical Engineering Laboratory II, or Senior lab, at UD allows students to pursue their career interests in either academia or industry by participating in undergraduate research or completing two pilot plant scale chemical engineering experiments. The three experiment options, DIST, FERM, and BIOD, allow students to work in teams to apply their knowledge from core CBE classes to actual equipment they may encounter in industry. The experiments give students the opportunity to design and execute experiments, analyze data with engineering software such as ASPEN Plus® and MATLAB,® lead projects, write and present engineering reports, and demonstrate time-management skills. Faculty interact with the undergraduates in Junior and Senior labs more like managers working with colleagues on projects rather than as professors teaching students. This experience has proven to be valuable to the students who often return after graduation and state that the Junior and Senior labs at UD were the most intense and memorable courses they took in engineering school. Many students discuss what they learn in their labs during job interviews, ask the faculty members to serve as references, and feel that the experience has helped them to obtain positions in industry and entrance to graduate schools. The knowledge and skills gained in Junior and Senior labs provide students with the necessary background to be successful at the start of their chemical engineering career. For this reason, the CBE department at UD has continued to provide significant resources to its undergraduate laboratories.

ACKNOWLEDGMENTS

The authors would like to thank Dr. James Seferis and the GloCal Company for the donation of the biodiesel processor. Additional thanks go to Agilent Technologies and Dr. James McCurry for donation and setup of the gas chromatograph. We would like to thank Professor Antony Beris who was instrumental in the initial development of the biodiesel experiment. Funding from Unidel Foundation Inc. was critical for implementation of the DIST and BIOD labs. We would also like to thank George Whitmyre, Neil Garrett, Weihua Deng, and Gary Wellmaker for maintaining the laboratory equipment. Thanked for their assistance are teaching assistants Trong Pham, Long Chen, and Edward Schreiner, as well as students Brianne Henry, Austin Kotch, Christine Muzzelo, and Kyle Tucker.

REFERENCES

1. Delluva, A., S. Salonga, B. Stewart, J. Arivalagan, R. Lehr, P. Dhurjati, and M.B. Shiflett, "Chemical Engineering Junior Laboratory and the New Kinetics Experiment at the University of Delaware," *Chem. Eng. Ed.*, **49**(3), 149 (2015)
2. Fredenslund, A., "A New Traditional Unit Operations Laboratory Course," *Chem. Eng. Ed.*, **7**(3), 142 (1973)
3. Sather, G.A., and J. Coca, "The Operations and Process Laboratory: A Unique Summer Course at Wisconsin," *Chem. Eng. Ed.*, **22**(3), 140 (1988)
4. Luke, J., and N.L. Ricker, "Unit Operations Lab: Mass Transfer and Axial Dispersion in a Reciprocating-Plate Liquid Extraction Column," *Chem. Eng. Ed.*, **32**(3), 202 (1998)
5. McCallum, C.L., and L.A. Estévez, "Introducing Process-Design Elements in the Unit Operations Lab," *Chem. Eng. Ed.*, **33**(1), 66 (1999)
6. Perkins, D.M., D.A. Bruce, C.H. Gooding, and J.T. Butler, "An Automated Distillation Column for the Unit Operations Laboratory," *Chem. Eng. Ed.*, **39**(2), 104 (2005)
7. Aronson, M.T., R.W.,Deitcher, Y. Xi, and R.J. Davis, "New Laboratory Course for Senior-Level Chemical Engineering Students," *Chem. Eng. Ed.*, **43**(2) 104 (2009)
8. United States Department of Labor, Bureau of Labor Statistics <www.bls.gov>
9. ABET <http://www.abet.org>
10. University of Delaware IT Academic Technology Services. CHEG 445: Chemical Engineering Laboratory II <http://udcapture.udel.edu/2014f/cheg445/>
11. Sakai <http://sakaiproject.org/about-sakai-project>
12. Aspentech. Aspen Plus. Vers. 12.1. N.p.: Aspentech, n.d.
13. Labview. NI Labview. Vers. 2011: National Instruments.
14. Doherty, M., and M. Malone, *Conceptual Design of Distillation Systems*, Boston: McGraw-Hill (2001)
15. Chalfie, M., "Green Fluorescent Protein," Photochemistry and Photobiology 62 651 (1995) Wiley Online Library. 2 Jan. 2008 <http://www.onlinelibrary.wiley.com>
16. New Brunswick™ Fermentation Systems <http://www.newbrunswick.eppendorf.com>
17. Junker, B.H. "Scale Up Methodologies for Escherichia Coli and Yeast Fermentation Processes," *J. Bioscience and Bioengineering*, **97**(6) 347 (2004)
18. <http://www.mathworks.com>
19. Sigma-Aldrich. "Specification Sheet." Specification Sheet. Sigma-Aldrich, 2014. <http://www.sigmaaldrich.com>
20. Sigma-Aldrich. "D-(+)-Glucose Product Information." Nov. 2002. <http://www.sigmaaldrich.com>
21. "KrosFlo® Research IIi Tangential Flow Filtration System." KrosFlo Research IIi TFF System for Tangential Flow Filtration at Spectrum Labs. <http://www.spectrumlabs.com>
22. Farrell, S., and E. Cavanagh, "Biodiesel Production, Characterization and Performance: A Hands-On Project For First-Year Students," *Educ. Chem. Eng.*, 9 e21-e31 (2014)
23. Farrel, S., and E. Cavanagh, "An Introduction to Life Cycle Assessment With Hands-On Experiments For Biodiesel Production and Use," *Educ. Chem. Eng.*, 9 e67-e76 (2014)
24. Tu, Q., C. Zhu, and D.C. McAvoy, "Converting Campus Waste Into Renewable Energy—A Case Study of the University of Cincinnati" *Waste Management*, **39**, 258 (2015)
25. GloCal Network Corporation <https://www.glocalcorp.net>
26. GE Healthcare Life Sciences. "Prepacked Chromatography Columns for AKTA Systems"
27. McCurry, D.J., and C.-X. Wang, Analysis of Glycerin and Glycerides in Biodiesel (B100) Using ASTM D6584 and EN14105. Agilent Technologies. 1-2
28. ASTM D6584-10a: Standard Test Method for Determination of Total Monoglyceride, Total Diglycerides ❒