Integrating Process Simulation into the Unit Operations Laboratory Through an Absorption Column Experiment

Zac Bunnell, Garett Scott, and Sundararajan Madihally

School of Chemical Engineering, Oklahoma State University

Abstract

Recent advances in computational tools have revolutionized the way graduating students will work and interact with multiple disciplines. This has necessitated an the integration of novel technologies into traditional courses, particularly into Unit Operations Laboratory (UOL). In UOL students obtain hands-on experience on the application of the theoretical concepts learned in core classes such as mass transfer operations, chemical reactor design, and transport phenomena.

We introduced process simulation into an absorption column experiment in UOL. Students were asked to evaluate the effectiveness of a packing material, known commercially as Nutter™ rings, relative to other common packing materials using an air-acetic acid-water system under isothermal conditions. The packed tower was equipped with a control system for on-line monitoring of pressure changes across the column and flow measuring devices to regulate liquid and gas flow rates.

First, students were asked to establish the operating range of the column by varying the air and water flow rates. Since the tower is made of a transparent material, they were able to observe the flooding conditions. Next, using a low concentration of acetic acid in water, they evaluated the percentage removal of acetic acid by titration of inlet and outlet streams. Using these data and assuming no pressure drop across the column, students were asked to determine the height equivalent to a theoretical plate (HETP) at various gas and liquid flow rates. To integrate the pressure drop measured during the operation of the column into the separation efficiency, they were asked to simulate the process using ChemCAD® (Chemstations, Inc) software. Additionally, they were asked to compare the separation efficiency of Nutter™ rings to other routinely used packing materials such as burl saddle and Raschig rings.

This multi-level experiment not only reinforces the concepts of mass transfer operations and process simulation but prepares the students for the challenges they will face in today's Chemical Engineering Industry. Implications of this experience will be discussed in detail.

Introduction

Recent advances in computational tools have revolutionized the way graduating students interact and apply chemical engineering principles at the workplace. A significant progress has enabled process simulation software, along the norm in the chemical engineering, to efficiently run on desktop personal computers. Process simulation software is useful to visualize plant processes, perform heat and material balances of process flowsheets, design new plants, or modify and expand existing plants. This has necessitated the integration of novel technologies into traditional courses, particularly into the Unit Operations Laboratory (UOL).

A laboratory course on Unit Operations is a critical component in the chemical engineering undergraduate curriculum, because it provides hands-on experience on the application of the theoretical concepts learned in core classes such as mass transfer operations, chemical reactor design, and transport phenomena. Typically students carryout a number of experiments related to each concept. This modular approach emphasizes the process concepts, but lacks interconnectivity and integration of novel computational tools such as simulation packages, statistical analysis tools and technical writing skills. At Oklahoma State University, one goal in chemical engineering education is to encourage students to connect concepts developed in multiple courses, and generate comprehensive solutions to engineering problems. The UOL is an ideal course to integrate all chemical engineering curriculum skills as well as the technical tools that are routinely used in the industrial environment.

One of the UOL courses is offered in the Fall of the senior year, where students work in teams of three, on three projects. Each project covers a 5-6-week period. It includes a week of planning, three 6-hr laboratory sessions, and two weeks of oral and written presentations. To ensure that students include all issues, and properly analyze the data, instructors actively observe and coache the teams as they work. Prior to this course, "Rate Operations I" is introduced in the fall semester of the junior year, and "Rate Operations II" is taught in the spring of the junior year. In Rate Operations I, they are introduced to the importance of pressure drop across various flow regimes. The Rate Operations II course introduces students to equilibrium separations [1, 2]. Furthermore, a process simulator, ChemCAD™ (Chemstations, Inc), was introduced in the Introduction to Chemical Engineering course to train students the basics of drawing the process and identifying the required inputs. The same ChemCAD™ is also used in Chemical Reaction Engineering Course which is taught in the Spring of junior year, hence minimizes time constraints involved in introducing a new software.

Project Statement

In one of the UOL projects, students were asked to evaluate the effectiveness of a packing material, known commercially as Nutter™ rings, relative to other common packing materials using an air-acetic acid-water system under isothermal conditions. The project stages were:

a) *determine the operating range of the column*: Since the tower is made of a transparent material, they were able to observe the flooding conditions.

b) *determine the height equivalent to a theoretical plate (HETP)*: They were told to use low acetic acid concentrations in water, and evaluate the percentage removal of acetic acid using air. The percentage removal was determined by titration of the inlet and outlet stream samples using sodium hydroxide and phenophthalene as the indicator. Using these data and assuming no pressure drop across the column, students were asked to determine the height equivalent to a theoretical plate (HETP) at various gas and liquid flow rates.

c) *determine the pressure drop across the column*: Using the pressure transducer, they were told to monitor the pressure drop during various flow conditions.

d) *compare the Nutter™ rings to other routinely used packing materials such as Burl saddle and Raschig rings [3]*: Using ChemCAD™ software, they were asked to simulate the process and predict the separation efficiency of the column. In addition, they had to change the packing material in the simulation and then compare the efficiencies for one operating condition. Further, to understand the effect on separation, they were also told to simulate the percentage removal by incorporating the pressure drop readings measured in the laboratory.

Experimental Setup

Figure 1 shows the schematic of experimental setup. It consists of two separate air and water inlets and are throttled through control valves. The flow rates are measured through rotameters which are also calibrated using the time to fill a bucket at various levels or through a hand-held anemometer. Two pumps are present in the system and the first pump is used to pump water to the top of the tank and other pump can be used either as a recycler or for pumping to the second tank for neutralization of acidic components prior to disposal. Water hold-up in the column is regulated through a solenoid valve, level transducer, and level controller assembly. Further, a pressure transducer is connected across the column to measure the pressure drop and is operated through Camile TG® (Argonaut, Foster City, CA). The outlet of air opened into the atmosphere i.e., vented into a chemical hood. The tower was packed with 1 inch Nutter Rings™.

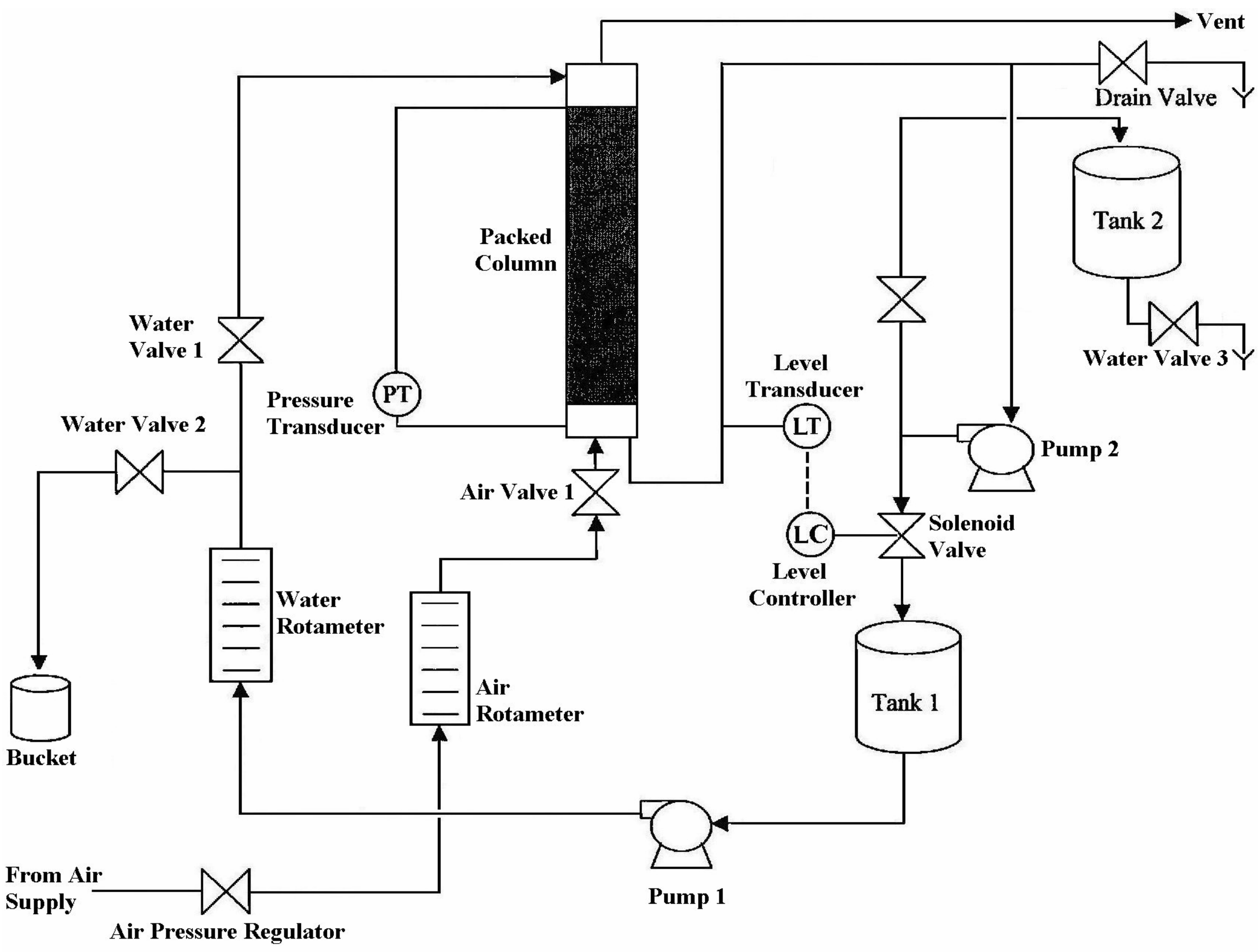

Figure 1: Process schematic showing the experiment set-up used in the project.

Using 0.05M acetic acid, the packed tower was tested to determine the range of allowable flow rates that were possible without entering the flooding regime. The flow rates of air was varied from 10 to 15 SCFM and the water flow rate was varied from 20 to 35 lb/min. By titrating the acetic acid solution exiting the column with NaOH, the amount of acetic acid stripped out of the water was determined using a material balance, and assuming no reaction in the column. For each flow rate combination, pressure drop across the column was recorded.

Analysis of Results
The packing efficiency can be modeled using transfer units, although the packed tower has a continuous liquid-gas interface. Using the HETP (height equivalent to a theoretical plate)[2], the efficiency of the Nutter Ring™ packing can be determined. The HETP was calculated using the equation

$$HETP = \frac{H_{OG} \ln\left(\frac{mV}{L}\right)}{\frac{mV}{L} - 1} \quad (5)$$

where the HETP is given in ft, *m* is the average slope of the equilibrium curve (which was determined using ChemCAD™ for water-acetic acid-air system to be 2.4305 $lbmol_{water}/lbmol_{air}$), V is the vapor flow rate in $lbmol_{air}/hr$, and L is the liquid flow rate in $lbmol_{water}/hr$ [2]. The overall gas phase transfer unit, H_{OG}, was calculated using the equation

$$H_{OG} = \frac{mV}{L} H_L + H_G \quad (6)$$

The height of the liquid phase transfer unit, H_L, was determined using the following equation

$$H_L = \frac{L}{k_L a A_C} \quad (7)$$

where H_L is in ft, a is the interfacial area per volume in ft^2/ft^3 given in the Nutter Ring™ packing manual to be 51 ft^2/ft^3, and A_C is the cross-sectional area of the column in ft^2 [4]. Similarly, the height of the gas phase transfer unit, H_G, is given by

$$H_G = \frac{V}{k_G a A_C} \quad (8)$$

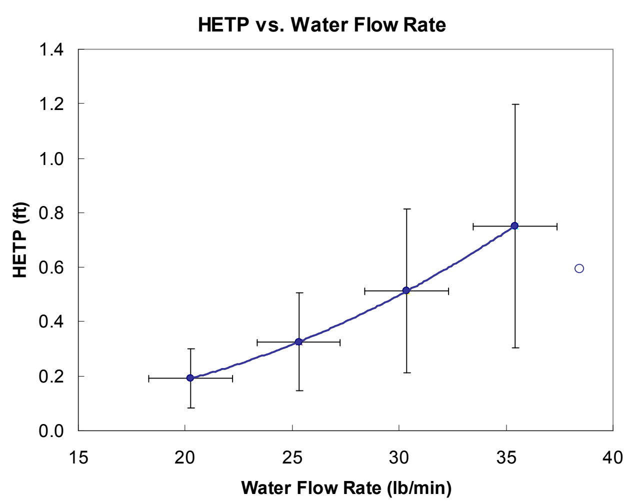

Figure 2: HETP values versus water flow rate for air at 12.8 SCFM. (data shows propagation of maximum uncertainty).

HETP values were calculated and graphed against liquid mass flux. As shown in **Figure 2,** HETP values increased as liquid mass flux increased. As liquid mass flux increases, mass-transfer decreases; therefore, HETP values increase to reflect the lower mass-transfer efficiency. The HETP values calculated were found to be reasonably close to the expected values (~0.8 ft) included in the Nutter Ring™ literature [4].

ChemCAD Simulation

To compare the efficiency of various packing materials and the effect of pressure drop, the packed column was modeled using ChemCAD™. The model was built using SCDS distillation column with two inlets and two outlets. Values from the packed column in the UOL were entered into the simulator. These specifications included column pressure, temperature, vapor and liquid flow rates, column size, HETP values, packing material properties, pressure drop, and packing height. For thermodynamic properties, the SRK model was used and for phase equilibrium Henry's law was used. For simulating, "Packed column mass transfer" was used.

ChemCAD™ simulation results using Nutter Ring™ packing, no packing, no pressure drop, and several other types of packing were compared to experimental results shown in **Table I**. An experimental HETP value of 0.8 ft was used as the input in ChemCAD™ to calculate percent removal of acetic acid. This value was chosen based on comparison to average HETP values for Nutter Rings found in literature. The simulation was then run with these specifications, changing specific variables, i.e. HETP, and packing materials. The output was then analyzed and compared to our experimental results. A process simulator, such as ChemCAD™, allows for simulation of situations not available in lab and provides deeper insight into the experiment.

Using HETP, the efficiency of the Nutter Ring™ packing was determined.

Table I: Comparison of experimental data to ChemCAD™ simulation and literature.

Method of Prediction	HETP Value (ft)	Percent Removal
Experimental for air at 12.8 SCFM and water at 30 max %	0.78 ft	18.90%
ChemCAD with Nutter Ring	0.80 ft	19.15%
ChemCad with Berl Saddle packing	0.80 ft	16.95%
ChemCad with Paul Ring packing	0.80 ft	19.07%
ChemCAD with Raschig packing	0.80 ft	19.07%
Nutter Ring Literature [4]	0.83 ft (average) varies by components	—

As the results show, ChemCAD™ provides a reasonable simulation for a packed tower and allows the modeling of many packing materials.

Discussion

Students can design and implement UOL experiments which integrate fundamentals, practice and tools from which experimental data reinforces classroom principles. While this provides an excellent learning experience, and develops student confidence, the primary <u>disadvantage</u> of this method is the time requirement; this experiment will require four to six weeks. The long timeframe minimizes the number of experiments that may be performed. The project oriented style will require a greater commitment from faculty members and less dependence on teaching assistants in the to maintaining equipment and to coaching the students. However, short experiments tend to become modular and lack integration of comprehensive concepts.

The advantage of this method is that it integrates concepts from various courses into the chemical engineering curriculum and gives a feel for the variety of aspects of equipment design including the utility of simulation. Also, this experiment gives students the opportunity to apply many of the fundamental concepts of chemical engineering, such as mass transfer phenomena, fluid flow, thermodynamics, and numerical methods. The hands-on learning achieved in the UOL help students to connect the abstract concepts of the classroom with the real world. By incorporating computer simulations into the project, students are given the chance to apply the techniques learned from the experiments to situations not available in the laboratory setting. Although experiments of this nature require more time from both the students and professors, the projects will prepare the student for the situations they will face in industry. This experiment also trains students to work on projects that are typical industrial problems.

Acknowledgement
The authors would like to thank Dr. Russell Rhinehart, Head and Professor, School of Chemical Engineering, OSU for careful review and constructive comments on the manuscript. Also, the authors would like to thank Cassie Mitchell and Angela Pantelogianis, Seniors, School of Chemical Engineering, OSU, for help on carrying out the experiments and useful discussions.

References
[1] McCabe, W.L., Smith, J.C., and P. Harriott, *Unit Operations of Chemical Engineering, 5th ed.*, McGraw-Hill, NY, 1993.
[2] Wankat, P.C., *Equilibrium Staged Separations*, Elsevier Applied Science, NY, 1988.
[3] Perry, R.H., and D. Green (eds), *Chemical Engineers' Handbook, 7th ed.*, McGraw-Hill, NY, 1997.
[4] http://www.sulzerind.com/internals.htm

Biographical Sketches
Zac Bunnell is a senior at Oklahoma State University. He is graduating with a BS in Chemical Engineering and a BS in Mathematics in May 2004.

Garett Scott is a senior at Oklahoma State University. He is graduating with a BS in Chemical Engineering and a BS in Mathematics in May 2004.

Sundararajan V. Madihally is an Assistant Professor in the School of Chemical Engineering at Oklahoma State University. He received his BE in ChE from Bangalore University and his PhD from Wayne State University in Chemical Engineering. He held a research fellow position at Massachusetts General Hospital/Harvard Medical School/Shriners Hospital for Children. His research interests include bioreactor development for tissue regeneration and the development of therapies for traumatic conditions.