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Tools for Teaching Batch Distillation Inductively using Process Simulation

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Abstract

One approach to active learning involves taking students through a guided exploration process; one which ensures students will observe phenomena and then be asked questions intended to draw conclusions regarding the observations. In recent years, this has been called inductive teaching, but more recently the expression “inquiry-based instruction” or related terms have become more common. Concurrently, simulation is becoming an increasingly important tool to perform these guided explorations as constrained resources prevent operation of laboratory equipment during lecture-oriented classes. This paper describes the development of a tutorial to teach students how to develop and conduct simulations using Aspen Batch Distillation, along with the design of four inquiry activities modeled after the work of Vigeant and Prince[1-2]. This model begins with consideration of a scenario, followed by prediction, exploration, conclusion, and reflection. The four inquiry activities are designed to explore key relationships in batch distillation involving pressure, heating rate, column internals, and reflux ratios, and to also consider the safety and economic factors in batch distillation design and operation. The tutorial and activities (complete with suggested solutions) will be made available to faculty members upon request while in the refinement and testing stages during fall 2013.

Introduction

As computers have become more capable of accurately simulating complex physical activity, traditional engineering laboratories have moved away from the laboratory and towards the virtual realm. Using simulation, an exploratory approach to learning is not hampered by physical resource limitations and time constraints. This paper describes the combination of a tutorial for batch distillation simulation with tools to engage students in an inductive learning process (the process of observation and interpretation based on factual evidence leading to generalized conclusions) and an optional experiential exercise incorporating experimental design.

A part of the aspenOne family of simulation software developed by Aspen Technology, Aspen Batch Distillation[3] may be used to teach the relationships of key batch distillation variables upon system performance. Using the approach described here, a student is guided through a detailed tutorial to model a laboratory batch distillation column, and then uses the results to predict the column’s performance. If the instructor would rather not have students invest time in developing the model, a complete model can be provided to students. Four inquiry-based activities have also been developed in which the effects of column pressure, heating rate, column internals, and reflux ratio are explored through simulation. The student is first asked to predict the effect of changing a process variable. Next, the student runs simulations to test their prediction. After observing these effects, the student is asked to apply what they have learned using several “what-if” type questions involving the key variables of batch distillation as well as economic and safety questions. As an optional activity, suggested protocols for two- and three-dimensional experimental designs are made available for verification of simulation accuracy via laboratory experimentation on a modeled column. This experimentation will likely reveal (as it did in our laboratory) the difficulty of accurate simulation and stresses consideration of sources of experimental error which are not incorporated into a particular simulation.

Background

Teaching can be generally classified as occurring by one of two methods: deductive or inductive. For the higher education in the United States, deductive instruction has long been the dominant form. This style involves the teaching of the conclusive description of a topic and then applying that description to specific scenarios. For example, the theory behind heat transfer is learned first and the student is then expected to apply this theory to a particular real-world design. This approach is essentially the opposite of the process by which a particular body of knowledge was originally developed.

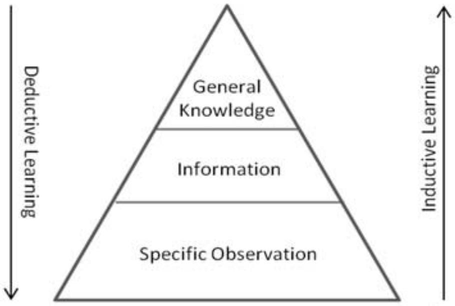


Figure 1. Inductive and deductive learning[4].

Inductive learning, on the other hand, involves the acquisition of knowledge through specific observation (see Figure 1). This learning style is the more natural form of learning in that human beings are creatures of observation. By observing complex phenomenon for particular situations, broader descriptions, models, and theory may be developed.

Deductive learning still has an important part in engineering education. In fact, a pure deductive or inductive learning style is neither efficient nor effective on its own. Therefore, the highest level of learning occurs with a combination of the two styles. To pique student interest and improve motivation, it is recommended that the inductive learning style precede the deductive style[5]. This arrangement would first require the student to observe some phenomenon and induce specifics from these observations. A deductive style would then be used to dissect the phenomenon and educate the student on the workings of the constituent parts and how they function as a whole. This combined learning style is called “student-centered” learning and depends heavily on student involvement. This combination is the justification for use of the inquiry-based methods described later.

Module Objectives

The modeling of real batch distillation systems is mathematically intensive and often requires numerical solutions. Engineering software packages can be used to familiarize students with practical design parameters associated with batch distillation units and to teach to role of key process variables (reboiler power input, reflux ratio, and operating pressure) on system operation. Nearly 95% of the chemical industry consists of processes with at least one distillation unit[6], with most operating in continuous mode. It is, however, becoming increasingly important that chemical engineering students develop a firm grasp on batch distillation principles because of the dependence of the pharmaceutical, food, and other high-purity or high material value industries utilizing on this mode of operation. In addition, understanding the influence of important distillation parameters like reflux ratio and the number of theoretical stages are applicable to both batch and continuation processes. It is both beneficial and advantageous for beginning chemical engineers to be fluent in the common design and simulation packages involving the batch distillation process.

This module consists of a student tutorial for the *Aspen Batch Distillation* user interface, an optional laboratory component incorporating experimental design, and multiple inquiry-based activities. The tutorial guides the student through the simulation setup and reduces the plentitude of controllable variables to a simplistic level suitable for a student beginner. The optional experimental design allows students to simulate an actual column and predict performance based on the system's variable settings (reboiler power input, reflux ratio, and operating pressure). Finally, this module presents inquiry based activities for students to perform using their newly formed simulation skills. These activities provoke the inductive learning process by asking students to alter key distillation variables and answer questions involving the relationship of those variables on performance. These printed materials are all suitable for standard typical chemical engineering Process Separations course and can be completed in a 50 minute class session.

The intended learning outcomes (and the component intended to contribute to the outcome) of this module are for students to:

1. Create a batch distillation simulation (tutorial)
2. Validate a batch distillation simulation (laboratory)
3. Apply experimental design principles to validation experimental protocols (laboratory)
4. Demonstrate an ability to troubleshoot batch distillation scenarios using conceptual understanding of batch-operated column behavior (inquiry activities)
5. Demonstrate awareness of safety issues commonly encountered in batch distillation operation (inquiry activities)
6. Apply the linkage between economics and operational decisions including collection time involved in batch distillation (inquiry activities)

An interesting suggestion for the module was to compare the results of several different simulation packages. It was determined that this was an unrealistic in that students would be required to become familiar with multiple software packages. Since the module intent is to expose students to the concepts of distillation while also becoming familiar with a common simulation tool, the simulation anomaly between packages was considered to hold no educational benefit. However, major commercial batch distillation simulators, including CHEMCAD Batch Distillation, Aspen BatchSep (predecessor of Aspen Batch Distillation), and BPRC MultiBatch DS, have been previously compared[7].

Batch Distillation Tutorial

The tutorial introduces the user to the *Aspen Batch Distillation* package by constructing a simple, binary simulation. The default simulation system consists of an equimolar toluene/ methylcyclohexane system to be separated using within a nine stage batch distillation column[8]. This system was selected due to the local availability of an experimental distillation apparatus capable of running in batch mode leading to the laboratory exercise below. The student is first shown how to configure a problem definition file which contains the component data pertinent to the system. As a second step, the student is instructed in the setup of the structural data block which defines the column design and sets the operating procedure. During the final step in the simulation procedure, the student is guided through the setup of profile plots in order to visually observe the simulation activity. Once these plots are configured, the simulation is performed and the results made available. As extra information, "FYI" boxes are scattered throughout the tutorial to define certain concepts or make students aware of more complex capabilities available in the simulation package. After performing the tutorial, the student should be able to develop similar simulations and effectively navigate the user interface. This 11-page fully illustrated tutorial can be completed in the standard 50 minute class period and serves as the foundation for the inquiry based activities included as part of the module.

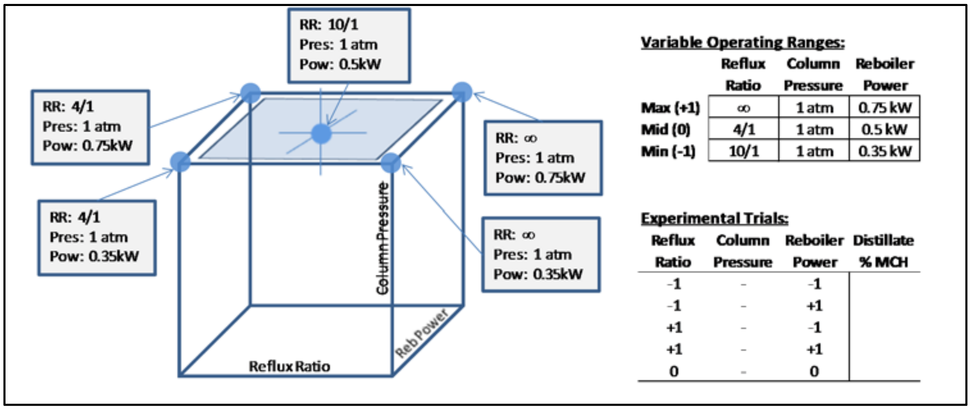
Optional Laboratory Exercise

This module allows an optional experimental exercise that utilizes the simulation skills developed in the Aspen Batch Distillation tutorial to model an actual distillation column operating in batch mode and predict its performance. A three-factor and two-factor design approach are available to introduce students to the operation of a batch distillation column and explore the effects of certain operating variables (reflux ratio, reboiler power input, and column pressure) upon the resulting chemical split. Operational safety concerns are also addressed for the general distillation column. The student should be aware that there are many factors that affect the congruency of the simulation and experimental results. Similarly, there are many factors that are not considered in the simulation that play significant roles in the performance of an actual column.

Due to the nature of vacuum distillation, many lab-scale columns cannot allow easy sampling when under vacuum. For this reason, a two-factor design is also presented in which the operating pressure remains constant at atmospheric conditions. This two-factor design is also more student-friendly due to its inherent simplicity.

Experimental Design (Two-Factor)

This design explores the effects of reflux ratio and reboiler power input upon the level of chemical separation. By implementing the two-factor approach, the design requires a minimum of five trials to obtain a statistically significant data set. Since this design is a simplification of the three-factor alternative, it considers only one face of a cubic design.



Variable Operating Ranges:

	Reflux Ratio	Column Pressure	Reboiler Power
Max (+1)	∞	1 atm	0.75 kW
Mid (0)	4/1	1 atm	0.5 kW
Min (-1)	10/1	1 atm	0.35 kW

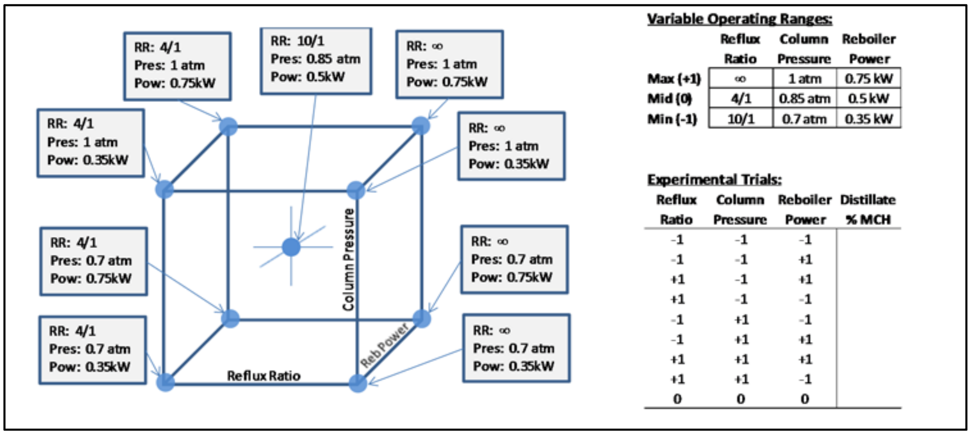
Experimental Trials:

Reflux Ratio	Column Pressure	Reboiler Power	Distillate % MCH
-1	-	-1	
-1	-	+1	
+1	-	-1	
+1	-	+1	
0	-	0	

Figure 2. Two-factor experimental design available for simulation validation.

Experimental Design (Three-Factor)

This design explores the effects of reflux ratio, reboiler power input, and column pressure upon chemical separation. To obtain a statistically significant data set, this design requires nine experimental trials. Eight trials represent the corners of a cube with the ninth being the center.



Variable Operating Ranges:

	Reflux Ratio	Column Pressure	Reboiler Power
Max (+1)	∞	1 atm	0.75 kW
Mid (0)	4/1	0.85 atm	0.5 kW
Min (-1)	10/1	0.7 atm	0.35 kW

Experimental Trials:

Reflux Ratio	Column Pressure	Reboiler Power	Distillate % MCH
-1	-1	-1	
-1	-1	+1	
+1	-1	+1	
+1	-1	-1	
-1	+1	-1	
-1	+1	+1	
+1	+1	+1	
+1	+1	-1	
0	0	0	

Figure 3. Three-factor experimental design available for simulation validation.

Inquiry Based Activities

As a supplement to the inductive learning process, five inquiry-based activities are available for student benefit. These activities follow a common procedure as described by Laws[9] and is visualized in Figure 4. First, the student is asked to predict the outcome of a variation in a process variable. Simulation or experimentation is then used to investigate the actual outcome and the student is asked to compare it with their prediction. If their prediction was wrong, they are asked to consider their thought process to identify where the conceptual error arose. Ideally, the student then adjusts his/her thinking to reflect the simulation results. Since this procedure involves the experimental observation of system changes

Figure 4. Inquiry-based activity structure.

and drawing conclusions from those observations, inquiry activities are considered to be an inductive learning method. Furthermore, the student evaluation of their predictions may help to correct misconceptions and translate into a higher level of understanding and retention.

The inquiry-based activities developed in this module are designed to teach students the effects of the major variables available in the batch distillation process as well as the effects of design variables associated with distillation equipment. Four inquiry-based activities are presented in which the effects of column pressure, heating rate, column internals, and reflux ratio are explored. The activities are guided with values of variables suggested based on real column operation, but students are encouraged to explore other settings in a systematic way. In addition, each activity addresses a supplementary objective related to economic and safety issues to apply the primary concept addressed in the activity. Safety issues addressed in the activities include pressurized operation, thermal safety, chemical compatibility, structural integrity, and electrical safety.

For example, the first inquiry activity addresses the effect of pressure on the distillate composition profile. The student is instructed on how to configure the simulation (assuming a basic familiarity with the software) for a specific scenario starting with total reflux operation. After predicting whether pressure will increase or decrease the recovery of the volatile component when pressure is dropped (with justification), the student then runs several simulations at different pressures so that a conclusion may be drawn. Next, the student predicts what will happen when running at higher pressures, but is also asked to consider why one would run this system at a higher pressure. The last step in the activity prior to preparing a set of deliverable answers to questions is to consider the safety issues associated with selection of an operating pressure. The instructor version of the current draft of this activity is included as Appendix A.

Each activity includes a set of questions intended to serve as a deliverable for the activity. Some of the questions ask how the student’s thought process has changed as a result of an incorrect prediction. Others ask students to consider what is happening at a molecular level, what are the economic constraints, or give the student an opportunity to demonstrate understanding related to the activity objectives.

These inquiry-based activities will begin classroom testing during the fall 2013 academic semester. Students will be administered pre- and post- tests assessing their ability to predict the effect of changes to the operating parameters of a batch distillation system. They will also be surveyed regarding ease of use and understandability of the materials provided.

Summary

This module involves the instruction of batch distillation principles by the inductive learning process. Students are presented with a guided tutorial of the Aspen Batch Distillation software package in order to establish familiarity with a computational simulation environment. Once competent in Aspen Batch Distillation, the student is assigned four inquiry-based activities which involve the variation of key variables pertinent to the process or equipment structure. By altering these key variables and observing the resulting effects, the student will induce the effects of those variables on the process performance. After observing the variable effects, the student is asked questions that reflect on his/her new-found knowledge in order to solidify understanding and exercise critical thinking skills. This module also presents an optional experimental design that involves a laboratory investigation of the simulation accuracy.

Faculty members interested in piloting these modules in whole or in part during Fall 2013 should contact the authors.

Acknowledgements

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References

1. Prince, M. J., Vigeant, M. A., & Nottis, K. E., "The Use of Inquiry-Based Activities to Repair Student Misconceptions Related to Heat, Energy and Temperature", *2011 American Society for Engineering Education Conference Proceedings,* ASEE, June 2011.
2. Vigeant, M. A., Prince, M.J., & Nottis, K.E., "Inquiry-Based Activities to Address Critical Concepts in Chemical Engineering", *2011 American Society for Engineering Education Conference Proceedings,* ASEE, June 2011.
3. Aspen Batch Distillation, Aspen Version 7.3, Cambridge, MA, 2011.
4. Johann Gasteiger, "Chemoinformatics: a New Field with a Long Tradition", *Analytical and Bioanalytical Chemistry*, 2005.
5. Prince, M. J., & Felder, R. M., "Inductive Teaching and Learning Methods: Definitions, Comparisons, and Research Bases", *J. Engr. Education*, 95(2), 123-138 (2006).
6. Wankat, Phillip C., *Separation Process Engineering,* Upper Saddle River: Prentice Hall, 2011.
7. Diwekar, Urmila, *Batch Distillation: Simulation, Optimal Design, and Control*, Boca Raton, FL: CRC Press, 2012.
8. Armfield, "Instruction Manual for Continuous Distillation Column UPO3CC", Issue 12, January 1998.
9. Laws, P., D. Sokoloff, & R. Thornton, "Promoting Active Learning Using the Results of Physics Education Research", *UniServe Science News,* 13 **(**1999).

Inquiry-Based Activity 1

Due DayOfWeek, Month Day, Year

Pressure Effects on Batch Distillation

In this activity, *Aspen Batch Distillation* will be used to simulate a batch distillation process and observe the effects of pressure on the resulting distillate composition profiles.

> NOTE: The *Aspen Batch Distillation* file developed in this activity is referenced in other activities. It is recommended that you retain the original file for future use.

Directions:

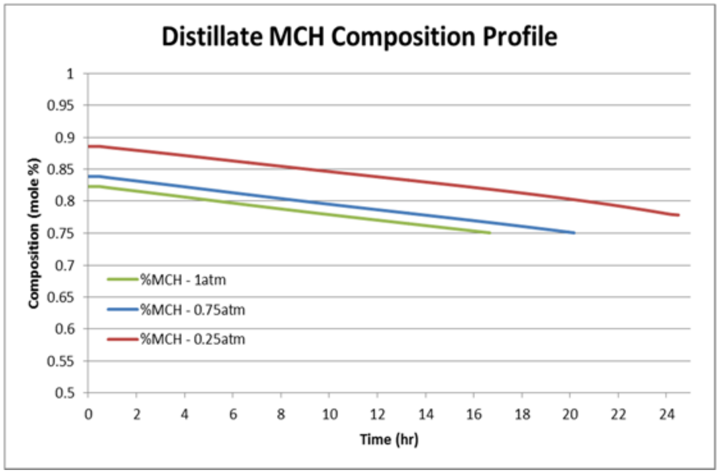
1. Open a new instance of *Aspen Batch Distillation* and add a column to the flowsheet. Set up an equimolar toluene/methylcyclohexane solution with NRTL as the property method.
2. In the column properties, set the pot geometry to horizontal with a 1 meter diameter and ½ meter length. Enable heating coils and set their heating option to specified duty of 5 kW. Set the pressure profiles and holdups to calculated and do not alter the column internals.
3. Set the initial condition to total reflux and initial charge to 5 kmol of a 50/50 solution.
4. Create an operating step to set the reflux ratio to total reflux (this is redundant) and then wait 30 minutes to achieve steady-state. Create a second step to change the reflux ratio to 4 and set the ending condition as the distillate receiver's MCH composition approaching 75% from above. How do you know when steady state is achieved?

   Composition and temperature profiles are non-changing (flat).

5. Create a plot and map the distillate product's MCH and toluene composition to the y axis.

   NOTE: Be sure to use the composition data from the Holdup Summary Results tab for the plot. This tab's data is the total distillate product's composition while the data on the Distillate Results tab is the distillate *stream's* composition.

6. Run the simulation and export the distillate MCH composition profile to Excel. Set the condenser pressure to 0.75 atm (Pressure/Holdups tab) and predict the effects of the change. Run the simulation and export the profile and overlay it on the previous graph. Was your prediction right?



7. Run the simulation a third time with operating pressure set to 0.25 atm. The simulation will automatically pause after 24 hours because the stopping criteria are not reached. Resume the simulation and let it run to 30 hours. Notice that the distillate composition profile bottoms out around 77.7%. Why does this happen? (HINT: check the holdup tab)

   Pot holdup reaches zero (pot is dry).

8. Based on the behavior of these simulations, how would the distillate MCH composition profile be affected if the operating pressure were *increased* to 1.5 atm? Can you think of any reasons to operate at high pressure? You may run the simulation at this pressure and any others you wish.

   The profile would shift downwards and the operating time decrease. Faster batches and less molar holdup in column. These can both be controlled by altering the reflux ratio as well. There is really no substantial benefit to positive pressure operation.

9. Pressure operation raises several safety issues. Can you think of at least two?

   Risk of implosion/explosion, air leaked into column (vacuum), fugitive emissions (positive pressure), condenser overload, reboiler strain.

Analysis *- to be completed after lab and* ***handed in:***

1. By manipulating pressure, what is occurring on the atomic scale? What happens to the solution's boiling point and heat of vaporization?
2. Why does decreasing pressure shift the distillate MCH composition profile up? Why does reduced pressure cause the column to take longer to reach the stopping composition?
3. Was your prediction in 6 of Directions correct? If not, how was your thinking revised?
4. Vacuum pressure operation is often a less-preferred option because it is expensive and introduces safety concerns. List a few of these safety concerns. What are two alternatives to reducing pressure that can produce similar results?
5. Where is the pressure greatest in a batch distillation column?
   a. Top (condenser)
   b. Just above the center
   c. Just below the center
   d. Bottom (reboiler)

Answers:

1) At higher pressures, there are more frequent collisions between molecules, resulting in more transient "dimer"-type structures being formed. Solution boiling point and latent heat are increased due to the increased molecular interactions. The opposite takes place for reduced pressures
2) Less energy is required to vaporize the MCH. The profile slopes are all the same, therefore, starting at a higher composition (y value) will take longer to reach the stopping criteria.
3) N/A
4) Risk of implosion/explosion, air leaked into column (vacuum), fugitive emissions (positive pressure), condenser overload, reboiler strain. Increase reflux ratio, optimize reboiler power input, use a partial condenser.
5) Answer D is correct. The high-to-low gradient up the column is the mass driving force.