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COMBINING EXPERIMENTATION AND SIMULATION IN A UNIT OPERATIONS LAB COURSE

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INTRODUCTION

In the chemical engineering curriculum, the Unit Operations laboratories and Process Design courses sit as capstones, where students combine theory with application. Typically, the Unit Operations laboratories are hands-on experiences with bench or pilot-scale equipment, while Process Design courses use simulations to explore a larger, industrial-scale plant environment.

Virtual unit operations laboratory experiments have been a topic of interest in the literature.[1-8] Some instructors have built simulations and let students experiment with those models to generate data that approximate what the physical set-up would do. Others have given students virtual access to the instructor's or teaching assistant's lab equipment via remote desktop or "remote control". The recent COVID-19 pandemic has renewed interest in and need for these virtual experiments to replace in-person experiments, but approach, implementation, and success have varied greatly.[9]

At the University of Florida (UF), the pandemic greatly restricted the number of students the teaching laboratories were able to support, down to a quarter of our original capacity in Fall 2020. The Unit Operations 1 course was moved online by shipping "kits" to students. The Unit Operations 2 course was split, with three of the experiments moved online and three of the experiments still performed in the lab. For the two online experiments that involved semiconductor manufacturing, no viable simulation alternatives were available. Students were therefore sent old data and asked to analyze it. For the online continuous distillation experiment, an opportunity presented itself to not only maintain the original learning objectives of the experiment, but also to expand the experiment and include a simulation aspect.

In the online continuous distillation module, students used old data and equipment specifications to simulate the distillation column in Aspen HYSYS®. Instructional videos were created by the Process Design instructor to guide students through a data-based simulation, something not usually explored in the undergraduate curriculum. Once students had a working simulation, they experimented in an open, "sandbox" style without the limitations of the existing equipment. For example, they were able to study the effects of changing the number of trays, something that is impossible with the physical set-up. Students completed the simulated version of the distillation experiment in Fall 2020 and Spring 2021.

In this work we present this Unit Operations module that integrated experimental data and simulation. This serves two purposes. First, we show how we integrated process simulations into a lab course, allowing students to utilize their data analysis skills in conjunction with their process optimization skills. Second, we discuss how this opens the door for further integration of other new aspects of our curriculum into

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the lab. We present the basic outline for the module, as well as discuss the students' experiences, students' feedback, and the department's plans for the module as we return to an in-person modality.

COURSE DESCRIPTION

General Description

At the University of Florida's Department of Chemical Engineering, we have 500-600 undergraduate majors. Roughly 120 of these students are seniors who are eligible for the Units Operations 2 course, with a 50:70 split in student enrollment in the lab between spring and fall semesters. Our undergraduate students primarily come from Florida (~90%). The gender ratio is roughly 40:60 female to male. Roughly 47% of the students identify as White, with Hispanic/Latino at 17%, Asian American Pacific Islander at 10.5%, and Black at 3.5%. Many students at UF are first generation college students, and over 70% receive some amount of financial aid during their education.

The Unit Operations 2 course is worth two credits (compared to three credits for a standard course). Students must have passed Unit Operations 1, Separations, and Process Safety prior to taking this course. Unit Operations 2 is usually taken concurrently with Process Economics (the first design course), Reactor Kinetics & Design, and Materials of Chemical Engineering, although none of these courses are a co-requisite with the lab. Students typically "mix and match" these courses to maintain certain numbers of credits due to scheduling conflicts with technical electives.

The course is taught by a single faculty member with the assistance of a lab engineer and both undergraduate and graduate laboratory assistants. These instuctors place students into groups of four based on a variety of factors, including their schedules, commitment level/desired grade, gender, and race/ethnicity, using the CATME® tool. This tool gives students a team-making survey and then instructors can tune the matching process based on which factors they want to prioritize. At UF, every chemical engineering course that includes teamwork emphasizes diversity in engineering teams, including the unit operations lab courses.

Six student groups take part in lab experiments per day. Each of these student groups is supervised by one of the laboratory assistants for the duration of the experiment, typically three to four hours. Groups rotate between six different experiments throughout the semester, with each group performing a different experiment during each rotation and each group going through all six experiments by the end of the semester. The faculty member rotates between groups throughout the lab period. The lab engineer is not responsible for supervising students but helps maintain the facilities and repairs equipment when necessary.

Students spend six to eight hours outside of class analyzing data and writing reports for each lab, according to end-of-semester surveys taken over the last two semesters. This is in line with the instructor/department expectation of three hours of outside work per credit hour. The faculty member is responsible for grading lab reports, with each student group turning in one or two reports a week. Reports are graded equally on technical content and writing quality.

Course Learning Outcomes

The course learning objectives (CLOs) for Unit Operations 2 are like those at other universities and are summarized in Table 1. To achieve these objectives, students work in groups and rotate through the following series of six experiments or modules: Liquid-Liquid Extraction, Cooling Tower, Batch Distillation, Continuous Distillation, Semiconductors 1, and Semiconductors 2. Each module has two experimental "run days," and students submit three technical reports per module. The course is offered in both the fall and spring semesters, as are all our core curriculum courses.

TABLE 1
CLOs for the Unit Operations 2 course.

CLO1	Reinforce classroom theory by the collection and use of data in practical experiments with all their inherent problems and limitations.
CLO2	Gain proficiency in writing technical reports.
CLO3	Gain experience working in teams.
CLO4	Create a sense of professional responsibility for the quality and integrity of engineering work.
CLO5	Learn safe working procedures.
CLO6	Learn equipment, instrumentation, and procedures not covered in lectures.

Rationale for This Study

Students work on separations experiments in this course, including continuous and batch distillations, that share a small control room. Due to the COVID-19 pandemic and resulting physical distancing requirements, the department had an opportunity to move one of the experiments online. Facilitated by the course's primary instructor, a group of chemical engineering faculty, including the instructor for the Progress Design course and the department chair, met regularly during Summer 2020 to discuss the course's learning outcomes and evaluate the resources available to meet this goal. Due to the conceptual difficulty of a transient batch process, the group chose the continuous distillation experiment for the online format. This presented an opportunity to enrich an existing lab experiment with some elements of the process design course.

IN-LAB EXPERIMENT - CONTINUOUS DISTILLATION

Learning Objectives

For this study the Unit Operations experiment selected was Continuous Distillation. The technical learning objectives for the in-lab version of this experiment are to reinforce and apply students' understanding of continuous distillation and demonstrate some of the intricacies of safe operation of a real pilot-size column.

Timeline and Assessments

The timeline for the in-lab experiment is shown in Figure 1. Prior to arriving in the lab, students are asked to review the manuals and documentation provided. This includes reading standard operating procedures (SOPs) and watching videos of the faculty performing the experiment step by step. Students then prepare an experimental design plan outlining their choice of variables to investigate, and they also prepare a safety analysis of the experiment as a team. This information is included in their preliminary report.

On the first run day, students run the column at total reflux to collect column efficiency data and predict the minimum reflux ratio necessary to meet the experimental objective. The specific objective varies from semester to semester, but typically involves a purity requirement and production amount for either the tops or the bottoms. This analysis is outlined in their progress report, submitted prior to the second run day.

On the second run day, students run the column at a range of values near their calculated minimum reflux ratio. They examine the relationship between product purity/amounts and their minimum reflux ratio to determine if the experimental objectives can be met. Finally, they write the final report and include a recommendation for an optimal set of operating parameters for the column to meet the experimental objectives.

Limitations

While the physical distillation columns can be beneficial to students in gaining experience with pilot-scale equipment, they also present significant challenges. Within the existing lab environment, many safety concerns and physical limitations exist that limit student creativity and exploration. Some examples include:

- Due to the size of the column, it takes a long time to reach steady state, and students do not have time for more than one or two tests.
- For safety reasons, students are not allowed to adjust the energy in (via steam) or out (via cooling water) of the column.
- The column reflux is subcooled to ~35°C, which requires a significant amount of subcooling and negatively affects the efficiency of the column.
- The feed is not pre-heated, as it typically would be in an industrial setting, and enters the column at room temperature (~30°C). This affects the performance of the feed tray and the overall process.
- Due to the height of the column, students may not change the feed tray or take a sample from any trays except the bottom two, the distillate and the reboiler. This limits the variables they can change and data they can access.
- Due to the permanence of the set-up, it is also impossible to change the number of trays, type of trays, etc.

Adding process simulation to the existing in-lab experiment could help us work around the limitations of the facilities and allow students to be more creative in their process optimization. This could deepen their understanding of the separation concepts they are applying. While students may not investigate every one of these limitations during a simulation experiment, the online experiment outlined below would allow students the ability to study the impacts of one or more of the limitations on the system's performance.

SIMULATION EXPERIMENT

Learning Objectives

The first learning objective remained the same between the two versions of this experiment. Students needed to reinforce and apply the concepts learned in the classroom. However, due to the change in modality to an online format, the second learning objective was changed from demonstrating safe operation of a real pilot-size column to exploring the relationship between experiments and simulation. This comparative skill is crucial to the students' development as engineers but is often left out of the traditional undergraduate curriculum. Instead, instructors rely on research experience to provide students with this insight.

Timeline and Assessments

The online experiment timeline is also shown in Figure 1. First, students were provided instructions on how to install the simulation software on their personal computers. To ensure smooth integration with the existing curriculum, Aspen Plus® and Aspen HYSYS were used. The installation instructions were the same as those provided to students taking the Process Economics and Process Design courses, many of whom were also enrolled in Unit Operations 2.

The preliminary report included a theoretical background section and a description of the basic experimental design

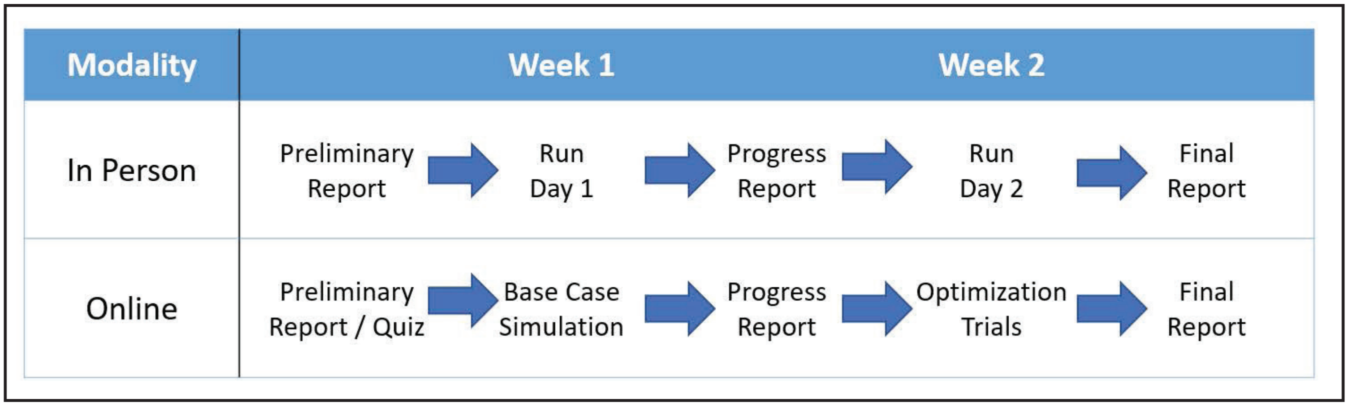

***Figure 1**. In-lab and online experiment timelines.*

variables the students planned to study. Students also had to verify that they had properly installed Aspen HYSYS and Aspen Plus.

The students were then given the background material from the in-person distillation experiment to provide them with insight into how the experiments were done originally and where the experimental data they were going to use came from. This information also helped students understand possible sources of error and inefficiency with the existing equipment that may not be present in the simulation.

Then, a separate set of instructional videos guided students through the process of creating a base case simulation using experimental data. The first video introduced the problem statement and outlined the objectives of the simulation experiment. The students were then shown how to add components and a fluid package to an Aspen HYSYS simulation. Students learned how to compare, select, and validate activity coefficients to supply to the simulation. A shortcut distillation column, a common feature in process simulation software, was then used to estimate a minimum reflux ratio as a starting point for their case studies. Finally, students simulated a full distillation column, including features of the existing column such as number and type of trays and degrees of subcooling in the condenser. Generally, students were easily able to converge this simulation since the separation is straightforward (no azeotrope) and easily achievable with our system.

The progress report outlined how the group created the base case simulation, including descriptions of the activity coefficient validation, decanter test, and shortcut column steps. Then, starting from the preliminary operating parameters that allowed the simulated column to match the experimental data, the students expanded on their original experimental design with more specific variables to study.

For the final part of this experiment, the students were asked to optimize the simulation for a given objective. The learning objective was to explore optimization in a space where they were not limited by the physical realities of the existing column. For example, some groups chose to optimize the feed temperature, something that is not possible with our existing system and presents a safety risk in person. Other examples of optimization included changing the number of trays and pressure in the column. Students were also able to study more variables across a wider range than they can normally do in the lab, where time constraints only allow for testing one or two levels of a variable.

In their final reports, students presented the results of their case studies, including the sensitivity to each of the tested variables, a brief economic analysis of their tested parameters, and their recommendation for the operating parameters the company should use for their optimization objective, based on both feasibility (whether the process could achieve the desired objective) and practicality (whether the process was efficient in terms of time, equipment, money, etc.).

Experimental Objectives

The first time this module was offered in Fall 2020, every group completed the simulation experiment as their first lab module. This was to allow a two-week buffer period between the start of the semester, when students were traveling back to the university, and the first time they came to an in-person lab. Since it was so early in the semester, the module's experimental objective was only to optimize the column for the existing experiment, where the goal was at least 2000 kg/week of 99% IPA distillate purity. This imitated the way a process engineer would optimize an existing process. While students were not prohibited from making changes to the equipment, they were advised to consider the economic impacts of their recommendations. Minor modifications to an existing process or piece of equipment, such as changing the feed tray or adjusting the reflux ratio, are much cheaper than major changes, such as adding trays, adding another separation process before or after the existing column, changing the tray types, etc.

In Spring 2021, this experiment was offered as part of the lab's regular rotations. Therefore, the optimization objectives were increased in difficulty as the semester progressed to match the students' increasing knowledge of the simulation software from their Process Economics class.

- Rotations 1 and 2: The first objective was the same as the Fall 2020 semester, where students optimized for at least 2000 kg/week of 99% IPA distillate purity.
- Rotations 3 and 4: The second objective was to optimize the same system for a different purity feed and different purity goal. This was to imitate how a company may change product specifications, perhaps based on market demand or feedstock fluctuations, and how a process engineer would need to adjust the process accordingly. The chemicals used in the experiment remained the same.
- Rotations 5 and 6: The third objective was to change the system to a new set of chemicals and optimize the process accordingly. This was a step further into process development where the same equipment and unit operation can be used for a different product/process and demonstrates to students one of the initial steps in production line changeover.

CURRICULUM AND STUDENT DATA ANALYSIS

It was necessary to ensure that our new simulation module still met all the course learning objectives listed in Table 1. The faculty involved in planning the module included the current course instructor, the undergraduate academic advisor, and the department chair. Together, we mapped the CLOs and ensured alignment between the in-lab module and the simulation module.

At the end of the Fall 2020 semester, students were surveyed via an anonymous Canvas® survey for specific feedback related to the simulation module. The results were generally positive, with some areas for improvement noted.

The two common issues identified in the comments were that the "sandbox" optimization was left too vague and that the experiment was either too easy or too difficult depending on where the students were in the Process Design sequence. Both problems were addressed in the Spring 2021 iteration of the experiment.

First, an additional video was created that guided students further into the optimization portion of the experiment. The lab instructor walked students through an example where a distillation column with different chemicals and parameters was optimized. Leading students through an example of this optimization greatly reduced confusion for the students enrolled in the Spring 2021 iteration of the module. In the video, the lab instructor also emphasized multiple times that students should ask questions if they were confused.

This experiment could be used in the case of students with physical disabilities or illness... In a discipline where hands-on labs are the standard, we need to be mindful of the accessibility of our courses and work towards becoming more inclusive.

Second, the instructors set new optimization objectives that increased in difficulty every two rotations of the course to better match the students' growing proficiency with the Aspen software and their progression through the Process Economics class. These objectives were outlined earlier in the Experimental Objectives section of this paper.

Students were also surveyed at the end of the Spring 2021 semester. Overall, the new optimization objectives were well received by the students and led to less difficulties with the module. However, the third objective (Rotations 5 and 6) confused many students. This confusion was expected to some degree since it is beyond the usual scope of the undergraduate curriculum to adapt old equipment for a new process. If the module is used in the future, further material or another video will be created to provide further explanation and reduce confusion.

Almost every student who left comments in the Spring 2021 survey noted that this was the only experiment run online and that they would prefer the experiment to be in person. For context, during the Fall 2020 offering of the course there were two other experiments also moved online where students were sent old data and asked to analyze it. In Spring 2021 those two other experiments were brought back into the lab, and only the continuous distillation experiment remained online.

STRATEGIES AND RECOMMENDATIONS

We have returned to a fully in-person modality in the Unit Operations lab courses. This is the format clearly preferred by our students. However, it is important to consider what students gained from the online version of this experiment and preserve some aspects of that experience.

In the future, the in-person experiment could incorporate a virtual simulation component along with the hands-on lab. Students could use their experimental data from the first day to generate a simulation. This could then be used to find a minimum reflux ratio and other optimal operating parameters for the second run day. The simulation could be done in addition to the "by hand" analysis (McCabe-Thiele diagrams). Students could then compare and validate their results and better predict the column's behavior for the second day of experiments.

This experiment could also be used in the case of students with physical disabilities or illness (such as COVID-19) that prevent them from participating fully in the in-person modality of the lab. In a discipline where hands-on labs are the standard, we need to be mindful of the accessibility of our courses and work towards becoming more inclusive.

Alternatively, this exercise could be incorporated into other classes. Rather than incorporating simulations into an experimental lab, the data could be incorporated into a process design simulation. A modified version of this simulation could even be given to a separations class to introduce both simulation software and experimental data earlier in the curriculum.

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