Undergraduate Laboratory Experiment

FACILITATING ACTIVE LEARNING OF CONCEPTS IN TRANSPORT PHENOMENA: EXPERIMENT WITH A SUBLIMING SOLID

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Chemical engineering education is widely considered to have undergone a paradigm shift around the 1960s with the incorporation of transport phenomena in the curriculum.[1] The publication of the text *Transport Phenomena* by Bird, Stewart, and Lightfoot helped popularize the unifying treatment of momentum, energy, and mass transport based on microscopic or molecular description of processes. Whether inclusion of such treatment is warranted at the undergraduate level was debated vigorously in the subsequent decade or so,[2] and despite occasional objections, a course (or sequence of courses) in transport phenomena is firmly entrenched in most undergraduate chemical engineering curricula.[3]

The junior year of the undergraduate chemical engineering program at the University of Idaho features a two-semester-long sequence of mandatory courses entitled Transport and Rate Processes I and II. The text *Fundamentals of Momentum, Heat, and Mass Transfer* by Welty, *et al*.[4] has been used for both courses for a long time by various instructors teaching the courses. Both Transport and Rate Processes I and II are four-credit courses with lecture and laboratory components, and may or may not be taught by the same individual. In a case where two different instructors are used for the course sequence, the syllabi are coordinated to prevent any overlap of topics.

Students typically perform three experiments each semester, with two experiments each related to the three types of transport phenomena. Prior to this author assuming the responsibility for the transport courses, the experiments included viscosity determination, drag coefficient determination using a wind tunnel, thermal conductivity determination, diffusivity determination for a volatile compound using Arnold cell, and two *gedankenexperiments*.

Students typically find mass transport to be the most difficult of the three transport phenomena, possibly due to exposure to concepts in fluid flow and heat transfer in earlier Fluid Mechanics and Engineering Thermodynamics/Heat Transfer courses. However, the thought experiments and analysis assignments therein were not particularly useful in helping the students understand the mass transport concepts. The experiment described below was introduced out of the need to develop a laboratory exercise that would reinforce the mass transport concepts, be easy to understand and conduct, and ultimately, maintain student interest in a complex, mathematics-intensive subject.

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LABORATORY DESCRIPTION

Theory and objectives

Students in Transport and Rate Processes II are concurrently enrolled in the Separation Processes course. As a result, they may already be exposed to the design of separation equipment wherein they use mass transfer coefficients, without truly understanding the theoretical basis, the significance, or the correlations used for obtaining those. The experiment devised was aimed at clarifying these concepts and impressing upon students the importance of the mass transfer coefficient while being in the realm of transport phenomena, not transitioning into a unit operations/separations experiment.

The experiment was based on using a solid that exerts substantial sublimation pressure at ambient conditions. The experiment is similar to and builds upon one that has been described previously by Rodriguez, *et al*.[5] Upon exposure to air, a subliming solid will lose its mass, and the mass flux can be determined by monitoring the mass or volume of the solid as a function of time. Both the diffusion coefficient of the component in air, and the solid-gas mass transfer coefficient can be obtained from the experimentally obtained mass- (or volume-) time data.

The governing equations for the two situations—sphere and cylindrical disc—are as follows:

1. A sphere of subliming solid A:

For mass transfer of A in stagnant air, the pseudo-steady state assumption leads to the following equation for evaluating the diffusion coefficient:

$$\frac{R_g T \rho_A}{2P\ \ln\left(1-y_{AS}\right) M_A}\left(R_t^2 - R_0^2\right) = D_A t \qquad (1)$$

The mass transfer coefficient is obtained by equating the rate of mass (or volume) change to the convective mass transfer

$$k_c = -\frac{\rho_A}{M_A}\frac{R_g T}{P y_{As}}\frac{dR}{dt} \qquad (2)$$

where,

D_A = diffusion coefficient of A
k_c = mass transfer coefficient of A
M_A = molar mass of A
P = total pressure
R = radius of the sphere
R_0 = radius at time t = 0
R_t = radius at any time t
R_g = gas constant
t = time
T = temperature
y_{As} = mole fraction of A in air at the surface of the solid
ρ_A = mass density of A

2. Cylindrical disc of the subliming solid A:

Mass transfer from the two circular faces of the disc (neglecting the mass transfer from the cylindrical surface) is treated as a case of transient diffusion into a semi-infinite medium. The governing equation for this situation is[4]:

$$\left(\frac{\Delta m}{4R^2 M_A}\frac{R_g T}{P y_{As}}\right)^2 = D_A \pi t \qquad (3)$$

Alternately, the governing equation can be expressed in terms of the thickness of the disc, as shown in Eq. (4).

$$\left(\frac{\rho_A\left(z_0 - z_t\right)}{2M_A}\frac{R_g T}{P y_{As}}\right)^2 = \frac{D_A}{\pi} t \qquad (4)$$

The mass transfer coefficient is obtaining by the mass balance as above:

$$k_c = -\frac{\rho_A}{2M_A}\frac{R_g T}{P y_{As}}\frac{dz}{dt} \qquad (5)$$

where,

Δm = change in mass of the disc from initial mass in time t
z = thickness of the disc
z_0 = initial thickness of the disc
z_t = thickness of the disc at time t

The mole fraction of A at surface, y_{As}, is obtained from the saturation pressure of A, P_A^s, at temperature T.

$$y_{As} = \frac{P_A^s}{P} \qquad (6)$$

Experimental measurements of mass and physical dimensions of samples as a function of time are used in the above equations to obtain both the diffusion coefficient D_A and the mass transfer coefficient k_c. (Details of the derivation of the above equations are shown in Appendix A).

Assignment statement

The laboratory assignment handed to the students is shown in Table 1. The salient features of the assignment are:

- *A concise statement of objectives and expected outcomes. Students are required to not only determine the parameters experimentally, but also to compare their results with theoretical predictions.*
- *A group assignment, with students having the responsibility for designing the experiments.*
- *Clear delineation of submissions/deliverables from the students. The required components of the pre-lab and lab report are listed along with the maximum credits for each component.*
- *Submission of an individual summary statement, wherein the student relates the experiment to theoretical concepts covered in the lectures.*

Each group consisted of three or four students, and the pre-lab and laboratory reports were evaluated as a collective submission. The experiments were conducted using mothballs made of naphthalene or discs of p-dichlorobenzene, available at any superstore. The pre-lab report was due one week after assigning the experiment. Groups with satisfactory pre-lab reports were allowed to conduct the experiment over the next week, and the laboratory reports as well as the summary statements were due one week after the completion of the experiment.

TABLE 1
Determination of Diffusion Coefficient and Mass Transfer Coefficient

What: Experimentally determine the diffusivity of the constituent of moth ball/disc and its mass transfer coefficient in air, and compare it to the theoretically predicted value.
Why: Both diffusivity and mass transfer coefficients are important parameters in mass transfer operations.
How: By designing the appropriate apparatus and making measurements.
Pre-Lab Report (15 points) 1. Define the objective of the experiment (2 points) 2. Present the theoretical principle, and identify the parameters that need to be measured (4 points) 3. Describe the apparatus that will be used (2 points) 4. Describe the procedure (5 points) 5. List any possible hazards, precaution/safety measures to be taken, and post-experimental cleanup (2 points)
Lab Report (30 Points) 1. Describe the actual procedure used (7 points) 2. Present the raw data collected (8 points) 3. Present calculations and data analysis, and numerical results (10 points) 4. State the conclusions (5 points)
Summary Statement (5 points) Write a summary statement discussing what has been learned from the experiment within the context of the concepts of Transport and Rate Processes. The pre-lab and lab reports are group submissions.

DATA ANALYSIS

Experimental setup

As no fixed setup was specified, the students designed their own apparatus. Approximately two-thirds of the groups chose to work with naphthalene balls, while the rest of the groups chose p-dichlorobenzene discs. Typically, these were exposed to air either by suspending by a thin wire from a stand or by supporting them on sharp, pointed objects. The students most often drilled a thin hole through the center of the sphere or disc and threaded a wire through this hole to suspend the object. Some groups did not drill a hole but fashioned a wire-loop around the sphere to suspend it. The pointed objects used to support the sphere or disc included push pins, nails, toothpicks, and straightened paper-clips. The other end of this pointed object was fixed in a variety of support material including wooden or foam block or even a Styrofoam cup. Students either measured the mass of the object using a balance, or the diameter/thickness using a Vernier caliper. Students were also creative in designing the flow environment for mass transfer coefficient determination. While many groups set up the apparatus in fume hoods, some groups designed their own flow channels using everyday objects such as a bucket or a shoe box. They cut out holes in sides and put in small fans for a better control of the experimental conditions. Some of the groups who used p-dichlorobenzene discs also sealed the cylindrical surface to restrict sublimation from the circular surfaces only. Yet another group constructed an aluminum foil cylinder the diameter of the disc, constructing essentially an "Arnold Cell" for the solid with the disc at the bottom of the cylinder. Figure 1 shows the schematic representation of these arrangements.

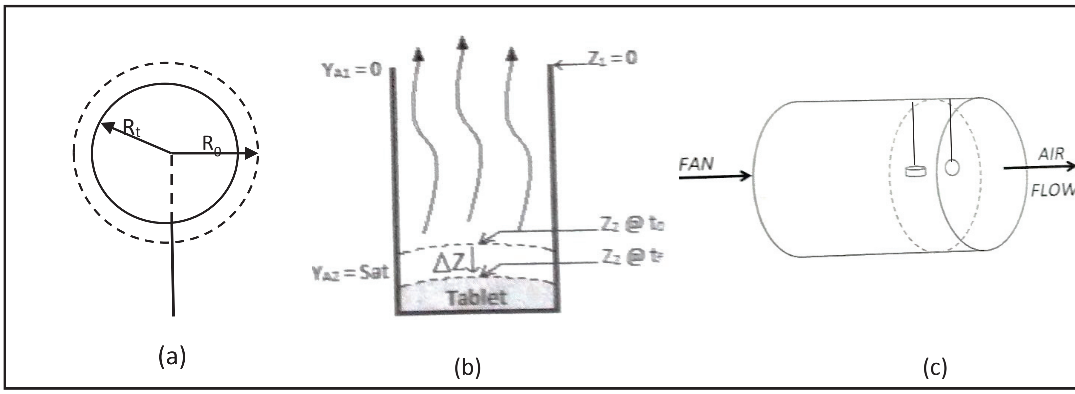

Figure 1. *Schematic representation of Experimental Setup (a) moth ball, (b) "Arnold Cell" setup for p-dichlorobenzene disc, (c) flow setup.*

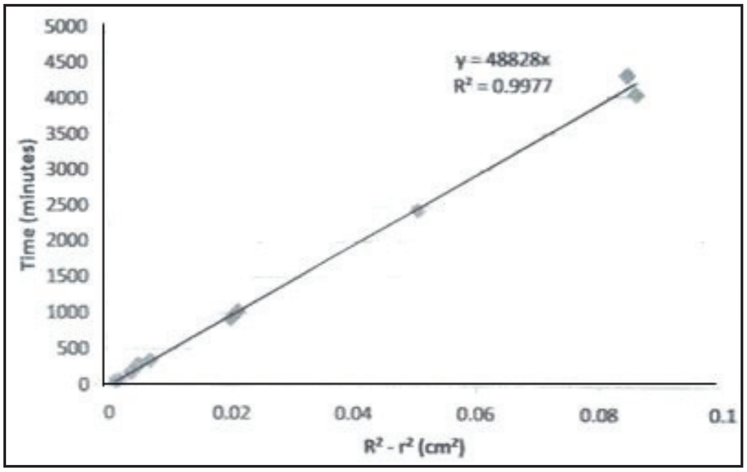

Figure 2. *Data for naphthalene sublimation.*

Numerical results

Diffusion Coefficient

Figure 2 shows a typical graph submitted by the groups showing the data obtained using the naphthalene ball.

As can be seen from the figure, the data follow the trend suggested by Eq. (1). The average diffusion coefficient value for naphthalene across all groups was 0.061 cm^2/s, which is in good agreement with the literature-reported value as well as that calculated using the Fuller-Schettler- Giddings equation, shown below:

$$D_A = \frac{10^{-3} T^{1.75} \left(\frac{1}{M_A} + \frac{1}{M_B} \right)^{1/2}}{P\left[\left(\Sigma v_A \right)^{1/3} + \left(\Sigma v_B \right)^{1/3} \right]^2} \qquad (7)$$

where,

M_B = molar mass of B (air)

Σv_A = sum of diffusion volume of A

Σv_B = sum of diffusion volume of B

It should be noted that considerable variation was observed across different groups, and not all groups were able to replicate the linear behavior shown in Figure 2. The values of the diffusion coefficient for naphthalene ranged from 0.01 cm^2/s to 0.15 cm^2/s. Fewer groups conducted the experiment using p-dichlorobenzene discs. The diffusion coefficients ranged from 0.01-0.06 cm^2/s, with the theoretically calculated value being 0.07 cm^2/s. This discrepancy between the experimental and theoretical values is attributable primarily to the uncertainty in experimental measurements, including those of mass/characteristic length, and air velocity. Further, the experimental specimens did not conform to the well-defined geometric shapes implicit in the calculations shown above. All the groups were able to identify and present explanations for the discrepancy in their values.

Mass Transfer Coefficient

The mass transfer coefficient values ranged from 0.3 cm/s to 0.85 cm/s for both naphthalene and p-dichlorobenzene. Unlike the diffusion coefficient, mass transfer coefficient is a function of the dimension of the solid, and groups presented their data as a function of time or diameter, as shown in Figure 3.

The experimental values of the mass transfer coefficient were compared to those predicted theoretically using the Frössling correlation shown in Eq. (8), or other applicable correlation depending upon the Reynolds number/Schmidt number ranges.

$$Sh = 2 + 0.552\ Re^{1/2}\, Sc^{1/3} \qquad (8)$$

Re = Reynolds number

Sc = Schmidt number

Sh = Sherwood number

The discrepancy in the experimental and predicted values ranged from -20 to -50%, again attributable to the uncertainty of measurements. However, as seen from Figure 3, the mass transfer coefficient increased with decreasing diameter, in accordance with the theoretical predictions.

SUMMARY OF EXPERIENCES

The single most important characteristic that distinguishes this experiment from the rest of the experiments across all undergraduate courses is the absence of a set procedure or apparatus. The students had the total control for designing

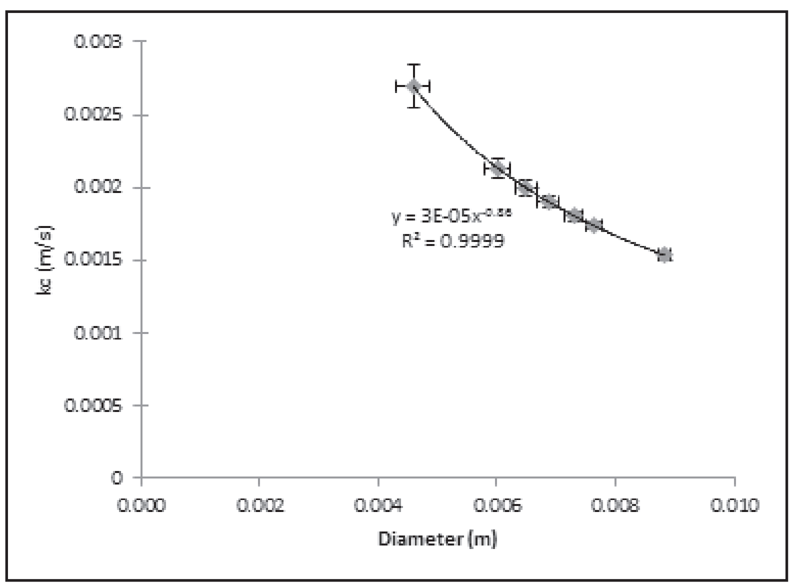

Figure 3. *Mass transfer coefficient as a function of diameter.*

their experimental setup and determining what measurements to make. After some initial apprehension by a few students, most groups readily accepted the challenge. Each group held brainstorming sessions and, working on a tight schedule for the submission of pre-lab, rapidly developed an experimental plan. Most of groups typically submitted their pre-lab reports before the due date, as they were eager to get into the lab to conduct the experiment.

Summary statements

The summary statements ranged widely in their length and content. While a few of the summary statements were brief and merely repeated the numerical results, most of the others contained comments related to the experiment and its value in understanding the transport phenomena. Some of the students also conveyed opinions regarding working in the team environment and the utility of this exercise in designing experiments in general. Some of the comments are presented below:

> *"Our final lab for transport was an enjoyable project. I particularly liked being able to take our idea and create a wind tunnel..."*

> *"I think having to find our own materials in the lab and construct our own apparatus was a useful experience that one doesn't often get in undergraduate labs."*

> *"This lab also allowed us to be more creative, because we got to make our own apparatuses. This again helped reinforce concepts we were learning in the class."*

> *"I feel as though I have a strong understanding of the theoretical principles covered in this lab, because of the independence we were given during the procedure."*

> *"Overall, this experiment helped me understand the concepts from Chapters 26-29 in the Transport book."*

> *"I learned a lot from this experiment socially and educationally. Working with a team is challenging sometimes, however, a lot of benefits could be harvested from it. One of the main benefits is to simulate the real-world job environment from brainstorming to balancing and checking our mistakes together."*

Instructor observations

A laboratory experiment should accomplish the following objectives as elucidated by Miller, *et al.*,[6] and Abu-Khalaf,[7] and re-emphasized by Fogler[8]:

1. *Plan the experimental set and measurements to be made*
2. *Start up and run experiment*
3. *Collect, analyze, and interpret data*
4. *Compare experimental results with theoretical predictions*
5. *Convey the results through clear and concise report and oral presentation*
6. *Work effectively in teams*

The experiment described above meets all of these objectives. Additionally, the students are not merely running the experiment, but actually developing the setup for conducting the experiment. Another important lesson the students learn is that they will frequently encounter a situation where the system they have to work with is not as ideal and well-defined as in the textbooks. However, they will still be required to provide realistic estimates of process parameters that are theoretically consistent. In that sense, this experiment is the closest to what they will come across in their workplace.

Above all, it is the belief of the instructor that any activity assigned to the students must serve to stimulate their thought process and encourage creativity. This experiment can be considered to be a resounding success from this viewpoint. The students were able to respond to the challenge by designing their systems, and *interpreting their results based on the theoretical concepts of transport phenomena.*

A review of the naphthalene sublimation technique has been presented in the past by Sousa Mendes,[9] and the mass transfer measurements has been explained by Goldstein and Cho.[10] The experiment described herein is a novel implementation of this technique with the objective of motivating the students to learn transport phenomena.

Future modifications

The following major modifications will be implemented for reducing errors and improving the experimental accuracy for the subsequent assignments of the experiment.

1. *One of the sources of errors is the uncertainty in the velocity around the objects. An anemometer has been procured for use in these experiments for accurate determination of the velocity.*
2. *The second source of error is the uncertainty in mass/volume measurements. The departure from sphericity becomes quite significant as the naphthalene balls undergo sublimation. Further, the rapid sublimation of both naphthalene and p-dichlorobenzene from samples of relative small initial mass results in amplification of errors in the time derivatives. Using larger samples wherein the rate of material loss is relatively a small fraction of the initial mass will minimize these errors. Highly complex, sophisticated measurement techniques have been proposed in such sublimation experiments, such as using collimated laser light and CCD camera.[11] While such techniques improve the accuracy of measurements, they are expensive and limit the ability of students to be creative with their experiments.*
3. *An additional concept to be incorporated in the experiment will be that of the boundary layer. Students will be calculating the boundary layer thicknesses from the mass transfer coefficient and diffusion coefficient values. The experimental values will be compared to those predicted using the boundary layer theory.*
4. *An important component of transport theory is the analogy between the various transport phenomena.*

Future experiments will involve modifications for obtaining the heat transfer coefficient and using the experiment to enhance the understanding of various analogies.

These modifications will help improve the accuracy of the experimental results and facilitate further understanding of complex concepts in transport phenomena.

CONCLUSIONS

An experiment based on the sublimation of a solid was implemented in the Transport Phenomena course. Students exhibited creative approaches to determine diffusion coefficients and mass transfer coefficients experimentally, and compare the values with theoretical predictions. The experiment proved to be of immense value in helping students understand the complex concepts and increase their interest in the Transport Phenomena course.

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APPENDIX A: DERIVATION OF THE GOVERNING EQUATIONS

Sublimation from solid sphere

The governing equation for steady state diffusion is

$$\nabla \cdot N_A = 0 \qquad (A.1)$$

Where N_A is the molar flux of A.

Symmetry considerations in the spherical coordinate system reduce this equation to:

$$\frac{d}{dr}\left(r^2 N_{Ar}\right) = 0 \qquad (A.2)$$

r is the radial direction coordinate and N_{Ar} is the molar flux in r direction. For diffusion of A through stagnant B,

$$N_{Ar} = -\frac{cD_A}{\left(1-y_A\right)}\frac{dy_A}{dr} \qquad (A.3)$$

Where c is the total concentration.

Since $r^2 N_{Ar}$ is a constant [from Eq (A.2)], substituting in Eq. (A.3) and integrating within the limits $r = R$, $y_A = y_{As}$ to $r = \infty$, $y_A = 0$ yields,

$$N_{AR} = -\frac{cD_A}{R}\ln\left(1-y_{AS}\right) \qquad (A.4)$$

where N_{AR} is the flux at the surface of the solid sphere. The mass balance for A is

$$4\pi R^2 N_{AR} = -\frac{d}{dt}\left(\frac{4}{3}\pi R^3 \frac{\rho_A}{M_A}\right) \qquad (A.5)$$

Substituting for N_{AR} from Eq. (A.4), and integrating between the limits t=0, $R = R_0$, and $t = t$, $R = R_t$, leads to Eq. (1), when ideal gas law is used to express the concentration.

N_{AR} can also be expressed in terms of the mass transfer coefficient k_c,

$$N_{AR} = k_c c\left(y_{As}\right) \qquad (A.6)$$

Substituting in Eq. (A.5) leads to Eq. (2).

Sublimation from a cylindrical disc

The solution to transient mass transfer in a semi-infinite medium is given by Welty, *et al*. as:

$$W_{At} = W_{A0} = S\sqrt{\frac{4D_A t}{\pi}}\left(C_{As} - C_{A\infty}\right) \qquad (A.7)$$

where,

C_A = concentration of A, subscripts S and ∞ referring to radial positions (surface and far away from surface, respectively)

S = mass transfer area

W_A = moles of A, subscripts t and 0 referring to times.

The mass balance on the disc is

$$2Sk_c C_{As} = -\frac{d}{dt}\left(Sz\frac{\rho_A}{M_A}\right) \qquad (A.8)$$

The coefficient 2 accounts for mass transfer from both the circular surfaces. Again, using ideal gas law for concentration leads to Eq. (4). ❐