AC 2011-711: INTEGRATION OF THE CHEMICAL ENGINEERING LABORATORY WITH A FOCUS ON BIO-FUEL PRODUCTION

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Prof. Pfaendtner's research group focuses on multiscale modeling of biophysical systems. His group develops and applies new computational methodologies for a wide range of problems of chemical engineering interest including biomaterials and biocatalysis. Prof. Pfaendtner earned his B.S. from the Georgia Institute of Technology and his Ph.D. from Northwestern University. After serving a two year post-doc at ETH Zurich in Switzerland, he joined the faculty of Chemical Engineering at the University of Washington in 2009.

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Marvi A. Matos is naturally from Puerto Rico. She obtained her BS in Chemical Engineering at the University of Puerto Rico-Mayaguez. Subsequently, she graduated from Carnegie Mellon University with a PhD in Chemical Engineering and a Masters of Science in Polymers, Colloids and Surfaces. Her dissertation work presented a novel technique to allow for the control of mass transport in crosslinked hydrogels with applications in the fields of biosensors and microfluidics. Under a fellowship from the National Research Council, Marvi worked as a postdoctoral fellow at the National Institute of Standards and Technology (NIST). Her project at NIST involved the study of encapsulated neural stem cell's viability and differentiation under AC electric fields. More recently (2008-2010), she worked as a Lecturer at the University of Washington teaching the Chemical Engineering Laboratories (traditionally the Unit Operations lab). Her worked as a Lecturer included the development of new experimental modules for undergraduate ChemE students, the submission of proposals with an educational focus and the supervision of the laboratories. During this time she also participated in outreach activities arranged by the College of Engineering to target increasing the number of students from underrepresented minorities in engineering programs. Today, Marvi serves as a Senior Research Scientist in the Bioengineering Department at the University of Washington and works as an independent consultant in engineering innovations.

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Integration of the chemical engineering laboratory with a focus on biofuel production.

Abstract:

The production of renewable energy is one of the most important technological problems that we face today. This challenge also offers us an opportunity to motivate and shape the early careers of Chemical Engineering undergraduate students. With this goal in mind, we have designed an innovative pedagogical model for the Chemical Engineering Laboratory that is based on the central theme of producing fuels from biomass. The most innovative component of the new laboratory is the complete integration of new and existing experimental stations. The second part of the unit operations laboratory course at the University of Washington was integrated to model a biofuel production plant where student groups work on individual operations that make up a complete process. This full-plant view of the laboratory allows students, for the first time, to evaluate the effects of their decisions on upstream and downstream plant operations. Furthermore, it also provides a common framework to promote active discussion and engagement amongst student groups. The transformation of the course included the development of completely new modules for fermentation of biomass and the modification of existing equipment and modules for the treatment, separation and extraction of product and waste streams. The new fermentation modules utilize internet-based remote monitoring technologies to track the development of fermentations while students are outside of the laboratory. Fully interconnected units now define a common goal of reducing costs and improving productivity and replace the original independent and unrelated experiments. The new structure also allows us to easily incorporate design concepts, such as cost analysis and environmental compliance, into the laboratory. The objective of the re-designed course is to provide a realistic structure that is congruent with what students will experience when they enter the workforce as chemical engineers. The new laboratory structure is also designed to foster leadership, creative thinking, composure under uncertainty and the critical review of information. Furthermore, with the new structure, we also continue to meet the original learning objectives of instructing students on the basics of experimental planning and reporting.

Introduction and motivation for a consolidated Chemical Engineering laboratory:

Most Chemical Engineering curricula include at least one or two laboratory courses where students develop their hands-on skills by working on experimental stations that are related to fundamental processes used in traditional chemical industries. Various independent stations are usually used in the laboratory to cover a wide range of important Chemical Engineering concepts such as heat exchange, distillation and chemical reactions. Typically, these individual units are conceptually independent from each other because the laboratory courses and the related infrastructure develop gradually over very long periods of time. Furthermore, many instructors often participate in teaching these courses and each of them contributes a part of their own experience to the development of the laboratory. This often creates laboratory courses that can be technically meaningful for the students but lack a central cohesive theme. Before this project was initiated, the laboratories at the Chemical Engineering program of the University of Washington fell into this category. For several decades, individual experimental stations provided our senior undergraduate students with valuable experiences that were aimed at directly correlating important concepts learned in classroom instruction with important issues encountered in practice by real process engineers (e.g. performing an analysis with limited information on process conditions).

A critically important limitation of this course structure is that students cannot easily recognize that, in real industrial processes, units are interconnected and their operation affects and is affected by changing process conditions in other upstream and downstream units. We realized that it was important for our students to develop a broad "full-plan" view of the processes that they analyze and that they needed to consider their individual tasks within this framework to make the laboratory experience more realistic, interesting and relevant. In this way, the focus would be shifted form individual units and short-term tasks to a comprehensive view of the whole process where the collective goal of all groups is to make a mock company more profitable. Such drastic modification of the old course structure required a complete revision of all laboratory experiments, the pedagogical model and the course structure as well as a significant investment of resources for new equipment. Furthermore, we still wanted to provide students with experiences covering fundamental concepts of the traditional Chemical Engineering curriculum (e.g. mass transport and heat exchange) but also to include new experiments dealing with important concepts for the energy and biotech industries that were not introduced in other courses.

All of these objectives have been met in an efficient way by focusing the theme of the laboratory course around the concept of commercial biofuel production for transportation. This contemporary subject nicely couples traditional chemical industry operations (e.g. distillation and absorption) with important processes that are used frequently in other industries that hire our graduates (e.g. enzymatic reactions and fermentation processes). Therefore, we are now able to provide a more realistic, comprehensive and contemporary educational experience to all of our undergraduate students. Furthermore, we have also modified the course framework to help students develop "soft" skills that were not explicitly targeted in previous versions of the course.[1] These skills include the critical evaluation of information, effective intergroup interaction, management of uncertainty, successful planning and effective oral and written communication. To accomplish all of these goals we have leveraged resources from the NSF, from our institution and from industrial partners; we reused a large part of the existing laboratory infrastructure (e.g. distillation columns) and we successfully harnessed local faculty expertise in this subject.

Educational objectives:

The original version of the laboratory course defined a base set of learning objectives. One important requirement for the new version of the laboratory course was that we would still continue to meet these educational objectives for all students. The basic educational objectives for students enrolled in the course were the following:

- To understand fundamental Chemical Engineering concepts through practical application.
- To develop skills for effective experimental planning.
- To develop skills related to data acquisition and error estimation.
- To develop an ability to analyze real experimental data.
- To enhance their ability to communicate technical information in oral presentations and in written reports.
- To learn to work effectively as integral members of a team (intra-team interaction).

In addition to these basic objectives, a new set of educational objectives was also defined for the new laboratory course. The new integrated laboratory modules would help students develop important skills that were not explicitly addressed in the previous version of the laboratory. The additional learning objectives for students participating in the integrated laboratory were the following:

- To develop effective skills for inter-team interaction and collaboration.
- To critically review information from previous reports and from the scientific and technical literature.
- To learn to transform results into clear recommendations for other groups collectively participating in solving a large problem.
- To understand how their work can impact processes upstream and downstream as well as to understand how other processes can impact their specific units.
- To work with contemporary unit operations involving fundamental concepts related to biotechnology and renewable energy.
- To learn to work within the framework of relevant environmental regulations.
- To evaluate the economic implications of changes to individual processes on the operation and profitability of a large chemical plant.

The new structure of the course allows us to meet these new educational objectives in a way that was not possible with the independent experiments that were previously used.

Course structure:

To graduate with a Chemical Engineering degree from University of Washington, students are required to enroll in a sequence of two Unit Operations laboratory courses (ChemE 436 and 437). Only the second course (ChemE 437) was modified in this project. The first course (ChemE 436) offers conceptually independent experiments in transport phenomena with specific focus on fluid flow and heat transfer. Therefore, the first course in the sequence serves as a good basis for assessment of the effectiveness of the new structure. The second laboratory (ChemE 437) offers experiments involving unit operations focusing specifically on mass transport and chemical reactions. This is the only laboratory course that has been modified to have the "full-plant" structure. In both courses, students work in groups of three for the whole quarter and do not change partners. Generally, a laboratory section will have a total of five groups so that there are five concurrent experiments in every section. There are four sections each week and every student in the Chemical Engineering program is required to take both courses in the sequence. Each engineering group participates in three different experiments during one academic quarter and each experiment takes three weeks to complete. A rotating leadership structure is used within the groups to help to identify individual contributions on the required tasks for each experiment (i.e. experimental planning report, oral report and final report). Each student serves as leader for a different task in each experiment and receives a larger percentage of the total points. This same structure is used for both laboratory courses to avoid any unintended bias when comparing both student experiences.

In order to engage the students in the class, all assignments are provided in the form of memos from the "Managers" of a mock company called "Northwest Biofuels". We find that students respond positively to assignments that are framed within a realistic narrative that is tied to a real-world application.[2] The problem description in the memo that is given to each group usually describes a large problem and a set of goals in broad terms and with limited detail. Frequently, the statements and goals are intentionally written to be highly ambitious. It is expected that these goals will not be achievable by any single group working within the three-week period of each experiment. This forces students to dissect the large problems into smaller achievable steps that need to be tackled in sequence to advance the project forward. For all experiments, students are also provided with initial reference materials including the final reports (uncorrected) of pervious groups working in this problem, relevant journal papers, material datasheets and equipment operational manuals. These are all provided electronically through a central course website and a discussion board. All additional references that are found by individual student groups and/or the instructors are also shared and placed on the website for all groups to access this information. In every experiment, students are evaluated based on their performance and on their technical knowledge instead of focusing on obtaining positive experimental results. Specifically, groups are judged based on their contributions to advancing the large-scale projects given the specific circumstances at the beginning of their experiment (e.g. how much information was available to them at the time they started each project).

For each of the three experiments, each team is required to present a planning report, an oral presentation and a final report and each student in the group acts as a “leader” for each task. Leaders obtain a larger fraction of the total points for their specific tasks but every student in a group also obtains points. This motivates all students in a group to actively participate in all tasks even when they are not leaders. The first task consists of a planning report that is submitted three days after the students receive their new assignments. These reports evaluate the capacity of students to develop effective and realistic experimental plans and also allow instructors to provide feedback to students so they can adjust their strategy, if necessary, before the first lab period. Oral presentations are open to the whole class so that students can probe and question the data of other groups and monitor the progress of the integrated projects. Because experiments are offered in three phases, each engineering group has an opportunity to explore the operation of three different units involved in the biofuel plant as part of their rotations. The results obtained by the groups in each phase are also made available to all teams in the form of uncorrected final reports. Teams can decide to use this information or to ignore it when designing and performing their own experiments. Students have embraced this structure and can usually identify all mistakes in previous reports. This also allows students to try ‘proof-of-concept’ experiments that are then followed by other groups. This structure is designed to allow students to develop their ability to ascertain both quality and utility of information from many sources. This is especially valuable in contemporary times because of the preponderance of data and information available throughout the decision making process.

Current status and implementation:

The modification of the infrastructure used in the Chemical Engineering laboratory was initiated in September 2010 and the first implementation of the new course structure was completed over the Winter quarter of 2011. Six integrated modules were implemented around the topic of biofuel production in the first iteration of the course. All modifications to the laboratory have been implemented with the central idea of creating a versatile laboratory infrastructure that can be equally used to emulate conditions used for the production of bio-ethanol or bio-butanol for use as liquid transportation fuels. Bio-butanol as an alternative fuel has several key advantages over bio-ethanol that originate from its higher energy density and higher hydrophobicity.[3] Nevertheless, there are important processing hurdles that still make bio-butanol technologies economically inferior to ethanol. The purification of ethanol is also significantly simpler because fewer products are produced during fermentation. For simplicity, ethanol production was chosen for the first implementation of the course to allow us to fine-tune the structure and to troubleshoot all of the new modules. Students will explore butanol production in future iterations of the course.

It is important to clarify that the physical experimental units in the laboratory are only *logically* connected to each other. In other words, materials do not physically flow from one unit to another (e.g. from the fermentors to the distillation column) because the size scales are usually very different. Instead, only the values of the key process conditions or numerical parameters (i.e. inlet/outlet composition, temperature, tray efficiencies etc…) are used to simulate the connections between units. The following is a brief description of the new experimental modules and the new course features that have been developed at this time and are currently being used in the laboratory:

<u>Process simulation tools and economic analysis:</u>

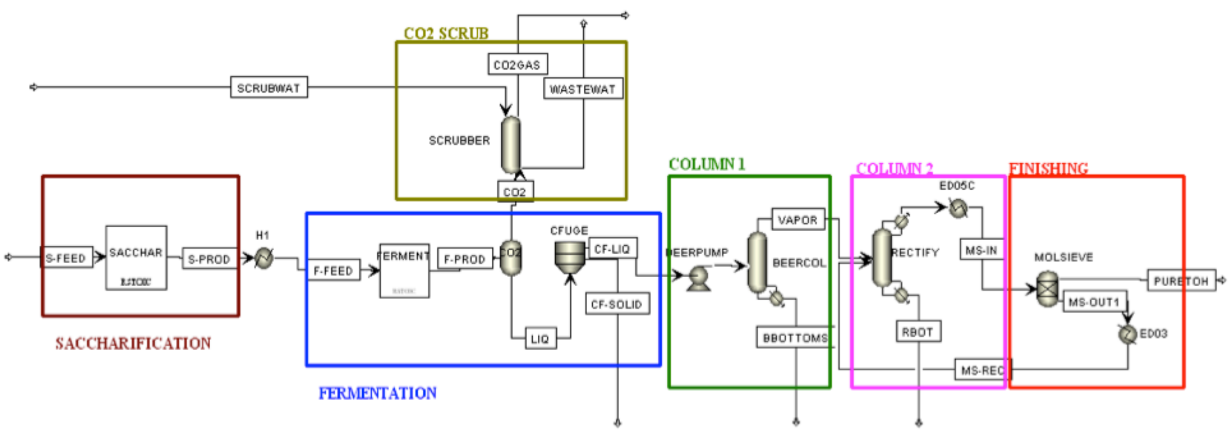

Figure 1: Screenshot of the ASPEN model that is provided to the students for the initial evaluation of the effect of changing process conditions.

A highly simplified ASPEN model for a bio-ethanol production plant was developed and provided to the students so they would use it as a basis for analyzing the impact of their results on the operation of the whole plant. The simulation model contains units that are representative of all the experiments that students perform in the laboratory. This common framework also serves to discuss the full process from a common perspective with all students. One typical example for the use of the simulation model is for students to evaluate real-world parameters in the laboratory (e.g. actual tray efficiencies in distillation) and feed this information back to the model to calculate the impact on plant operation. In another example, students can evaluate the propagating effect that a very low ethanol concentration in the fermentors will have on the distillation processes. Figure 1 shows a screenshot of the ASPEN model that the students use. Together with the process simulation model, the students are also provided with a simple Excel spreadsheet that they can use to evaluate basic operating costs for each section of the plant. This spreadsheet also defines all of the economic parameters that the students use in their economic evaluations (e.g. cost of raw materials, energy and sale price of products).

Because the initial models are oversimplified, students are encouraged to modify the models so that they can add detail to the calculations if they so desire. The only requirement that plays a role in the final grading is that they need to discuss the implications of their findings on the performance of the whole plant. They can choose to discuss and quantify this with these basic tools or they can use other resources to do this. The incorporation of modeling and experiments related to the full-plant view is a unique and powerful concept of this new laboratory structure. However, we also think that it is necessary to reach a reasonable balance between the positive educational outcomes that can result from this additional analysis and the level of complexity involved in the calculations. It is also important to also set reasonable expectations for undergraduate students at this level. Students enrolled in this laboratory course are concurrently taking the first of two process design courses so they only have very limited experience in rigorous economic analysis of processes. Nevertheless, most of them already have some ASPEN experience from previous courses and electives.

Biomass saccharification:

One of the largest contributors to the final cost of producing fuel grade alcohol is the price of biomass. Therefore, the choice of feedstock is key to making a commercial biofuel process profitable. Three types of feedstocks are generally used in the commercial production of alcoholic biofuels; sugar, starch and cellulose. Of these, only the sugar feedstocks (e.g. sugar cane juice) are fermented directly by yeast without any necessary pre-treatment. We have incorporated the pre-treatment stage of the biofuel production process into the new laboratory through the design of an experimental module that focuses specifically on the bioconversion of low-cost cellulosic biomass that could be obtained, for example, from municipal waste or from byproducts of the forestry industry. At various stages of the project, some students are asked to evaluate the possible use of raw sugar-cane juice as a feedstock to the process while other groups are asked to evaluate the use of cellulosic feedstocks. Cellulosic feedstocks are provided at a much lower cost, when compared to sugar-cane juice, but they now need to consider the added cost of pre-treatment. Students performing these experiments are specifically asked to consider an enzymatic saccharification process in which the price of the enzymes can account for a substantial fraction of the final cost of the product. Frequently, there is also a set of acid and steam expansion stages that are used to expand and expose the cellulose before enzyme reactions can take place. For safety and cost reasons we have not included these pre-treatment steps in the laboratory. Instead, students directly use cellulose pulp that is ready for enzymatic breakup of the polymers into glucose units.

For the saccharification reaction, students use various commercial cellulase and β-glucosidase dispersions (Accellerase 1500 and 1000) that have been generously donated by Genencor (Palo Alto, CA) for use in the new laboratory. In the memo, students are asked evaluate the optimal enzyme dose and reaction conditions for the saccharification of the specific cellulosic feedstock that they are using at the time. The students use a temperature-controlled automated shaker table with several baffled Erlenmeyer flasks to initiate the enzymatic reactions and to track their progress as a function of time. Suggested initial conditions are provided to the students by posting the datasheets from the manufacturer in the course website. These are very general guidelines that are not specific for each feedstock. Therefore, the students still need to optimize the specific reaction conditions for their feedstock. In the laboratory, students track the formation of glucose in the reaction via one or more of several possible analytical methods (see "Composition analysis" section of this paper). Some of the parameters that students can change in their experiments include the reaction temperature, the agitation level, the concentration of biomass, the concentration of enzyme dispersion and the type of enzyme (e.g. Accelerase 1000 or 1500). Students are also asked to model the reactions using Michaelis-Menten kinetics or any other suitable kinetic models.[4, 5] In making their final recommendations, students need to consider important pros/cons including the lower cost of the biomass feedstock but also the added cost of the enzymes and the heating duty that is required to maintain the slurry at the required temperature. It is our goal that this decision making process will be based on the economic implications of these actions in the performance of the whole plant.

Fermentation:

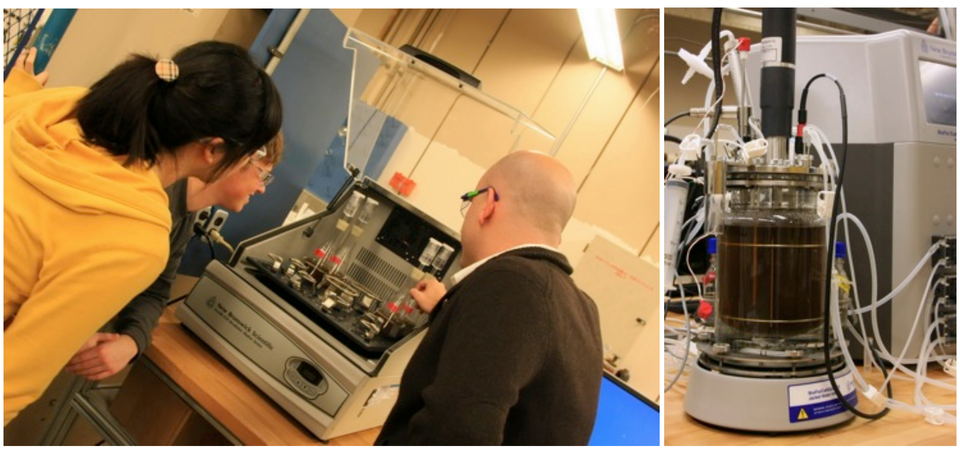

Figure 2: Images from the two fermentation experiments using automated bioreactors (right) and several parallel flask-scale experiments (left) allowing simultaneous variation of multiple parameters.

The next stage of the biofuel process after pre-treatment is the fermentation of the biomass. The new version of the laboratory has two stations and modules that are used for analysis of the parameters affecting the fermentation process. In an industrial fermentation the main parameters that will usually affect the economy of the process are the fermentation time, the yield and the final alcohol concentration. Ideally the fermentation is fast so that more biomass can be processed in smaller or fewer bioreactors. A high conversion efficiency and final yield are also essential to ensure that most of the biomass is used effectively. Finally, a high alcohol concentration is also important to reduce the cost of running the separation and purification units downstream.

Two new fermentation modules using automated bioreactors and flask-scale experiments have been implemented for the students to use as part of the new course (Figure 2). In these modules students evaluate the economic and technical effects of changing the biomass feedstock (e.g. sugar cane juice vs paper from municipal waste), the yeast strain and/or the process conditions (e.g. temperature, pH, concentrations). One of the modules makes use of small flask scale fermentations that can be run in parallel so that students can evaluate up to eight different conditions in the fermentors throughout their experiment (left Figure 2). This module also makes use of a temperature controlled automated shaker and various analytical techniques to track the progress of the fermentation (i.e. consumption of sugar, production of biomass and ethanol). Groups performing this experiment are asked to explore the effect of changing initial conditions in the flask reactors but they are unable to perform online control of these critical parameters as the reaction proceeds (e.g. pH, dissolved oxygen or the rate of biomass addition). On the other hand, these groups can explore several process conditions in parallel and collect enough samples to accurately fit their reactions rates to published models.[6]

In a second fermentation station, other groups use one of two new automated bioreactors (Bioflo 115 from New Brunswick Scientific) to run and evaluate fermentations (Figure 2). The use of these automated bioreactors reduces the number of fermentations that these students can run in the three-week experiment period to just two. However, these reactors can be configured to monitor and control all of the most relevant parameters to the fermentation and they can also be configured to run complex methods that are not possible to run in simple flask reactors (e.g. fed-batch or pseudo-continuous fermentations). Furthermore, the automated bioreactors are more representative of the level of control and online monitoring that would be available in larger industrial vessels. Thus, they allow students to closely mimic industrial conditions. For the fermentation of sugar feedstocks the students have been assigned with comparing the performance of three possible commercial yeast strains used in the biofuel industry. The yeast samples were generously donated by Lallemand (Montreal, Canada). The assignments that are given to all fermentation groups are very similar we observed that the students used effective inter-group communication to collectively maximize the amount of information that is collected from the bio-reactors. Thus far, this sharing strategy to be extremely successful and students from different groups and from different sections have coordinated their experiments using the online discussion boards. Students are encouraged to share the raw data between groups but they need to explicitly state and acknowledge the contributions of other groups and also identify their own work in all of the reports. Furthermore, groups are allowed to share data but they still need to perform their analysis of the data independently.

It is important to note that fermentations can take place over very long periods of time (typically 10-48 hr). This presented a logistical challenge because each laboratory period is only four hours long. Two solutions have been used to allow students to collect data and track the progress of their reactions outside of the regular lab period. First, an online desktop broadcasting tool (Procaster) has been used in the bioreactors to allow students to monitor the progress of their reactions at any time even when they are not present in campus. This allows them to identify problems such as unexpected changes in pH and to take corrective action. We also added webcam capabilities that allow students and instructors to visually observe the bioreactors from any computer with an internet connection. The webcams allow students to identify common problems (e.g. empty acid/base bottles) and also allow them to qualitatively observe changes in broth properties (e.g. to assess yeast growth). Secondly, we have also given students controlled access to the laboratory outside of the regular class period during regular university work hours (i.e. when staff is present). This has required additional help from staff, teaching assistants and faculty to serve as chaperones to groups that need to enter the lab briefly to withdraw samples from the bioreactors. The response from students for offline access was overwhelmingly positive and it showed that they were extremely engaged in the course. On the other hand, this also created an added burden on their already heavy course load.

<u>Distillation:</u>

In ethanol fermentation, most beer broths will typically contain a final alcohol concentration that ranges between 5-20 % by volume. Fuel grade ethanol must contain less than 1% by volume of water. Therefore, the purification requires significant removal of water and this accounts for a large fraction of the costs for producing ethanol and other alcoholic fuels. Industrially, there is a strong drive to increase the final alcohol content by using higher biomass concentrations. Nevertheless, this still requires surpassing several technological hurdles that include making yeast strains that can survive the stress imposed by high sugar or product concentrations. Most of the removal of water in fuel grade alcohol production is performed through continuous distillation. Fortunately, the old version of the laboratory course already included two distillation experiments and it was possible to reuse these facilities in the new version. This resulted in significant cost savings for the implementation of the new laboratory course and also allowed us to continue teaching these important Chemical Engineering concepts while still keeping the realistic full-plant theme.

Two existing distillation columns, of different size, were upgraded and new experimental modules were created to process the ethanol-water feeds. One of the columns is a tray column with four stages that can be fitted with different types of trays (i.e. sieve, bubble cap or valve trays). This column was completely retrofitted with new RTD sensors at each stage, new sampling ports for vapors and liquids, flow sensors for the reflux and for the steam condensate, differential pressure sensors in the pot and at the top tray, an ultrasonic level sensor in the reflux drum and a control valve in the reflux line. An interactive interface was also created to emulate an industrial control room (Figure 3). The smaller spinning band column was also retrofitted with new RTD sensors, a controller for the reflux valve, a controller for the reboiler, new sampling ports and a weighing balance to track the total product collected in the receiver. A new user interface was also created for this column to emulate an industrial control room.

Figure 3: Images of the two distillation columns used in the laboratory (left) and an example of the new interface and online monitoring capabilities that have been added to them (right).

The two columns represent the two primary stages in the separation process for the ethanol (Figure 1). The larger tray column emulates the condition of the first beer column in the process flow sheet. Therefore, the input composition in the pot is representative of the outlet from the fermentation vessels. Groups performing the fermentation experiments provide typical values of the input composition based on their experimental results. In a typical assignment, the students performing this distillation experiment are asked to evaluate the efficiency of each type of tray and to input these values to the simulation model to evaluate the effect on the performance and economics of the whole process. A smaller rectification column (i.e. spinning band column) follows this first column to further increase the ethanol content. The typical input to this column is an ethanol-water stream that is usually > 50 % ethanol by volume but this depends on the specific operating conditions of the beer column that precedes it. Students performing this experiment are asked to analyze the performance of the column (i.e. find the equilibrium number of stages) and to use the method of Smoker and Rose to model the production of high concentration ethanol for batch distillation under unsteady state conditions.[7] Ethanol-water mixtures have an azeotrope at around 95 % by volume. Therefore, this second distillation column will never be able to produce a product that meets fuel grade specifications. The outlet of the rectification column needs to be further dehydrated and this represents the primary goal of the molecular sieve dehydration experiment in the last experimental station of the new laboratory.

<u>Molecular sieve dehydration:</u>

Because of the homogeneous ethanol-water azeotrope, it is impossible to reach fuel grade specifications with simple binary distillation. In industrial plants, one commonly used process to further dehydrate the ethanol is to use pressure-swing absorption in pairs of packed bed columns containing molecular sieves. Molecular sieves are inorganic zeolites with molecular sized pores (~3-10 Å) that can be used to selectively absorb molecules based on their size.[8] Depending on the molecular sieve that is used, it is possible to completely exclude ethanol from the pores so that only water is selectively absorbed. This results in significant enrichment of the ethanol concentration that make it possible to reach fuel grade concentrations without resorting to more complex forms of distillation. Industrial absorbers also need to be regenerated after they reach saturation of the packing material. This is performed by applying heat, changing the pressure and by passing a purging stream of clean gas (e.g. CO_2). It is well known that the performance of the absorber is tightly associated to the molecular structure of the molecular sieves that are used in the packed beds. Thus, we find that this is a very effective experiment to also introduce important molecular engineering concepts into the laboratory.

Figure 4: Student performing the jar-scale molecular sieve experiment (left). Image of the thermogravimetric analysis (TGA) equipment used in the desorption experiments (right).

In this assignment, students are asked to evaluate several possible molecular sieve materials having variable pore sizes. They are asked to run bench-scale experiments and to make recommendations to the process engineers for what type of packing they should use in the actual process. Students use simple experiments to characterize the parameters that are most relevant to an absorption process occurring in the liquid phase. It must be noted that liquid-phase absorption was used in the early days of this technology but it is now more common to perform the adsorption directly from the vapor phase. Typically, students choose to prepare jars with different types and weight ratios of the molecular sieves and with an alcohol-water mixture that is typical of the outlet stream from the distillation columns (Figure 4). They then track the changes to the concentration of water in the mixture as a function of time by using gas chromatography. This allows them to evaluate the kinetics of absorption and the total absorption capacity for each molecular sieve type. The rate of absorption is typically fast enough to track changes in ethanol concentration and reach equilibrium over the course of the laboratory period (4 hr). Students also have access to a thermogravimetric analysis (TGA) instrument that they can use to further evaluate the absorption and regeneration properties of each material. With this instrument they can track changes in mass for saturated molecular sieves as a function of temperature or as a function of time for a fixed temperature. This allows students to evaluate and compare the minimum temperature that is necessary to regenerate each type of molecular sieve and also to track the kinetics of desorption at a fixed temperature to make their final recommendations. In future upgrades we plan to develop a bench scale version of an absorber that students will use to measure the breakthrough curves for each type of packing.

<u>Composition analysis:</u>

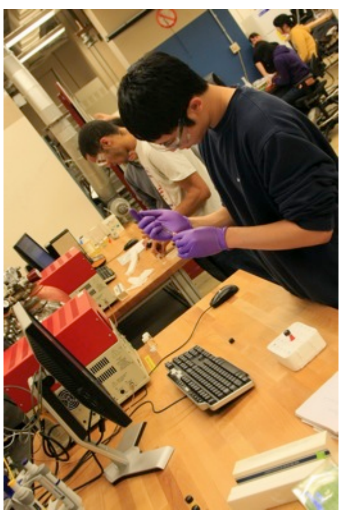

Figure 5: Image of students using the two GC instruments in the new version of the unit operation laboratory.

The new version of the laboratory is much more challenging and complex than the previous version of the course. Therefore, we wanted to simplify the analytical process as much as possible so that students could focus their effort on the assignments that have been described in the previous paragraphs rather than on developing composition analyses. One way to do this was to consolidate the effort on a few streamlined methods that students can use for composition analysis in most of their experiments. For accurate analysis of ethanol content, which is important to most of the stations, we have chosen to use gas chromatography (GC). Two GC instruments with thermal conductivity detectors were purchased from SRI instruments to accurately analyze ethanol content over the range of 500 ppm to 100% (Figure 5). Since all stations (with the exception of the saccharification process) need to analyze ethanol concentrations, students only need to learn how to perform the GC analysis once and they can reuse this technique for all of their other experiments. This reduced the number of analytical techniques that students needed to learn and greatly simplified the instruction process. Furthermore, the students gain valuable experience because the use of GC is extremely common in a variety of industries.

For sugar analysis the students can currently use several possible methods. The most accurate, but also the most difficult and laborious, is to use a colorimetric method based on dinitrosalycilic acid (DNS).[9] This assay can quantify the concentration of any reducing sugar (including glucose), is inexpensive and only requires a simple UV-Vis spectrophotometer. The students are also given the option to use a hand-held glucose meter that is designed for blood-sugar monitoring for patients with diabetes. This is a simpler assay to perform in the lab but can be more expensive so that it is necessary to limit the total number of strips that are provided to each group. So far students have predominantly chosen to use the DNS method because they are not constrained in the number of samples that they can run. As with the GC, monitoring sugar content is also necessary in more than one station. In fermentation students use these methods to track sugar consumption by yeast cells while in the saccharification experiments students track the production of sugars from the enzymatic lysis of the cellulosic biomass. In the future we would like to include liquid chromatography (HPLC) for sugar analysis as it is faster, more accurate and simpler to perform. Finally, students performing the fermentation experiments are also provided with all of the tools that they need to perform optical density (OD) measurements to track cell growth. It has been documented that ethanol production is intimately tied to the growth of yeast.[8] Therefore, this is a very important parameter in modeling the fermentation process and students need to track its development. For this assay, students in the laboratory can use the same UV-Vis instrument that is used for the DNS sugar assay.

Assessment of the course:

Students enrolled in this new course have already experienced independent experiments in the first Chemical Engineering laboratory course (ChemE 436). Therefore, the two courses provide a useful basis to compare the effectiveness of the two instruction strategies in the form of independent modules vs. integrated modules. We evaluate the learning objectives using a combination of surveys, direct quantification (i.e. grading scheme) and through the use of an external consultant from the Center for Engineering Learning and Teaching (CELT). The external consultant also conducted an un-biased evaluation of the laboratory in the form of a small group instructional diagnosis survey (SGID) to provide mid-course feedback to the course instructors.

Table 1 summarizes all of the new and old course objectives and also indicates the specific form of assessment that is being used for each item. Because the course has just finished the first iteration at the time of writing, not all of the assessment results have been completed to be included in this paper. We expect to report on the complete findings in future publications after data analysis.

Table 1: Assessment Matrix for Learning Objectives
Improvements to Chemical Engineering Education

	Assessment Method			
Objective	**Pre and Post Course Surveys**	**Grading Scheme (reports and oral presentations)**	**External Reviewer Formative Assessment**	**Student Peer Review**
Traditional Objectives Development of:				
ChemE understanding through practice		X		
Experimental planning skills		X		
Data acquisition skills		X		
Data analysis skills		X		
Oral and written reporting skills		X		
Intra-team working skills			X	X
Novel Objectives:				
To develop inter-team working skills			X	X
To critically review technical information		X		X
To formulate clear recommendations		X		
To understand the impact of their work on a large scale plant operation		X	X	
To work in contemporary unit operations	X		X	
To learn to work with environmental regulations	X		X	
To evaluate the economic implications of project decisions		X		

Pre and post-course surveys were also prepared and administered by the assessment consultant from CELT (Dr. Jim Borgford-Parnell). Table 2 quantitatively summarizes the questions and results from the pre and post-course surveys that were administered to the students. The pre-survey sampled 94% (51/54) of students enrolled in the course and the post-survey sampled 89% (48/54) of the students.

Table 2: Pre & Post-Course Surveys

1	**2**	**3**	**4**	**5**	**6**
Strongly Disagree	**Disagree**	**Somewhat Disagree**	**Somewhat Agree**	**Agree**	**Strongly Agee**

To what extent do you agree with the following statements?	Pre Answers	Post Answers	% Agreed or Strongly Agreed
Intra-team communication was fostered in my prior laboratory work.	4.755 [< Agree]		
The new laboratory infrastructure fostered within-team communication.		5.08 [>Agree]	79.16%
Inter-team communication was fostered in my prior laboratory work.	2.80 [< Somewhat Disagree]		
The new laboratory infrastructure fostered communication between teams.		4.98 [<Agree]	75%
My previous labs were structured around one theme.	2.82 [< Somewhat Disagree]		
The new laboratory structure is designed around one theme.		5.38 [>Agree]	91.49%
In my previous lab courses, assignments were integrated and students worked together as parts of a whole plant.	1.78 [< Disagree]		
With the new laboratory structure students work as part of a plant rather than as separate teams in non-integrated modules.		5.06 [>Agree]	76.60%
I already have a good understanding of the Chemical Engineering discipline.	3.96 [< Somewhat Agree]		
ChemE Lab II expanded my understanding of the Chemical Engineering discipline.		5.0 [Agree]	81.24%
I learned teamwork and leadership skills in my prior coursework.	4.33 [> Somewhat Agree]		
I learned new "soft skills" (such as leadership and team work) not learned previously.		4.21 [>Somewhat Agree]	42.20%
I am interested in pursuing a career in Chemical Engineering.	5.22 [> Agree]		
The laboratory increased my interest in Chemical engineering.		4.83 [<Agree]	70.83%

I am interested in learning about energy problems.	4.96 [< Agree]		
The laboratory increased my interest in Energy related problems.		4.27 [>Somewhat Agree]	43.75%
I am interested in pursuing a career in biotechnology.	3.48 [> Somewhat Disagree]		
The laboratory increased my interest in Biotechnology.		4.25 [>Somewhat Agree]	45.83%
I am interested in learning about environmental issues.	4.16 [> Somewhat Agree]		
The laboratory increased my interest in environmental issues.		3.92 [<Somewhat Agree]	31.25%
I have taken Chem E Lab I *Circle one*: Yes No	Yes = 51 No = 0		
Previous lab courses I have taken were interesting.	4.00 [Somewhat Agree]		
The new laboratory structure and theme offered a more interesting perspective than other lab courses I have taken (e.g. ChemE Lab I).		5.19 [> Agree]	81.25%
The new laboratory structure and theme offered a more useful learning experience (e.g. I feel like I have learned more than in other traditional laboratories).		4.96 [<Agree]	81.25%
Compared to previous lab courses I've taken, the new laboratory structure is more relevant to the work done by real chemical engineers.		4.89 [<Agree]	74.47%
Compared to previous lab courses I've taken, the new laboratory structure is a better translation of industrial experiences and can serve as an appropriate practice.		4.98 [<Agree]	81.25%

Conclusions:

The integration of all experimental modules in our laboratory exposes students to a broader, more complete perspective of how a real chemical plant works. This is something that is truly unique to our new course design. In past versions of the course, all experiments were independent and had no clear or direct relationships to each other. In Chemical Engineering curricula, students complete a series of courses in transport phenomena (momentum, heat and mass) and reactor design before they enroll in the Chemical Engineering laboratories. By the time the students start in the laboratories, most of their experience with chemical unit operations is from the mathematical exercises of the lecture courses. Consequently, they have not really been able to apply the concepts to real practice. The laboratory is intense training in which students are exposed to a variety of practical and interdisciplinary concepts and charged with reporting experimental results and conclusions in written and oral forms. This new course structure also enables the integration of plant economics and design concepts that have traditionally been left out of the laboratory experience. This will contribute to providing a more realistic and broad educational experience for the students.

We believe that integrating the experiments into a coherent theme and operating the course as a full plant is a complete change from the traditional practice and that it can have a substantial positive impact on how students learn. It is also our hope that it will motivate other academic institutions to follow this example to provide similar experiences to their undergraduate students. With this setup, the traditional boundaries of team-work extend beyond individual groups, because students are required to work as a whole company. In addition, the usual framework of isolated independent experiments is replaced by interconnected units that define broader goals and represent a more realistic coordinated framework. This is much more congruent with what students will experience after graduation. Student teams will have to work effectively, create innovative and efficient experimental designs and connect their findings to the work of others in parallel and in series. This will require them to extend their focus beyond their specific operation and to carefully consider unit operations upstream and downstream. The complexity of this matter also offers a unique pedagogical approach with a more realistic perspective.

In this new framework, student work is also shared directly among teams in the form of presentations and reports. Such exchange of information helps students to acquire the necessary skills to prepare good technical oral presentations and written reports that are understandable by others and not just by the instructors. In addition, this will foster effective communication and collaboration among different groups of students. In some cases, students need to critically review the work of other students before moving along with their planned experiments. This skill will help them to rapidly filter material under tight time constrains in a realistic situation. Throughout their work, students also review recent scientific literature in the topics of fermentation and separation for biofuel production. They also evaluate the economic and environmental implications of their decisions and recommendations. The new laboratory experience provides a bridge between academic instruction and realistic engineering applications.

Finally, a big part of an engineer's work is to find practical and cost effective ways to solve problems. In integrated laboratory modules, the students must consider the impact of their decisions on upstream and downstream operations. They are not working with independent units; rather they are working with tightly inter-connected operations of the entire biofuel plant. Their decisions on laboratory conditions will be based on real and achievable parameters and on their economic effects.

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