EXPERIMENTAL INVESTIGATION AND PROCESS DESIGN

in a Senior Laboratory Experiment

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A drying experiment for the senior unit operations laboratory course at Villanova University is described in this article. This experiment involves the determination of the drying rate of a solid material in a forced convection drying apparatus and the scale-up design of this process. The experimental drying rate data is used to determine the appropriate transport coefficients to mathematically describe the drying process. The students then use this mathematical model for the design of a large-scale dryer for a specified production rate of the material under study. Various solid materials such as sand, gravel, clay, sawdust, natural and synthetic fibers, and agricultural products have been used in this experiment. This variety of materials is intended to provide each student group with a different experience that can be compared and contrasted during student group oral presentations at the end of the semester.

The main goal of the laboratory exercise documented in this article is to provide the students with hands-on experience in the analysis and design of drying processes. Drying is an essential unit operation in the chemical process industries with applications ranging from forest products[1] and mineral processing[2] to food products[3] and pharmaceuticals.[4] Although this technology has been a key component of chemical engineering since its inception as an academic discipline, the science of drying continues to remain an active area of research and development.[5] Despite its widespread industrial importance, however, it is not emphasized in the heat and mass transfer courses due to time constraints. This laboratory experiment provides an opportunity for students to apply transport phenomena concepts presented in the classroom to the process of drying, while becoming familiar with this common unit operation.

A second goal of this experience is to provide students with an opportunity to apply the results of their experimental study to a process design. A similar approach to the unit operations laboratory course is advocated in Reference 6. The emphasis of this experiment is not simply to obtain data to determine transport coefficients. The students must also use their results in the scale-up design of the drying process. This addition of a design element to the laboratory provides a more practical objective for students and a more realistic application of their experimental investigation. This experience also provides additional learning objectives in the laboratory course, such as the development of engineering awareness, mathematical modeling, scale-up, and economic evaluation.[7]

There have been a number of chemical engineering laboratory drying experiments reported in the literature such as microwave drying of sand[8] and convection drying of a towel.[9] A bench-scale experimental drying apparatus[10] and the statistical treatment of drying data[11] have also been reported. The unique aspect to the experiment described in this article is both the incorporation of a design element and the study of a wide variety of materials with drastically different drying properties for each group.

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LABORATORY EQUIPMENT

The experiment is carried out using a batch cross-circulation cabinet dryer. A schematic of the dryer system is shown in Figure 1. Air is supplied at a rate of 0-440 ft^3/min by a centrifugal blower with a gate-valve arrangement to adjust the air flow into the cabinet. The air is heated by two steam coil heaters located in the bottom half of the cabinet. The inlet air is passed through the heaters in the bottom half of the cabinet before being redirected to the drying section in the top half of the cabinet. A baffle arrangement (not shown in Figure 1) provides uniform air flow in the drying section. The volume of the drying section is 5.6 ft^3 and the cross-sectional area for the air flow is 1.9 ft^2. The material to be dried is contained within a shallow tray or wire basket that is suspended in the air stream.

Dryer air temperature is controlled by a valve on the inlet steam line to the heaters with manual valves to isolate either heater. Steam is supplied from the high-pressure building main. Source pressure to the dryer is maintained at 30 psig by a regulator on the supply line. This reduction in the heater steam supply pressure is for both safety considerations and improved temperature control by increasing the normal operating range of the steam valve. The maximum dryer temperature is restricted to 200 °F for all experiments to prevent the possibility of thermal decomposition or ignition of the solid material.

Dryer process measurements include: the mass of the tray and material using a load cell (Transducer Techniques EBB-5 load cell [0-5 kg] and DPM-3 digital panel meter with analog output); the air temperature and relative humidity in the dryer using a humidity probe (Omega Engineering HX94C relative humidity/temperature probe); the surface temperature of the material in the tray and a second dryer air-temperature measurement using thermocouples (Analog Devices 2B52A type T thermocouple transmitters); and the inlet air flow rate inferred from the differential pressure across an orifice in the inlet air header to the blower (Setra Systems C239D differential pressure transmitter). The approximate cost of this instrumentation at the time of installation was $400. In addition to the electronic measurements, there is a thermometer inside the dryer cabinet, a wet/dry bulb thermometer to determine the conditions of the inlet air, and a water manometer connected to the orifice in the inlet air line. The data acquisition and control computer system displays the process measurements in real time and also records these values in a data file for later analysis by the students. A data sampling period in the range of 0.25 to 1 min is suggested to the student groups for data collection by the computer system.

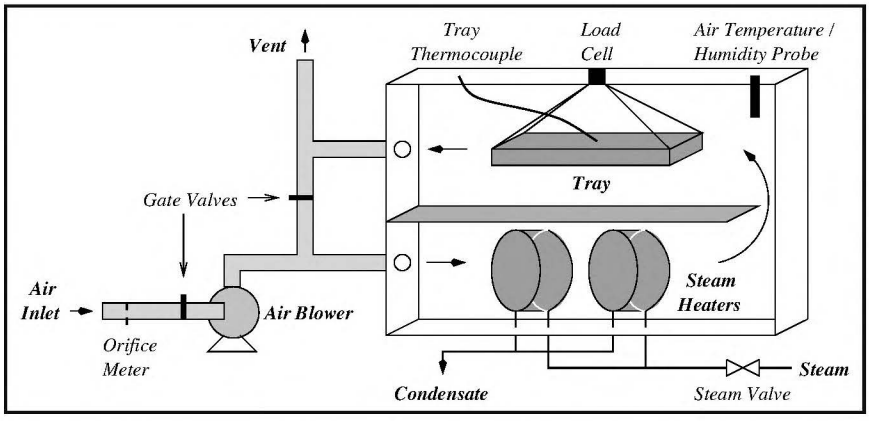

Figure 1. *Experimental cabinet dryer system.*

LABORATORY EXERCISE

There are two three-hour laboratory sessions each week for the experiments in the senior laboratory course. These sessions are composed of two planning, experimental, and analysis sequences. The students only have access to the drying apparatus during the two experimental sessions. In the first planning session, the group is introduced to the material that they will be studying, the range of moisture content that they must consider, and the specifications on the final dried product. Because the students have essentially no practical experience with drying processes, they are expected to research the drying process and plan their experimental study during this session. Presentations on drying can be found in Perry's chemical engineering handbook[12] and other process engineering handbooks such as Cooper, *et al.*,[13] along with unit operation and mass transfer texts such as McCabe, Smith and Harriot,[14] Geankoplis,[15] and Treybal.[16]

The initial drying experiments are carried out during the first experimental session where the students determine the character of the drying curve for their material. An initial estimate for the tray loading is based on rules of thumb for the design of drying processes such as presented in Reference 13. The drying rate and moisture range for the constant rate period and the transition to the falling rate period are determined from the students' initial experimental results during the first analysis session. An initial determination of the corresponding mass transfer coefficients is also carried out during this session.

In the second planning session, the group develops its experimental plan for the second experimental session. Depending on the drying behavior of their material and the results of their initial experimental session, during this session the student groups usually either concentrate on the falling rate period or consider the effect of different tray loadings. The incorporation of a second experimental session allows for the unstructured experimental approach adopted in this laboratory course. Benefits to the students of an unstructured approach include exposure to a more realistic experimental study, since

in reality the precise details of a procedure are rarely known in advance and the experimental plan often evolves as more information is discovered.

The final analysis session is used to analyze the data obtained from the second experimental session, and to design the scale-up drying process. Each student group is informed of the required production rate and initial moisture content of their source material after completion of the second experimental session. This practice is implemented in order to prevent the students from specifically targeting their experimental study to their scale-up process design requirements.

EXPERIMENTAL ANALYSIS

The process of drying can be described using an energy balance determined by the heat transfer rate between the hot gas and the moist solid, and a material balance determined by the mass transfer rate between the moist solid and the hot gas. The corresponding liquid component evaporation rate can be calculated using the relationships[14]

$$\dot{m} = M_w hA \frac{T_g - T_i}{\Delta H_i} = M_w A k_y \frac{(y_i - y_g)}{(1-y)_{LM}} \qquad (1)$$

where $\dot{m}$ is the evaporation rate of the liquid component from the moist solid, M_w is the molecular weight of the liquid component, A is the contact area between the moist solid and hot gas, h is the heat transfer coefficient, T_g is the bulk hot gas temperature, T_i is the solid-gas interface temperature, ΔH_i is the latent heat of vaporization of the liquid component at the interface temperature, k_y is the mass transfer coefficient expressed on a mole fraction basis, y_g is the bulk liquid component mole fraction in the hot gas, y_i is the liquid component gas-phase mole fraction at the solid-gas interface, and $(1 - y)_{LM}$ is the log mean value of $(1 - y_i)$ and $(1 - y_g)$.

For reasons of convenience and safety, the liquid component is water and the gas is air for all of the experimental studies in this laboratory. Due to the time constraints in the laboratory sessions and the relatively slow evaporation rates for the materials under study, the dryer system is typically operated at near maximum air flow rate to maximize the evaporation rate. Under these conditions, constant air temperature and humidity can be safely assumed. Because the thermal changes to the system occur at a faster time scale than saturation changes, it is also appropriate to assume that the drying rate can be expressed in terms of the mass transfer relationship for drying process design calculations.[17] This assumption is applied in both the experimental data analysis and scale-up design. It should be noted that a similar mass transfer expression to Eq. (1) in terms of partial pressure and humidity driving forces can also be employed. Although partial pressure has been the most popular, the student groups have tended to be rather evenly divided in their choice of driving force for their mass transfer coefficient.

The determination of the mass transfer coefficient depends on the behavior of the drying curve for the material under study. All of the materials considered in this experiment exhibit a constant-rate drying period and some exhibit a falling-rate drying period for the moisture range of interest. The constant-rate period is characterized by a constant rate of drying that is independent of the moisture content. During this period, a continuous film of water exists on the solid surface that is constantly replenished as the surface water evaporates. The falling-rate drying period occurs when the moisture content of the solid falls below some critical point. After this point, there is insufficient moisture present to maintain a continuous liquid film on the solid surface and the liquid mass transfer in the solid phase becomes limiting as opposed to interfacial mass transfer during the constant-rate period. This critical point is a function of the material, the material thickness, and the driving force for mass transfer that usually must be determined experimentally.[14] The drying rate typically decreases as the moisture content in the solid decreases during this period.

Under typical experimental conditions during the constant-rate drying period, all of the parameters in Eq. (1) are relatively constant. The constant-drying-rate mass transfer coefficient can then be determined from the slope of the total tray mass vs. time, total material mass vs. time, or the free moisture mass vs. time drying curves as follows

$$k_y = \frac{-a(1-y)_{LM}}{M_w A(y_i - y_g)} \qquad (2)$$

where a is the slope of the drying curve during the constant-rate drying period, y_g is the mole fraction of water in the inlet air determined from the humidity probe in the cabinet and checked with the wet/dry bulb thermometer, and y_i is the mole fraction of water at the solid-gas interface. The interface mole fraction is assumed to be the equilibrium saturation value at the interface temperature, which can be determined using steam tables or the Antoine equation. As discussed in Reference 15, it is possible to apply the dilute gas-phase mole fraction approximation $(1 - y)_{LM} \approx 1$ in the analysis of the constant-rate-period mass transfer coefficient.

The determination of the mass transfer coefficient for the falling-rate period is more problematic due to the changing conditions of the material and the interface. Although many approaches exist to describe the falling-rate period,[14-15] one of the simplest is to assume that the drying rate is proportional to the difference between the free moisture in the solid and the equilibrium free moisture,

$$\dot{m} = M_w A k_X (X - X^*), \quad \dot{X} = \frac{M_w A k_X}{m_s}(X - X^*) \qquad (3)$$

where k_X is the solid-phase mass transfer coefficient, X is the bulk solid free moisture, X^* is the equilibrium free moisture, m_s is the dry mass of the solid material, and the

other parameters are as defined previously. Eq. (3) can be integrated yielding

$$t = \frac{m_s}{M_w A k_X} \ln(X - X^*) + b = a \ln(X - X^*) + b \qquad (4)$$

where a is the multiplicative constant and b is the constant of integration. The solid-phase mass transfer coefficient can be determined from the constant, a, obtained by a logarithmic fit of the free moisture vs. time experimental data.

$$k_X = \frac{m_s}{a M_w A} \qquad (5)$$

This fit is easily accomplished using Excel or any curve-fitting numerical package. The use of the free moisture vs. time curve, as opposed to the drying rate computed by central differencing the data as discussed in Reference 8, provides a more accurate estimate of the mass transfer coefficients for both the constant and falling rate periods because of the noise inherent in the load cell measurements.

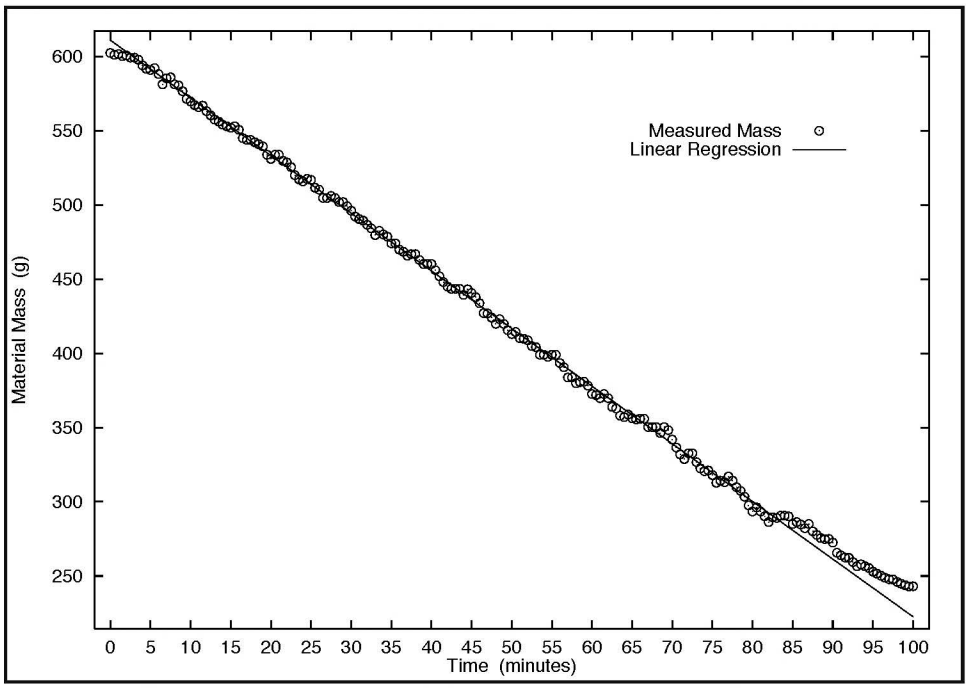

Figure 2. *Total material mass drying curve.*

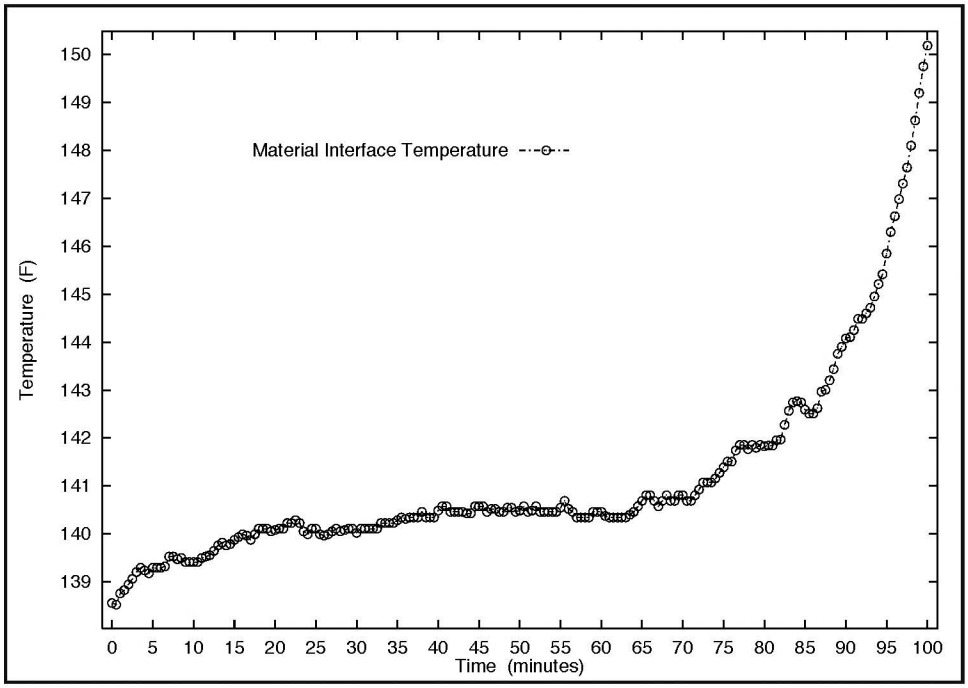

Figure 3. *Material interface temperature.*

EXAMPLE EXPERIMENTAL RESULTS

Figure 2 presents the total material mass (load cell reading minus the empty-tray mass) drying curve for a coarse sawdust material initially composed of a 2:1 volumetric mixture of sawdust (900 ml–156 g) and water (450 ml–450 g) with an average material thickness in the tray of 1 cm. Figure 3 presents the material interface temperature for this system determined by a thermocouple embedded in the surface of the sawdust. Linear regression on the material mass for the constant-rate drying time period between 5 and 80 minutes resulted in a slope of -3.89 g/min with a correlation coefficient of 0.992. Assuming an equilibrium interface temperature of 140 °F for the constant drying period, the resulting constant-rate drying period mass transfer coefficient is k_y = 11.2 gmol/m^2-min. Although not used in this calculation, the log mean value was $(1 - y)_{LM}$= 0.895, which is close to the dilute-gas phase mole fraction approximation. The specification on the final dried sawdust product was that it must be free flowing without any lumps. The student group concluded that the dried sawdust material met this criterion after

85 minutes at the end of the constant-rate drying period with a moisture content of 0.8 g H_2O/g dry solid.

Figure 4 presents the free- moisture drying curve for a clay absorbent material initially composed of a 3:1 mixture by mass of absorbent (474 g) and water (158 g). The average material thickness in the tray was 0.375 in. A logarithmic regression on the falling-rate data after 31 min resulted in a value of the multiplicative constant of -61.6 min with a correlation coefficient of 0.953. The corresponding solid-phase mass transfer coefficient is k_X = 0.427 gmol/ft^2-min. The specification on the dried absorbent product was that it must be dry enough to package. The student group concluded that the dried absorbent met this criterion after 60 minutes with a moisture content of 0.075 g H_2O/g dry solid. The justification of this decision was that the drying rate essentially goes to zero after this time resulting in little further drying being possible without a very long additional exposure.

SCALE-UP PROCESS DESIGN

The scale-up process design is based on manufacturing a specified production rate of some product from the wet solid material with a specified initial moisture content. The final moisture content of the material must be determined by the student group based on a subjective performance criterion as indicated in the initial laboratory handout. Examples of this criterion are that the solid must be dried to the point that it is free flowing or dry to the touch. This subjective criterion requires both experimental data and engineering judgment to determine the final moisture content. The intent is to demonstrate that product specifications are often not directly measured physical quantities.

Because the mass transfer coefficient is a function of the operating conditions of the dryer, the scale-up process operation must not deviate significantly from the experimental operating conditions if the experimental mass transfer coefficient is to be used in the design. This restriction does impose limitations on the scale-up design depending on the experimental conditions that were considered. Specifically, the material depth in the tray and the air velocity in the scale-up design must be representative of the experimental conditions used to determine the mass transfer coefficient. For example, the air velocity is determined by the air flow rate and cross-sectional flow area. To change the driving force for mass transfer by changing the air flow rate, the cross-sectional area must also be changed to maintain a similar air velocity. A benefit of the design aspect in this experiment is the exposure to this relationship between experimental investigation and scale-up design through hands-on experience. Such exposure is not available in the process design course because of the lack of an experimental component.

A further objective of the design aspect is the development of a physically realistic process design. Although most student groups have little difficulty in determining the required surface area and air rate for the scaled-up process, the actual physical design of the dryer can often be unrealistic. A common initial approach is to scale up the experimental apparatus to handle the required production rate. The result is a design with trays that are often too large and heavy to be physically managed. More realistic process designs evolve as the student groups are prompted to consider the size and weight of the material that must be handled. An example initial approach for the scale-up design of a wood-chip dryer with a production rate of 1 ton/day consisted of a batch cabinet dryer using a single 50-ft-long tray containing almost one-half ton of wet wood chips. The final design was also a batch cabinet dryer, but instead consisted of ten 5-ft-long trays stacked on top of each other where each tray initially contained approximately 120 lbs of wet wood chips. The consideration of the practical aspects of a process design is an additional benefit of this experience.

PRESENTATION OF RESULTS

The experimental and scale-up process design results for each student group are reported in a formal written

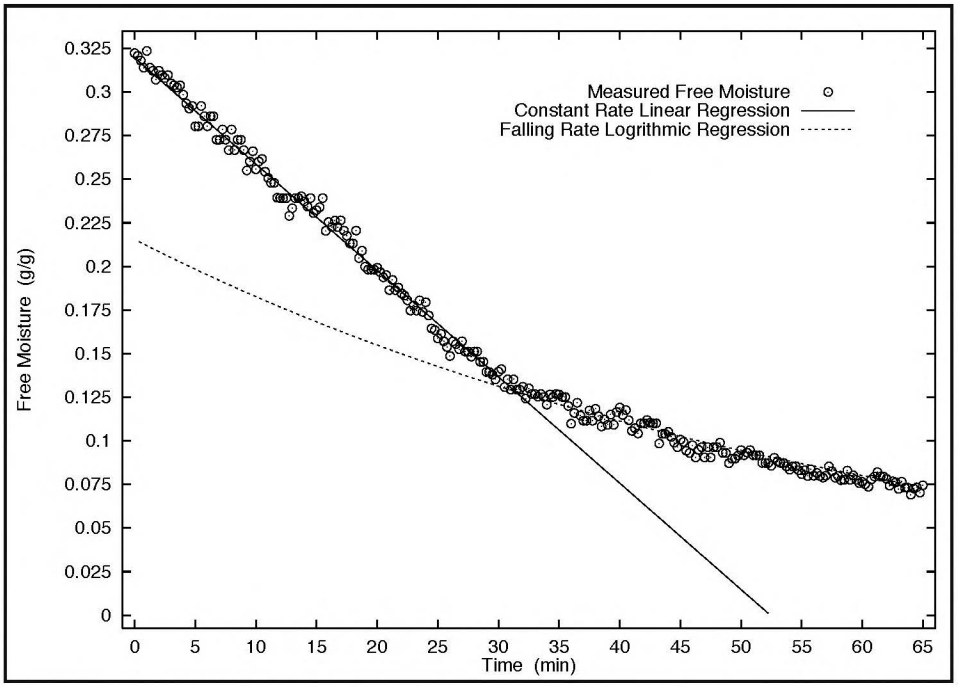

Figure 4. *Free moisture drying curve.*

report and an oral presentation. The formal written report is due 10 days after the last analysis session for the experiment. The oral presentations are scheduled over a series of presentation days at the end of the semester. There are also short memo reports required after each laboratory session to document the planning, experimental, and/or analysis results and conclusions. The different materials and scale-up requirements incorporated into this experiment provide each group a slightly different experience that can be shared with the class during the presentation sessions.

Each student group is composed of three students and there are three experiments in the senior laboratory course. Therefore, each student in the group takes on the responsibility of group leader for one experiment in the sequence. Each group leader is responsible for the formal written report, oral presentation, and short memo reports on their experiment. The group leader responsibility also includes coordinating the activities of the other group members. When the class is not evenly divisible by three, there will either be one two-member or four-member student group. A two-member student group will not prepare a formal written report and oral presentation for one of the experiments although they will carry out this experiment and prepare the short memo reports. A four-member group will be given two objectives with a separate group leader for one of the experiments. Because of the length of the drying experiments, they have not been considered for two objectives in a four-member group.

STUDENT RESPONSE

There are no formal course evaluations for laboratory courses in the chemical engineering department at Villanova University. Student response data for the senior laboratory course is obtained from departmental surveys administered at the end of the semester. Qualitative assessment of the students' experiences is also based on their comments during and after the experiment. This assessment indicates that the experience has been generally well received by the students. Student comments concerning the drying experiment reveal that the group spent more time on this experiment because of the design aspect, which required more use of the second planning and analysis sessions.

CONCLUSIONS

The laboratory experiment documented in this article has been developed and implemented over the past two years in the chemical engineering senior laboratory course at Villanova University. Based on the results of informal course surveys, the students have found the experience both challenging and worthwhile, in addition to providing an applied mass transfer and process design experience. The experiment has also provided valuable documentation of students' ability to design, conduct, analyze, and interpret experiments for ABET[18] Criterion 3b and their ability to perform as part of a team for ABET[18] Criterion 3d.

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REFERENCES

1. Denig, J., E. Wenger, and W. Simpson. "Drying Hardwood Lumber," *Technical Report FPL- GTR-118*, USDA Forest Products Laboratory (2000)
2. U.S. Department of Energy, *Mineral Processing Technology Roadmap*, Office of Industrial Technologies (2000)
3. Clark, J., "Drying Still Being Actively Researched," *Food Technology* **56**(9), 97-99 (2002)
4. Guerrero, M., C. Albert, A. Palmer, and A. Guglietta, "Drying in the Pharmaceutical and Biotechnological Industries," *Food Science and Technology International*, **9**(3), 237-243 (2003)
5. Mujumdar, A., "Research and Development in Drying: Recent Trends and Future Prospects," *Drying Technology*, **22**(1/2),1-26 (2004)
6. McCallum, C., and L. Estevez, "Introducing Process-Design Elements in the Unit Operations Lab," *Chem. Eng. Ed.*, **33**(1), 66-70 (1999)
7. Abu-Khalaf, A., "Getting the Most Out of a Laboratory Course," *Chem. Eng. Ed.*, **32**(3), 66-70 (1998)
8. Steidle, C., and K. Myers, "Demonstrating Simultaneous Heat and Mass Transfer with Microwave Drying," *Chem. Eng. Ed.*, **33**(1), 46-49 (1999)
9. Nollert, M., "An Easy Heat and Mass Transfer Experiment for Transport Phenomena," *Chem. Eng. Ed.*, **36**(1), 56-59 (2002)
10. Moor, A., "A Benchscale Drying Laboratory Illustrating Combined Heat and Mass Transfer," *ASEE Mid-Atlantic Section Conference*, Rowan University, April (2001)
11. Prudich, M., D. Ridgway, and V. Young, "Integration of Statistics throughout the Undergraduate Curriculum: Use of the Senior Chemical Engineering Unit Operations Laboratory as an End-of-Program Statistics Assessment Course," *2003 ASEE Annual Conference*, Nashville, June (2003)
12. Moyers, C., and C. Baldwin, "Psychrometry, Evaporative Cooling, and Solids Drying," *Perry's Chemical Engineers Handbook*, McGraw-Hill, New York, 7th Ed. (1997)
13. Cooper, J., W. Penney, J. Fair, and S. Walas, *Chemical Process Equipment Selection and Design*, Gulf Professional Publishing, Burlington, MA, 2nd Ed. (2004)
14. McCabe, W., J. Smith, and P. Harriott, *Unit Operations of Chemical Engineering*, 5th Ed., McGraw-Hill, New York, (1993)
15. Geankoplis, C., *Transport Processes and Unit Operations,* 3rd Ed., Prentice Hall, Englewood Cliffs, NJ (1993)
16. Treybal, R., *Mass Transfer Operations*, 3rd Ed., McGraw-Hill, New York (1980)
17. Keey, R., "Process Design of Continuous Drying Equipment," *Water Removal Processes,* C. King and J. Clark, editors, AIChE Symposium Series, **73**(163), 1-11. AIChE, New York (1977)
18. ABET, "Criteria for Accrediting Engineering Programs," Engineering Accreditation Commission, <www.abet.org> (2004) ❐