UNIT OPERATION EXPERIMENT LINKING CLASSROOM WITH INDUSTRIAL PROCESSING

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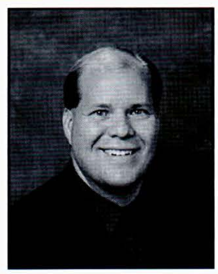
In an effort to provide a link between classroom/textbook learning and real-world application, senior-level chemical engineering students at Lamar University performed a unique crossover experiment within their unit operations laboratory. Adjacent to Lamar University is a two-year technical school, Lamar Institute of Technology (LIT), that has a training program for technicians going into the chemical operations occupations. As a teaching tool, LIT has a 20 ft tall, 1 ft diameter packed distillation column (Figure 1), complete with reboiler, pumps, holding tanks, fin fan, control valves, and Distributed Control System (DCS) control board.

The students were tasked with performing a non-structured (*i.e.*, open-ended) unit operation experiment that would challenge their ability to apply knowledge learned in the classroom to a distillation system that would be comparable to that found in industry. By breaking into 4- or 5-membered teams, a class of 19 students could evaluate several parameters, including feed flow rate, reboiler temperatures, and vacuum pressures. The overall objective was to suggest ways of operating the column, which distilled propylene glycol and water, with greater efficiencies and lower energy costs. LIT and Lamar University would mutually benefit by the students' findings —LIT with better operating parameters and Lamar with a challenging unit operation experiment.

In recent years, the availability of laptop computers and chemical engineering simulation software, such as Aspen Plus, has brought about a decrease in physical chemical engineering unit operation laboratory experimentation with an increase in virtual laboratory simulation. Virtual labs create a "lab anywhere" atmosphere whereby students can redo experiments or try out new parameters far more cheaply than performing the actual experiment.[1] There are, however, pros and cons to this concept. Complex concepts, such as vapor liquid equilibria, are easily understood[2] using Aspen Plus, which is the standard in the chemical process industry and should be incorporated throughout the chemical engineering curriculum.[3,4] As Feisel and Rosa pointed out, however,



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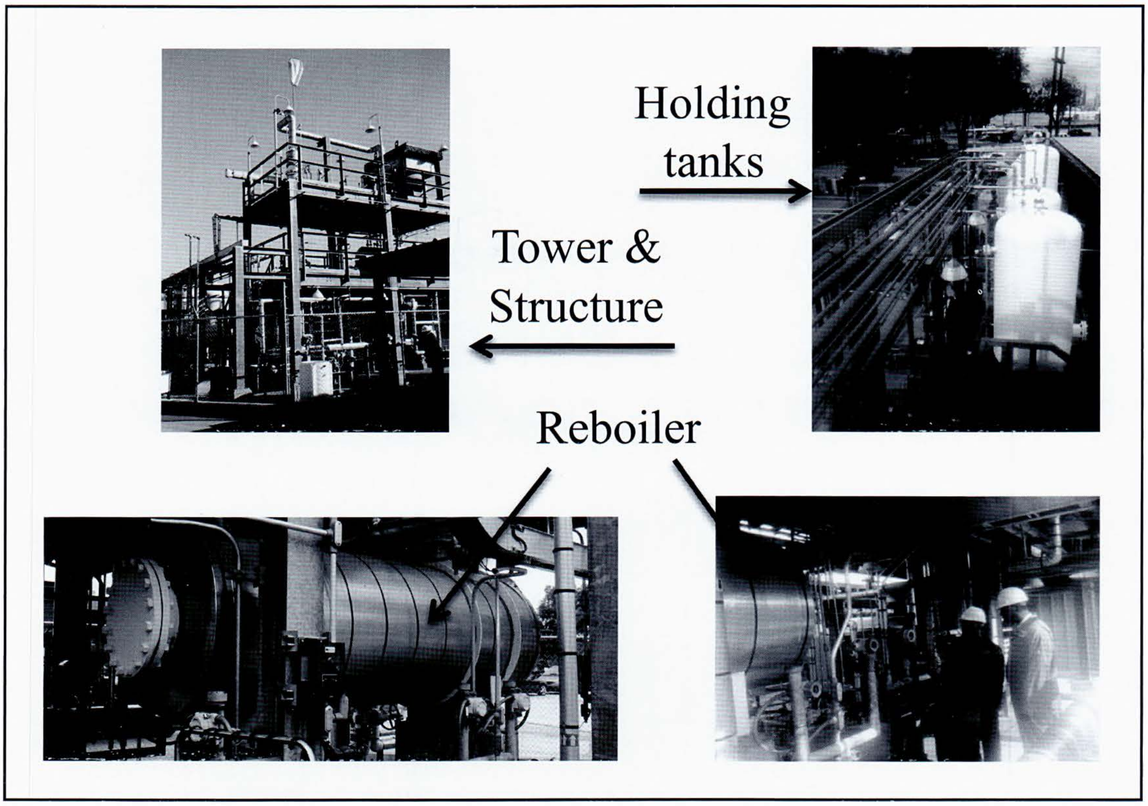

Figure 1. *Pictures of the distillation unit illustrating the relative size of the vessels and personal protective equipment worn by the students. The distillation column is tucked within the steel structure shown in top left.*

simulation cannot completely replace physical, hands-on experience. A chemical engineering student that is ready for the industrial world is one that has sufficient aptitude for safety, communication, teamwork, and sensory awareness issues that are best learned within the physical laboratory.[5]

By giving students a limited amount of information, we required them to assess what additional information was needed to solve the problem at hand. This open-ended nature was designed to enhance creativity among the students and to empower them as young engineers through the development of their own unique experiment.[6] Teamwork, chemical process safety, increased communication skills, and sensory awareness were the goals of this experiment. (Sensory awareness is the use of our human senses to gather and interpret experimental results with the purpose of making well thought-out, engineered decisions.)

DESCRIPTION OF THE EXPERIMENT

As a final laboratory experiment during their senior-level unit operations class, the four teams each had a specific parameter to investigate (Table 1). The main objectives of the experiment were 1) to increase problem-solving skills for distillation systems and 2) to bolster teamwork and communication skills. These objectives were developed to engage the students' ability to think outside the typical classroom setting. Distillation also served as a great example for teaching energy conservation as distillation accounts for 20% of the energy consumption by the U.S. manufacturing sector.[7]

The students were provided a short list of operating parameters, which included feed composition (60 wt % propylene glycol) and typical column pressure (28.05 in Hg). Since the density (*i.e.*, specific gravity) of a mixture changes with respect to changes in concentration, product purity was measured by specific gravity of the propylene glycol stream using a coriolis meter. The students were provided this information, along with the process flow diagram (Figure 2), two weeks prior to the experiment. As student safety was a top priority, students were required to use the personal protective equipment (PPE) appropriate for this facility. This included hard hats and safety glasses, which were already part of the unit operations laboratory protocol. Other PPE required were safety shoes, flame retardant clothing, and leather gloves for climbing up

TABLE 1
Description of the testing parameters

Team #	Parameter to be tested	Comment
1	Feed Flow Rate	Measured density and flow rate of bottoms product
2	Column Pressure	Measured column temperatures and density of bottoms product
3	Reboiler Temperature	Measured density of bottoms product and reflux rates (liquid feed kept constant)
4	Feed Location	Aspen 7.0 process modeling program

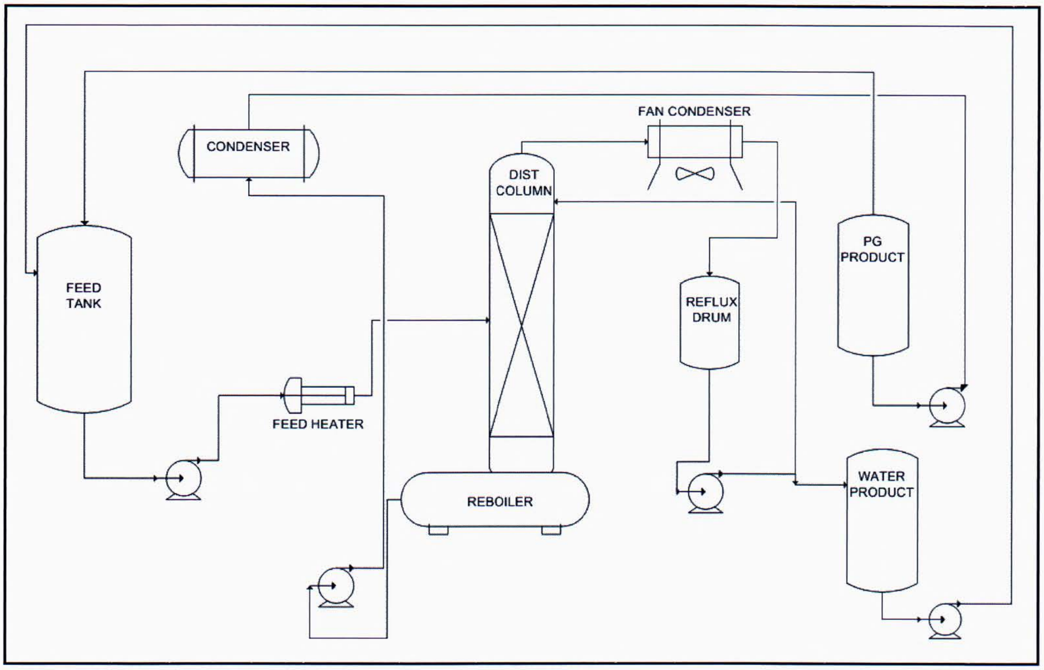

Figure 2. *Process flow diagram for the packed column distillation unit.*

stairs and ladders. Many of the students used flame retardant clothing obtained from their co-op or intern positions. Others were able to borrow what was needed.

On the day of the experiment, the class assembled at the experiment site. The student teams were organized into inside DCS operators and outside crew sub-teams. The sub-team members used two-way radios to maintain constant communication. The inside sub-team took turns making step changes from the DCS control board, and the outside crew monitored the effect on product purity using field readings from a coriolis meter. The coriolis meter was only readable locally, as there was no connection to the DCS board. The total time for the experiment was approximately six hours, most of which was spent waiting for the process to come to steady state after a step change was made. All operating results shown are those obtained from the students' experimental results.

RESULTS OF EXPERIMENTS

Team 1's task was to evaluate the feed flow rate, given a constant feed concentration, on purity of the bottoms product. The feed flow rate generally used by LIT is 45 gph and was taken as the starting point. Step changes of 5 gph were made above and below the starting point with a range of 30 – 60 gph. The students found that as the feed flow rate increased, so must the reboiler steam flow rate, linearly, to maintain proper operating temperature. The optimum feed rate was found to be 50 gph, and the estimated steam usage would cost ~$32,850 per year for a continuously operating column. One can see in Figure 3 where regions of flooding and entrainment, which lead to poor efficiency, occurred due to changes in feed flow rates. Students of this group were able to identify the loading and flooding regimes based on product purity. What was believed to be entrainment and flooding led to maldistribution of the vapor and liquid streams causing poor vapor-liquid equilibrium within the packed column.

The effect of column vacuum pressure was the target of Team 2's investigation. Vacuum distillation increases the relative volatility of the distilling components, thus lowering temperature requirements.[8] In the case of aqueous/organic mixtures, however, underwetting can be problematic with pressures < 2 psia. Underwetting, a phenomenon due to high surface tension, causes height equivalent to a theoretical plate (HETP) to increase towards the aqueous end of the column.[9]

The distillation system was found to be sensitive toward steam rate and vacuum. As vacuum decreased, distillate and bottoms temperatures increased, as well as bottoms product density. Higher vacuum produced higher product purity and needed lower reboiler duty for a particular product purity. Although vacuum distillation can be quite expensive in energy costs, it is often used in the distillation of crude petroleum where vacuum columns are operated at reduced temperatures, thus preventing the cracking of paraffins to olefins.[10]

The students realized from this exercise that higher vacuum pressures are difficult to maintain over extended periods of time due to potential leaks within the equipment. This illustrated mechanical limits to a chemical process, which would

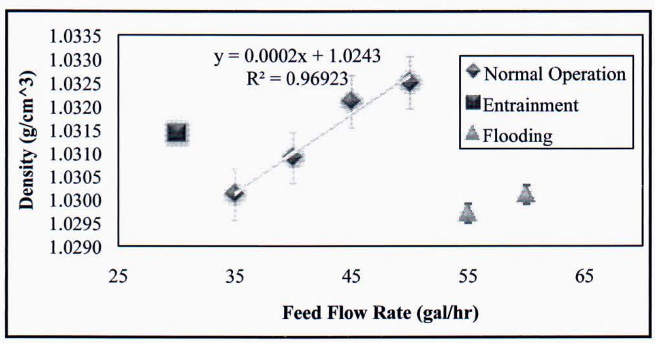

Figure 3. *Effect of feed flow rate on density. The outliers were taken as conditions of poor liquid/vapor contact.*

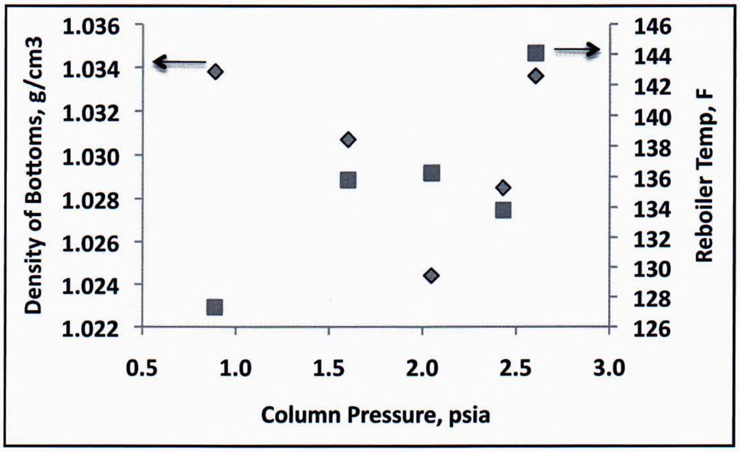

Figure 4. Effect of column pressure on product purity and reboiler duty (F = 30 gph).

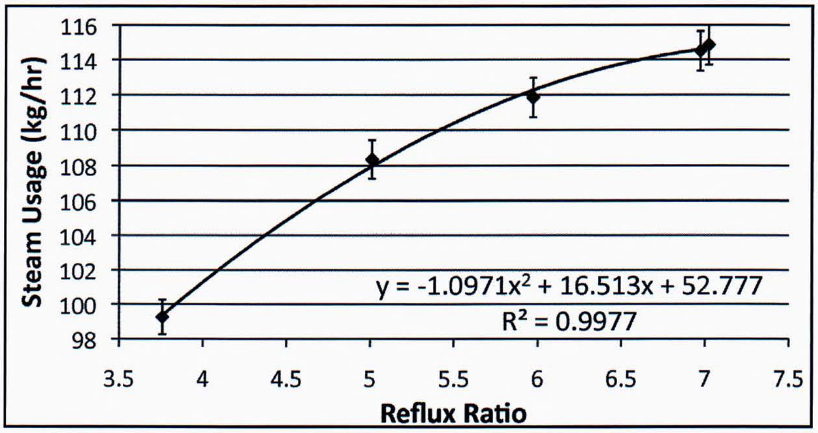

Figure 5. Effect of reflux ratio on steam usage. Operating conditions: F = 30 gph, P_{col} = 27.4 in Hg.

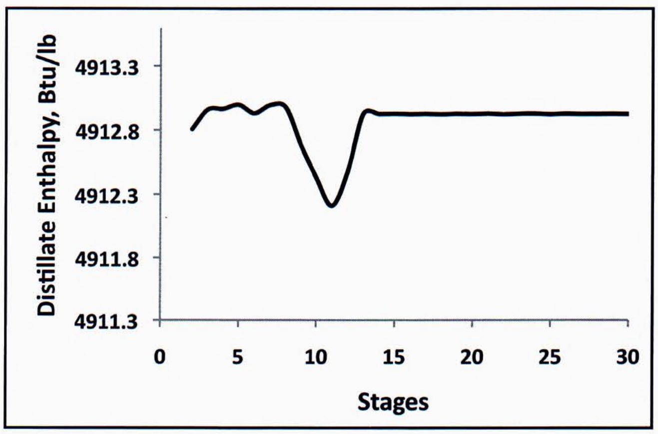

Figure 6. Aspen modeling results demonstrating the enthalpy of the distillate stream at various feed locations. The minimum, found at Stage 11, is the optimum feed stage. Note that due to a relatively easy separation, there are only marginal differences in enthalpies for various feed stage locations.

not be as quickly learned in a classroom setting. As can be seen in Figure 4, a high product purity can be achieved either by reducing the column pressure (P = 0.8 psia) while maintaining a lower reboiler temperature (T = 127 °F) or vice versa (P = 2.6 psia, T = 144 °F). Intermediate values of temperature and pressure resulted in poorer product purity.

Energy efficiency and energy conservation with emphasis on the column's reboiler were the focus of Team 3. Reboilers drive separation by providing heat to the column thus creating vapor traffic in the distillation column. Higher vapor velocities improve vapor-liquid contact through high dispersion but can lead to excessive entrainment of liquid in the vapor and high pressure drops in the column. It should be noted, however, that if one increases the fluid velocities inside the column, the contact time (*i.e.*, space time) between both phases decreases, resulting in poorer separation. In addition, higher reflux ratios require more reboiler duty to vaporize the additional liquid inside the column. Steam usage vs. reflux ratio is graphed in Figure 5 and was fitted using a quadratic equation. Results indicated that, on an annual basis, the incremental cost of additional production was $13,500 for 30 vs. 55 gph (feed rate). Also, $2,000/yr could be saved if 4 vs. 7 gph (reflux rate) was used. (Steam costs taken as $7.70/GJ.)

Team 4 had a slightly different task and that was to determine the optimum feed location; however, the feed line to LIT's distillation unit entered at a single point only. The column was originally designed for the separation of ethylene glycol and water, but propylene glycol was substituted for environmental reasons. The actual column packing was a structured wire mesh material manufactured by Sulzer Chemtech, Ltd. (Humble, TX); however, specific information concerning the column packing was not known. For modeling purposes, therefore, 1-inch Raschig rings were assumed. From the outside of the column, the students measured and determined that the column had ~14 ft of packed bed height and the feed was located 5 ft from the reboiler. Aspen Plus was used to model the process using the Non-Random Two Liquid (NRTL) thermodynamic model. To determine the optimum feed location, the enthalpy of the product streams was compared at different feed stages (Figure 6) and then applied the HETP calculation for a packed column.[8] This was a small column (*i.e.*, $D_{col} < 0.67$ m); therefore, HETP = $18{*}D_p$, where D_p and HETP are in meters.[11]

In distillation process optimization (*i.e.*, once column is built and in operation), one does not want to sacrifice product quality simply for lower production costs. Aspen results indicated that Stage 11 was the optimum, which is equivalent to

5.5 ft. Therefore, the team found that the current feed location is appropriately placed even though the column was originally designed for another purpose.

If one considers the McCabe-Thiele analysis for binary distillation, the optimum feed tray location will be the tray (*i.e.*, stair step) where the Top and Bottom Operating Lines intersect. If the feed stage is too low (or too high), however, additional equilibrium stages will be necessary to achieve a desired separation. For a column that is already built and in use, this means that the desired separation will not be achieved for a feed stage improperly located. Therefore, one would expect a difference in the enthalpies of both the top and bottom products due to the thermodynamics of mixtures at various concentrations. In this exercise, the students chose to model and report their findings based upon enthalpy rather than convert to a concentration.

ASSESSMENT OF THE EXPERIMENT

For assessment, the students completed a questionnaire that consisted of questions concerning appropriate health and safety, increased communication skills, and independent thinking skills. A 5-point Likert scale was used with 1 being "strongly disagree" and 5 being "strongly agree." Of the 19 students, 16 responded to the questionnaire. The students rated the laboratory experiment a 4.37 (out of 5), indicating a good overall rating for the experiment. The students were very positive with the competency of instructors, the amount of information given prior to the experiment, and health and safety measures. Some of the responses, however, indicated that additional experiment time would have provided better results. This was also made apparent during the poster presentations. A summary of the students' responses can be seen in Table 2.

An overall assessment by the instructors was that the students developed increased interpersonal communication skills with the team/sub-team structure. While half the team was inside at the control board, the other half was outside monitoring various gauges and reporting back to the inside team members. Giving the students flexibility to establish testing parameters using a unit operation most likely to be found in industry allowed them to feel more like engineers, and less like students. Realization of integrated mass and heat flows (*i.e.*, changing one variable, such as reboiler temperature, also changed required reflux ratio and/or feed flow rate) by the students was also accomplished. In addition, the students had a heightened sense of personal safety, both for themselves and their fellow team members. With ABET's new emphasis on process safety, this was certainly an added bonus.

POSTER PRESENTATIONS

Each team was required to present, as a team, a poster demonstrating their results. This was an exercise to enhance the students' ability to give oral presentations with the hopes of less anxiety within a group format. The teams were asked to allow for approximately 3 minutes of speaking time for each team member. The presentations were scored based on format and layout of posters, speaking time for each member, and responses to questions. According to the questionnaire, most students responded very positively to the poster presentation format; however, 2 out of 16 respondees reported that the poster was no better or worse in terms of nervousness than a typical oral presentation. One comment made was "a presentation is still a presentation." Overall, the posters had good visual quality and were easy to follow. The students appeared to be more relaxed during these presentations compared to oral slide presentations that were also a requirement of the Unit Operations class. Individually, however, some students tended to rely more on fellow team members for technical explanations of the experiment. Subsequently, technical questions can be asked by the instructors to separate those who have been diligent thinkers and those who were not.

SUMMARY

This paper describes a Unit Operations experiment for linking classroom learning with industrial application. The students gained hands-on knowledge for teamwork, process safety, sensory awareness, and communication. Although much of chemical engineering education is focused on segregating each unit operation from a process, each of those units come together to form that process, and what changes are made to one unit will affect the remainder of the process units. With this experiment, students were able to see these intertwined facets firsthand. The chemical engineering students at Lamar University are chiefly employed within the many local chemical process industries of Southeast Texas. Prospective employers, therefore, would hold this type of experiment in high regard. Future iterations of this experiment, or ones similar, are expected to include "other" uses for old or existing columns and multi-component separations.

TABLE 2
Summary of student responses to questionnaire

Statement	Agree (%)	Neither (%)	Disagree (%)
1. This experiment led to increased experimental design and problem-solving skills in a real-world setting	84	11	5
2. Sufficient information was provided prior to experiment and instructors were sufficiently competent	89	11	0
3. Experiment is likely to be discussed with potential employers and should be used for future laboratory classes	90	8	2
4. This experiment led to increased teamwork and communication skills development	88	8	5

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