AN AUTOMATED DISTILLATION COLUMN
For the Unit Operations Laboratory

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Distillation is one of the most common separation processes; hence, it is important for undergraduates to have some hands-on exposure to this unit operation during the course of their studies. Additionally, working with batch distillation towers provides students with an opportunity to experience and learn about a dynamic process that is widely used in the pharmaceutical and specialty chemical industries. It is equally important for undergraduate students to work with automated processes, since similar control features are commonly implemented in industry to reduce labor costs, provide greater processing flexibility, and improve process safety and product purity.

For these reasons, an automated batch distillation column was designed and constructed on-site for the Clemson Unit Operations Laboratory (UO Lab). The pilot-scale tower uses sieve plates to separate a mixture of 2-propanol (IPA) and 1-propanol (NPA). Some background information about the Clemson curriculum should be mentioned before going into more depth about this particular experiment. First, the senior-level UO Lab course consists of groups of students (typically three to four students per group) conducting four experiments during the course of the semester. Lab groups develop their own experimental procedures to accomplish the assigned objectives. They are given three three-hour lab periods to conduct each experiment, and the results of these experiments are presented either in writing or orally in front of a panel of their peers and professors.

Having three lab periods to perform each experiment provides students with the ability to explore different aspects of a particular process and to collect enough data to explore statistical variations. Additionally, the process control course, while providing students with both the practical aspects and fundamental mathematical principles of control, has no lab associated with it. Therefore, students can only simulate how a change in a manipulated variable affects a process control variable using software such as Control Station.[1] Thus, the senior-level UO Lab course incorporates process control concepts into several of the classical unit operations so that students gain hands-on experience working with and tuning controllers in automated chemical processes. Ultimately, this better prepares them for work with complex, real-world processes than would conducting experiments with idealized processes, such as simple tanks in series.

EQUIPMENT

Though the basic design for the batch distillation apparatus was developed by Clemson faculty, the detailed design and most of the construction was accomplished by undergraduates involved in the project.[2] This afforded the students an opportunity to gain hands-on knowledge about metal machining and process engineering. The apparatus required approximately six months and $25,000 to build. The key components of the apparatus (shown in Figure 1) include

- *A 180-liter jacketed vessel (Owens Mechanical & Fabrica-*

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tion), which serves as the reboiler

- *A 4-in. diameter glass distillation tower (Labglass) with six aluminum sieve plates vertically spaced 6 in. apart*
- *A single-pass shell-and-tube heat exchanger using water coolant in the tube side*
- *Coriolis flow meters (Micromotion CMF025) and pneumatic control valves (Fisher Rosemount 5100 valves with 3661 positioners) on both the reflux and distillate lines*
- *Data acquisition and control hardware and software (National Instruments).*

All vapor lines are 2-in NPT pipe and all liquid lines are 1/2-in NPT steel pipe. Additional information, such as vendor addresses, a wiring diagram, and a more detailed equipment list, are available upon request from Professor Bruce.

DESIGN METHODOLOGY

The chemical system as well as several key components of the batch column were chosen to be similar to those previously used in another pilot-scale distillation apparatus in the UO Lab. This was done because the previously built column had proven to be very safe to operate and there were well-established performance characteristics, such as optimal flow rates, heat duty for the condenser, and stage efficiencies. Specifically, the IPA/NPA binary system was chosen because: 1) the chemicals are relatively inexpensive; 2) they have low toxicity; 3) they have high short-term exposure limits (> 250 ppm); 4) fires can be extinguished by water, dry chemical powder, or CO_2; 5) they have moderate vapor pressures (< 45 torr) at STP conditions; and 6) mixture compositions are easily analyzed by gas chromatography.

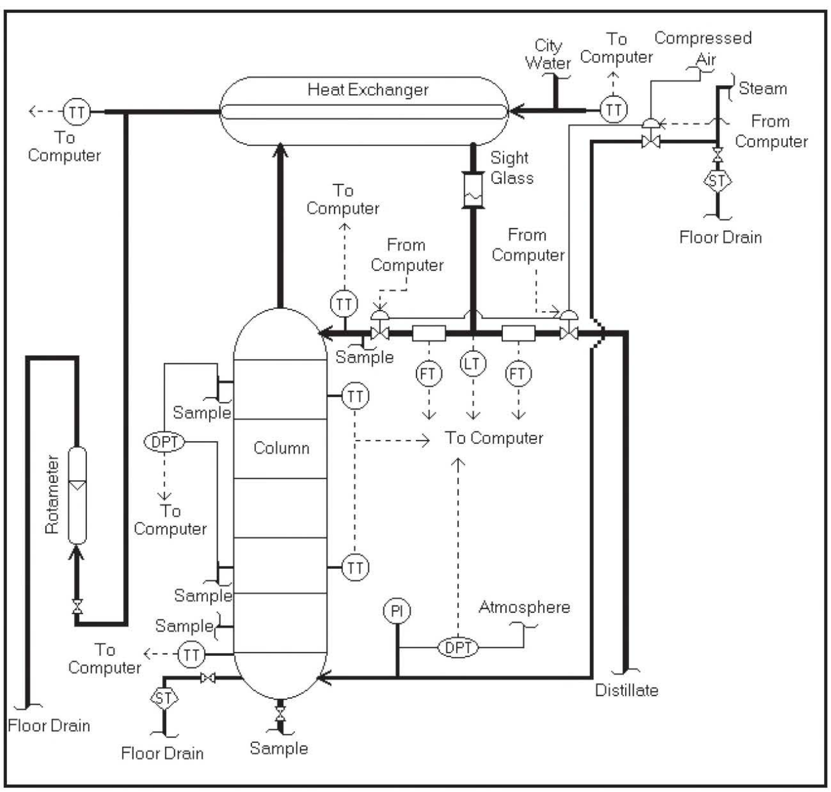

Figure 1. *Schematic for batch distillation apparatus.*

The IPA/NPA system also exhibits relatively ideal behavior as indicated by the vapor-liquid equilibrium (VLE) data shown in Figure 2. This figure includes both experimentally measured data[3] and compositions predicted by a Margules activity coefficient model.[4] Binary parameters calculated by Gmehling[4] for other activity coefficient models are shown in Table 1 along with the mean deviations associated with each model. As can be seen in the table, all of the activity coefficient models do an excellent job at describing the VLE data for this non-azeotropic system.

Another key design feature was the choice to use gravity, rather than a pump, to return reflux flow to the column. This necessitated that the pressure drop across all flow measuring/control devices be kept to a minimum. For this reason, Coriolis flow meters were chosen both for their accuracy and their low-pressure drop character istics. An added bonus in using the Co-

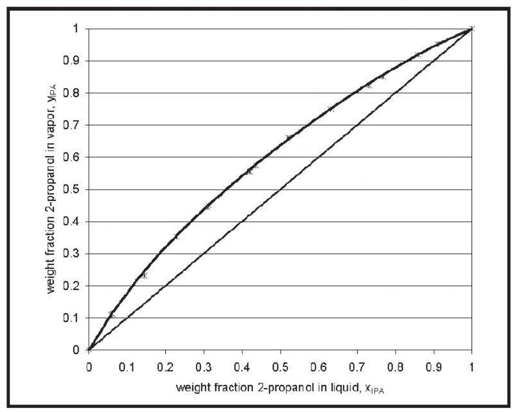

Figure 2. *Vapor-liquid equilibrium data for mixtures of 2-propanol and 1-propanol at 1 atm. Curve corresponds to compositions predicted using the Margules parameters listed in Table 1 and (*) represent experimental data collected by Ballard and Van Winkle, 1952.*

TABLE 1
VLE Binary Parameters for Activity Coefficient Models of the 2-propanol/1-propanol System[4]

Activity Coefficient Model	*Binary Parameters* $A_{IPA\text{-}NPA}$	*Binary Parameters* $A_{NPA\text{-}IPA}$	*Mean Deviation from Experimental Vapor Fractions*
Margules	0.1321	-0.0621	0.0046
Van Laar	0.0100	25765.3800	0.0090
Wilson	818.3291	-481.0590	0.0048
UNIQUAC	-399.2278	575.0956	0.0050

riolis flow meters is that they can not only measure mass flow rate, but also volumetric flow rate, density, and temperature of the fluid in the pipes.

The reflux and distillate control valves were selected for their ability to control low flows with a very small pressure drop across the valve. At first, an actuated ball valve was considered to allow for the low-pressure drop requirements, but after talking with several vendors it became apparent that a ball valve would not be a viable option, due to sizing difficulties. The selected needle valves were chosen based on their ability to meet the above criteria, specifically, a low-pressure drop across the valve and integral positioners for adjusting the needle.

The Labview data acquisition and control software (National Instruments) was chosen because it has several key features that make it attractive for use in the UO Lab. These include low cost, widespread use across campus, user-friendly graphical programming language, and most importantly, preprogrammed algorithms for PID control of specified measurables. Labview can also log data that is acquired during an experiment (*e.g.*, temperatures, flow rates, and pressures) and store them in an Excel spreadsheet. This not only allows students to have more time during the lab period to observe column dynamics, but it also allows for post-lab analysis to be conducted virtually instantaneously.

Another important aspect of Labview is the graphical user interface, which allows students to look at pictorial representations of numbers (*e.g.*, a virtual thermometer) instead of simply looking at the raw data/numbers. This interface also allows for plots of time variations in process variables to be continuously displayed so that students can easily observe when the process or a measured variable reaches steady state.

Finally, safety limits can be programmed into the software, meaning that a student would not be capable of running the column under dangerous conditions, such as an over-pressurized reboiler. If a student did try to run the column at an unsafe condition, Labview would override the student's attempt and bring the column back within safe operating limits. A copy of the distillation control program used in Labview can be obtained from Professor Bruce.

MODES OF OPERATION

There are four basic modes of operation and control for the column: total reflux, constant distillate flow rate, constant distillate composition, and fixed reflux ratio. For all modes of operation, the liquid level in the sight glass is maintained at a setpoint using either the distillate or the reflux valve. Additionally, the column is currently operated so as to maintain a constant reboiler steam pressure (4-6 psi) for all modes of operation. Future experiments may examine the possibility of manipulating the steam pressure to control the reflux or distillate rate.

Initially, the column is started in total reflux. This is the simplest mode of column operation and involves control of only the reboiler steam pressure and liquid level in the sight glass. This mode allows the rising vapor and falling liquid to heat the tower. Once the column has reached steady state, the overall tray efficiency can be determined from composition analysis of samples collected from the top and bottom of the column. By varying the setpoint of the reboiler steam pressure, it is possible to determine how tray efficiency varies with vapor flow rate.

The column can be operated in three other modes. Constant reflux ratio is used least often because it requires that the distillate and reflux valves be adjusted to maintain a constant fluid level in the sight glass as well as a fixed reflux ratio. Noise and interaction make this mode difficult to tune and operate in a satisfactory manner.

Rather than filter the signals from the two Coriolis flow meters to improve control, a simpler yet related mode of operation has been used more commonly by the students. This mode of operation maintains a constant distillate flow rate, which is essentially the same as maintaining a fixed reflux ratio if the steam pressure remains constant and the composition of the pot does not vary significantly over the course of the lab period. The control scheme for constant distillate flow is very straightforward; the reflux valve maintains a constant fluid level in the sight glass, while the distillate valve controls the distillate flow rate. Common operating conditions maintain a distillate flow of 5 kg/hr and a reflux ratio of approximately 3. Running the column at a constant distillate flow rate allows the students to see how column temperatures and both the distillate and reboiler composition change over time.

The final mode of operation is constant distillate composition. This is accomplished by adjusting the distillate flow rate to keep the temperature at the top of the column constant, while using the reflux valve to control the fluid level in the sight glass. The purity of the distillate product is checked periodically by GC to adjust the temperature setpoint or ensure that the composition is not varying.

ASSIGNMENTS

As described earlier, each student group has three lab periods to work with the distillation column. Essentially, this allows for three different experiments to be run by each group. During the first day, students typically run the column in total reflux with preset controller tuning parameters and note changes in column operation and the visual appearance of liquid hold-up on the trays at different reboiler duties (*i.e.*, different reboiler steam pressures). Three or four conditions can be tested after the initial 30- to 45-minute start-up time for the column to reach pseudo steady state. Temperatures are monitored and samples of liquid and vapor are collected for GC analysis from the sample ports indicated in Figure 1.

On the second day the students start the column at total reflux and then shift to either a constant distillate flow or constant distillate composition mode to accomplish a particular assigned objective. The third day is typically devoted to basic closed-loop tuning exercises on the control loops (reboiler steam pressure, sight glass fluid level, distillate flow, or distillate composition). The controller tuning assignment can be made somewhat more challenging by making it the first-day task with little or no guidance on where to start.

RESULTS/DISCUSSION

Experience during the first two semesters of operation has confirmed some of the design objectives, while at the same time revealing a few surprises. Overall the column has performed very well. The six trays yield approximately 4 equilibrium stages with the pot functioning as a 5^{th}. The 60 to 70% range of tray efficiencies is consistent with predictions of various correlations for overall plate efficiency.[5] The constant distillate rate scheme can provide a distillate composition ranging from 60 to 80% IPA with an initial pot composition of approximately 25% IPA. The constant composition scheme can provide a small amount of product up to 70% IPA with the expected tradeoff between product quantity and purity.

During initial column operation, the severity of reset windup[6] in the control loops was not anticipated. The basic controller logic provided by Labview did not include anti-windup, and the column was started with each loop configured for PI control. As a result, both the steam pressure and sight glass level control loops substantially overshot their setpoints, and the students were puzzled as to why the control loops "didn't work." It is relatively easy to program anti-windup measures into Labview, but the faculty decided not to do this. Having the students experience setpoint offset and reset windup first-hand rather than just hearing the principles behind them in class is thought to be highly beneficial.

The students are initially told to turn off integral action before starting the program, and leave it off until each control variable nears its setpoint. This allows them to observe the offset, which disappears when integral action is added. Later they are told to make substantial changes in setpoint, which allows them to observe reset windup when the controller fails to reverse the control action until the controlled variable is well past the setpoint.

Another anomaly in the column is a tendency to drift and exhibit hysteresis during total reflux operation. This means that the "steady state" conditions seemingly change during the course of a lab. In essence, students believe that they have reached a steady-state condition as evidenced by constant fluid level in the sight glass and apparently constant temperatures and flow rates, but closer observation shows that the temperatures and flow rates tend to drift slowly. Also the apparent steady-state temperatures and flow rates one achieves at a particular reboiler duty/steam pressure may actually depend on the history of the steam pressures used since startup. The main reason for this anomalous behavior is thought to be heat loss from the uninsulated column. Essentially, each glass section has to heat up to the temperature of the vapor and liquid inside the column before it can correctly be considered to be at steady state. To minimize this drift and cut down on startup time, students are initially told to start the reboiler with a high steam pressure of 10 psig until condensate begins to leave the condenser, and then return the steam pressure to the desired operating condition.

Once the column has reached steady state, the response time for the controlled process variables is rather fast. The reboiler steam pressure, distillate flow rate, and sight glass fluid level respond fully to a change in setpoint in less than a minute. When the steam pressure is changed, however, it takes approximately 5 to 30 minutes for the column to reach a new steady state, depending on the magnitude of the change. When controlling the exiting vapor temperature (distillate composition) at the top of the column, the response time is approximately one to two minutes depending on the size of the change in setpoint.

An example of batch column performance when operated in constant distillate flow rate mode is shown in Figure 3. The column was initially allowed to reach steady state conditions at total reflux before data collection began. Specifically, Figure 3 shows how changes in distillate flow rate and sight glass level setpoints affect the sight glass level, reflux mass flow rate, temperature on the top tray of the tower, and distillate mass flow rate. The following changes in setpoint were used for this study: 1) at 48 seconds, the distillate flow rate setting was increased from 0 to 5 kg/h; 2) at 86 seconds, the distillate flow rate was increased further to 10 kg/h; 3) at 402 seconds, the sight glass level setpoint was changed from 28 to 27 inches; 4) at 546 and 599 seconds, the sight glass level setting was increased by 1 inch; and 5) at 734 seconds the distillate flow rate was changed from 10 to 7 kg/h. As mentioned earlier, the column rapidly adjusts to changes in both distillate flow rate and sight glass level; however, the temperature within the column and purity of the distillate stream are much slower to respond, as evidenced by the slow rise in temperature of the top tray of the tower. This increase in temperature is the result of less separation in the tower, which is caused by the reduction in reflux ratio. Additionally, it can be observed that the sight glass level controller is tuned much more tightly than one would normally operate in industry. Most industrial operators would prefer to let the level adjust slowly and not subject the column to dramatic changes in reflux flow rate that could affect product purity.

The undergraduate students who built this batch column certainly gained from the experience by designing and building a real process. The students were involved in, and for the most part headed up, the design of the column from the beginning. They learned how to communicate with equipment vendors, how to recognize the need for and implement design changes during construction, and how to make other daily engineering decisions based on their classroom experience. Finally, these students learned that start-up of a new process is as much about troubleshooting as it is about testing the capabilities of the equipment. In essence, the student builders were able to see what they would likely do as an entry-level process engineer, while at the same time solidifying the application of classroom theories in all aspects of the design.

UO Lab students who operate the automated batch distillation apparatus benefit in several ways. All of our lab experiments require the students to investigate background information and prepare a brief literature review on the subject, to design an experiment (logically, if not statistically), to write an operating procedure, to evaluate their procedure for safe operation, to present their plans for approval before running the experiment, to operate the equipment, to collect data, to analyze their data, and to prepare a final report. And, of course, all of these experiences involve teamwork.

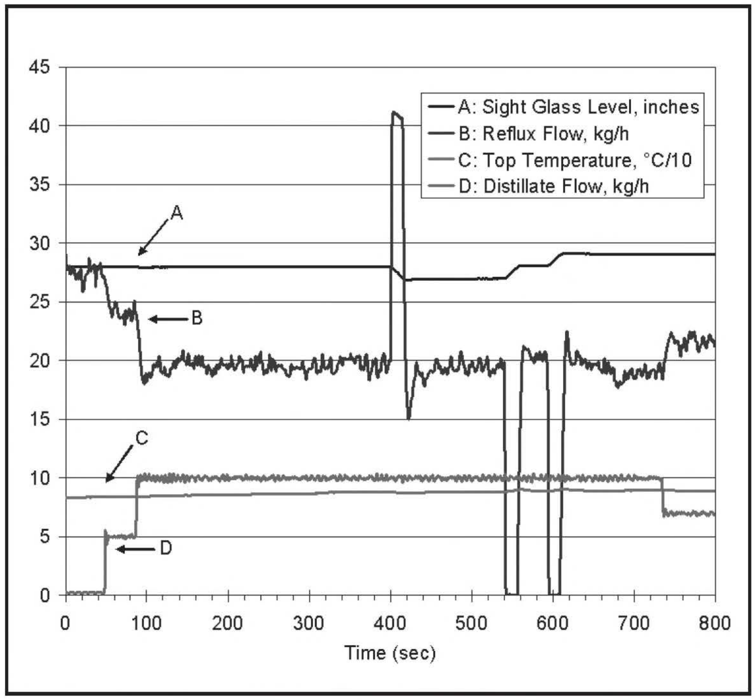

Figure 3. *Non-steady-state operational data from the batch distillation tower.*

This particular experiment involves one of the most common unit operations used by chemically related industries. The experiment is driven primarily from a computer interface, much like the control room environment students will encounter in industry; unlike industry, however, the actual apparatus is only five feet away. The glass column allows the students to observe tray phenomena, such as weeping and entrainment flooding. Specific assignments can be varied so that the students learn how to achieve an operating or production objective, such as a specified distillate purity. The control environment provides hands-on experience with numerous concepts that might remain abstract if taught only in the classroom or even if supplemented with computer simulations. Some of the concepts we have explored already include offset, reset windup, control valve saturation, loop interaction, and nature and magnitude of disturbances. There are surely others that experience and ingenuity will reveal.

CONCLUSIONS

The batch column performs in a manner similar to that of a typical industrial column with an overall tray efficiency ranging from 60 to 70%, a distillate composition ranging from 60 to 80% IPA for the constant distillate mode when there is 25% IPA initially in the still pot, and up to 70% IPA for the constant composition mode. The inherent characteristics (*i.e.*, graphically based interface) of the Labview software make operating the column easy. Once the students gain a little experience with the controllers, they are able to manipulate and optimize the process in a control room environment, while at the same time observing common tray phenomena and the effects of different operating conditions on column performance. Finally, the students are able to gain insights into aspects of process control that might otherwise elude them in class work or simulation assignments alone.

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