A NOVEL CHEMICAL ENGINEERING LABORATORY MODEL AT THE UNIVERSITY OF KANSAS

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INTRODUCTION

Laboratory courses provide a tangible means of gaining understanding of abstract concepts and solidifying students' confidence in a curriculum.[1] The University of Kansas (KU) Department of Chemical and Petroleum Engineering (CPE) requires chemical engineering students to complete two Unit Operations Laboratory (UOL) courses during their senior year: Chemical Engineering Laboratory I (CPE616, 4-credit hours) in the fall and Chemical Engineering Laboratory II (CPE626, 3-credit hours) in the spring. This laboratory course sequence strengthens student understanding in areas of chemical engineering theory,[2] laboratory safety,[3, 4] communication of results,[5] and team leadership and time management[6] while practicing fundamental skills to prepare students for careers in industry and academia.[7]

Due to large enrollments and limited time, some UOL's only go in depth (requiring a report and presentation) over one or two experiments per semester.[8-11] The new UOL structure at KU offers six comprehensive experiments over two semesters that cover the fundamentals of chemical engineering. Each experimental cycle follows a sequence with a pre-lab tour, pre-lab meeting and presentation, laboratory experiment, data analysis and modeling, and final lab report. A formal oral presentation or instructional video at the end of each semester brings together all the core aspects of the course, and the instructional videos can be used as a teaching tool for the next incoming class of laboratory students.

The CPE616 course has three experiments that cover the core subjects taught during the junior year in thermodynamics, fluid mechanics, and kinetics. The CPE626 course has three pilot-scale experiments (distillation, absorption, and controls) that are covered in the Design of Unit Operations (CPE611) and Process Dynamics and Controls (CPE615) courses during the first semester, senior year. This article provides an overview of the new course structure and details for the three experiments taught in the first semester UOL CPE616 course. Student feedback about the new course structure has been very positive, and some results are provided for assessment.[12]

CHEMICAL ENGINEERING LABORATORY I

CPE616 is the first course in which KU chemical engineering students apply their knowledge from junior-level core chemical engineering courses to data collected in a lab. The course is taught by three professors (one for each of the three experiments), a lab manager to supervise the course, and a lab assistant to make sure that the day-to-day operation of the lab runs smoothly.

Students rotate through three experiments in CPE616: Vapor-Liquid Equilibrium (VLE), Fluid Mechanics (FLU), and Kinetics (KIN). The VLE experiment uses a four-

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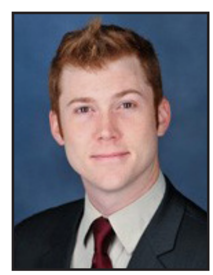

David M. Griffin *directs the undergraduate chemical engineering laboratories at the University of Kansas (KU). Dr. Griffin joined KU in 2013 and has overseen the senior laboratory courses ever since; teaching students as well as commissioning new experiments and maintaining current ones. He received his Ph.D. in Chemical Engineering from the University of Massachusetts-Amherst and his B.S. in Chemical Engineering from the University of Kansas*

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ebulliometer apparatus to find infinite dilution activity coefficients for binary mixtures of three solvents in order to model *Txy*-diagrams and evaluate ternary phase behavior. The FLU experiment measures pressure drop of an incompressible fluid (water) flowing through a variety of pipe segments to determine frictional losses in laminar, transitional, and turbulent flow. The KIN experiment determines reaction orders and activation energy for the iodination (or bromination) of acetone using hydrochloric acid as a catalyst.

The educational outcomes of this course, in alignment with the Accreditation Board for Engineering & Technology (ABET) learning objectives,[13] are to:

1. Reinforce chemical engineering fundamentals taught in lecture courses (such as thermodynamics, fluid mechanics, and kinetics) via specific lab experiments.
2. Plan an optimal set of experiments that meet well-defined objectives.
3. Collect data, analyze, and interpret experimental measurements and compare to existing theories.
4. Estimate error (uncertainty in measurements) and how it affects the final results.
5. Recognize and properly use laboratory safety procedures. Identify major hazards in an experiment.
6. Work effectively in teams by optimal distribution of workload, achieving common objectives within time constraints, and demonstrating leadership skills in a group context.
7. Communicate results and conclusions effectively through both written reports and oral presentations or instructional videos.

The CPE616 course spans a standard 15-week semester. Students attend a weekly 1-hour lecture session and enroll in one of four 4-hour lab sections, either Tuesday or Thursday morning (08:00 – 12:00) or afternoon (12:00 – 16:00). Each experiment is completed over a 5-week cycle, and the semester concludes the final week of class with an oral presentation or instructional video by groups covering one of their first two experiments performed in the lab.

During the enrollment period of the previous spring semester, students receive an email to submit group member requests so experiments can begin the first week of classes. Group size is typically four students in order to distribute the workload, but groups of three are also occasionally necessary. Students choose a group leader for each experiment and the final presentation, which gives every student an opportunity to lead their group. The responsibilities organized by the group leader allow students to participate in a variety of roles and practice individual accountability similar to that seen in industry.[9] The group leader is responsible for communication between the group and with the professor overseeing the experiment as well as distributing and organizing the group workload, submitting assignments, and scheduling group meetings.

At the beginning of the semester, faculty provide several resources to the class, such as equipment operating procedures, safety data sheets, error analysis and statistics guides, and literature relevant to the experiment. These handouts are available upon request from Dr. Mark B. Shiflett at mark.b.shiflett@ku.edu. In the first week students attend a pre-lab lecture conducted by the professor responsible for the experiment, which gives students the opportunity to ask questions and understand expectations. During the laboratory section, groups attend a pre-lab tour where the lab assistant and lab manager show students how to operate and calibrate the apparatus and discuss hazards and safety concerns. Safety is the course's highest priority, and hands-on experience provides an effective way for students to analyze hazards found in a chemical engineering lab.[3, 4] Students also collect some of their initial data during the 4-hour pre-lab tour and begin data analysis.

In the second week, the group conducts a pre-lab meeting with the professor responsible for the experiment. In the pre-lab meeting, students cover lab objectives, safety and hazard precautions (discussed during the pre-lab lecture and pre-lab tour), theoretical background, experimental plan and procedure, literature review, and preliminary data analysis and modeling. Meetings typically last two hours, which provides the time for students to present for 30-35 minutes, the professor to go over additional procedures, and students to ask questions.

In the third week, students return to the laboratory to complete the experiment. During the 4-hour lab section, students operate the experimental apparatus, collect additional data, shut down, and clean up the equipment before the next session. During the fourth week, students submit a draft of the abstract and figures to be used in their final report to the professor responsible for the experiment in order to receive initial feedback before the report is due. At the beginning of the fifth week, students submit their final report and start the next experiment cycle. At the conclusion of the experiment, each member of the group submits a confidential peer evaluation form to rate their own performance and that of their group members on each aspect of the experiment: preparation, presentation, and contribution of work. This evaluation is used to ensure group work was distributed equally and to assign grades fairly.

The overall point distribution for the course and the point value (pts) of each experiment are shown in Table 1. The pre-lab meeting is graded on presentation, underlying theory, experimental plan, data analysis, safety, teamwork, creativity, and initiative. In-lab performance grades are determined by the lab manager and lab assistant based on student safety performance (such as wearing the correct personal protective equipment, proper handling of chemicals, safe startup and shutdown of the equipment), experimental plan preparation, understanding equipment functions, experimental technique, understanding of the limits of the equipment, teamwork and organization, and overall lab etiquette (such as efficient data collection, punctuality, time management, and maintaining a clean work station). Written report grades are based on the abstract, introduction, theory and experimental procedure, results and discussion, conclusions and recommendations, references and appendices, and formatting. In the final presentation students are graded on visual aids, delivery, technical content, and response to questions. All course documents and grade forms are available upon request.

All pre-lab meeting presentations, lab reports, and peer evaluations are submitted electronically via Blackboard, an online platform used by KU for students to access class resources and upload assignments. Blackboard is an effective tool for teaching engineering courses and enhances student performance by streamlining communication and information sharing between students and faculty; online submissions also eliminate the need for hard copies of deliverables.[14] Instructors are expected to provide comments and grade assignments within one week of submission so students can incorporate improvements prior to their next experiment.

In Fall 2018, 89 students were enrolled and organized into 23 groups. In order to accommodate the class size in a limited space, the course was divided into two staggered sequences, A and B, with sequence A performing all tasks one week ahead of sequence B. This staggered schedule effectively doubles the number of students who are able to enroll in the UOL. Table 2 is an example of a weekly schedule that provides details for each sequence and experiment. On days where both sequences A and B are not scheduled for a pre-lab lecture, the lab manager and lab assistant hold lectures addressing topics such as how to write an effective report (particularly the abstract, results, and discussion), calculate uncertainty, prepare a comprehensive literature review, and deliver an oral presentation. Continuous communication between the lab manager, lab assistant, and the professors regarding common difficulties also determines special topics that can be addressed during the lectures.

TABLE 1
CPE616 point distribution.

Course Grade Overview		
VLE Experiment	150 pts	30%
KIN Experiment	150 pts	30%
FLU Experiment	150 pts	30%
Video and Oral Presentation	50 pts	10%
Total Points for Course	*500 pts*	*100%*
Experiment Grading		
Pre-Lab Meeting Presentation		30 pts
In-Lab Performance		10 pts
Peer Evaluations/Discretionary		10 pts
Final Written Report		100 pts
Total Points per Experiment		*150 pts*

In the last week of the semester, after all the students have completed the three experiments, groups either give a final oral presentation or present an instructional video based on one of the first two experiments completed in the lab. Both presentations and videos are approximately 20 minutes long and provide details on the background, apparatus, theory, experimental results, and overall conclusions, followed by a question and answer period with the instructors. All students are encouraged to attend the presentations to learn more about the other experiments from their classmates and the instruc-

TABLE 2
CPE616 course schedule.

Timeline	***Section A***			***Section B***		
Week	**In Lecture**	**In Lab**	**Assignments Due**	**In Lecture**	**In Lab**	**Assignments Due**
1	Pre-Lab Lecture 1	Pre-Lab Tour 1	-	Pre-Lab Lecture 1	-	-
2	-	-	Pre-Lab Meeting 1	-	Pre-Lab Tour 1	-
3	-	Experiment 1	-	-	-	Pre-Lab Meeting 1
4	Pre-Lab Lecture 2	-	Draft of Figures 1	Pre-Lab Lecture 2	Experiment 1	-
5	-	Pre-Lab Tour 2	Lab Report 1	-	-	Draft of Figures 1
6	-	-	Pre-Lab Meeting 2	-	Pre-Lab Tour 2	Lab Report 1
7	-	Experiment 2	-	-	-	Pre-Lab Meeting 2
8	Pre-Lab Lecture 3	-	Draft of Figures 2	Pre-Lab Lecture 3	Experiment 2	-
9	-	Pre-Lab Tour 3	Lab Report 2	-	-	Draft of Figures 2
10	-	-	Pre-Lab Meeting 3	-	Pre-Lab Tour 3	Lab Report 2
11	-	Experiment 3	-	-	-	Pre-Lab Meeting 3
12	-	-	Draft of Figures 3	-	Experiment 3	-
13	-	-	Lab Report 3	-	-	Draft of Figures 3
14	-	-	Final Presentation	-	-	Lab Report 3
15	-	-	-	-	-	Final Presentation

tors. The best instructional videos are then provided as a resource for the next incoming class of students. To reward the best overall group performance, the instructors created an award for the "Best Senior Lab Group," and plaques are given to each student in the winning group at the annual departmental banquet. In addition, a plaque with the names of these students is displayed in the lab for all incoming students to see and serves as an inspiration for future groups.

VLE EXPERIMENT

In the VLE experiment, students calculate infinite dilution activity coefficients of binary mixtures for three solvents and predict isobaric VLE for a ternary mixture.[15] Students first learn multicomponent phase equilibrium in Chemical Engineering Thermodynamics II (CPE512) and become familiar with modeling binary and ternary mixtures using Aspen Plus™ software during the fall semester of their junior year.

Groups begin with three pure solvents (e.g. methanol, acetone, methyl acetate) and add small amounts of solute, keeping the composition less than 0.01-0.02 mole fraction total solute. Students record the equilibrium temperature of the binary VLE upon solute additions and calculate the activity coefficients at infinite dilution using Equation (1).

$$\gamma_j(x_j \to 0) = \gamma_j^{\infty} = \frac{P_i^{vap} - \left(\frac{dP_i^{vap}}{dT}\right)\left(\frac{\partial T}{\partial x_j}\right)_{P,(x_j \to 0)}}{P_j^{vap}} \qquad (1)$$

The infinite dilution activity coefficients can be used to determine the parameters for activity coefficient models such as van Laar, Margules, Wilson, and NRTL to create binary phase diagrams (*Txy*). Students compare their model predictions with literature data[16] and identify which model most accurately predicts known azeotropes found in literature.

Groups input their best-fit model parameters into Aspen Plus to simulate the recovery of a solvent from a ternary mixture using an isothermal or adiabatic flash module. Students also compare simulation results to hand calculations. The design problem scenario proposes that the group works for a company that received a shipment of one hundred 250-liter drums of pure acetone contaminated with methanol and methyl-acetate. Since the purification of the acetone is time sensitive, the company leader proposes that a flash process be used to remove the acetone from the methanol and methyl acetate to purify it. Groups are asked to design an isothermal flash process for the separation and determine the highest purity of acetone that can be recovered. The location of all binary and ternary azeotropes and justification for the design are provided by the students in their reports.

The VLE experiment has several safety hazards that must be carefully considered: chemical toxicity, flammability, high temperature, electrical, sharps, slips/trips/falls, and ergonomics. Students must understand the extent of the health, flammability, and reactivity hazards of the three solvents prior to beginning their experiments. Students are also expected to estimate the amount of each chemical that, if spilled, would exceed the allowable exposure limit (AEL) and lower and upper flammability limits (LFL and UFL, respectively).

Students discuss hazards with the lab instructor during the pre-lab tour, prepare operating procedures and a "what-if" analysis, and present these during the pre-lab meeting. After touring the apparatus, students prepare a Process Flow Diagram (PFD) and Piping and Instrumentation Diagram (P&ID) to document the process. Students learn how to prepare PFDs and P&IDs in the Introduction to Process Dynamics and Control course (CPE615) supplemented with additional information provided in the CPE616 lecture.

The experimental apparatus consists of four 28×500 mm Swietoslawski ebulliometers as shown in Figure 1. Connected to the bottom of each ebulliometer is a 28×150 mm glass boiler equipped with a 156-watt Omega Duel-Element heating tape. At the top of each ebulliometer is a 250 mm double-jacketed condenser cooled by an ethylene glycol solution at 15 °C recirculated by a VWR Benchtop Chiller. Ebulliometers and boilers are custom-built products of Ace-Glass. The top of the condenser is open to the atmosphere, and a Paroscientific Inc. Model 745 barometer monitors ambient pressure. Resistance Temperature Detectors (RTDs) connected to an Omega DP9602 High Accuracy Digital Thermometer are used to measure the VLE temperature and are housed in a glycerol-filled thermowell near the top of the ebulliometer. National

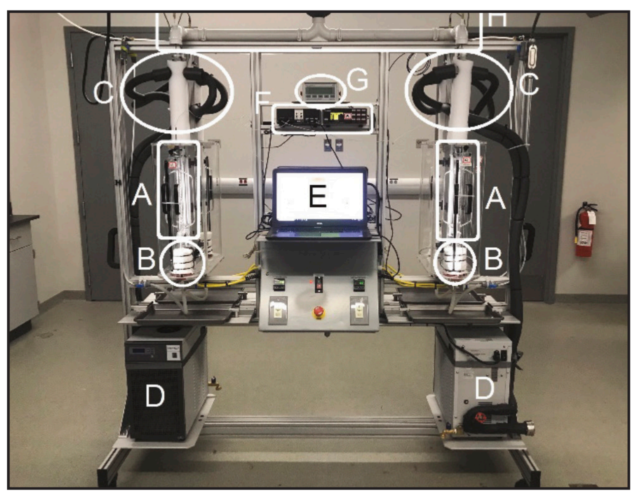


***Figure 1**. VLE experimental apparatus: Solvent is injected into the ebulliometer (A) and heated in the reboiler by heat tape (B). Vapor is condensed in an insulated condenser (C) with cooling provided by a recirculating chiller (D). A laptop (E) records the temperature in the thermowell of the ebulliometer with an RTD thermometer (F). Equilibrium pressure is measured with a high-accuracy pressure standard (G) at the top of the condenser underneath safety exhaust vents (H).*

Instruments™ LabVIEW software and measurement computing hardware are used to control the heating elements and to collect temperature and pressure data as a function of time.

Students use Highland HBC123 milligram-precision scales to weigh the amount of solvent or solute injected into the ebulliometer using a 60 mL syringe (for the solvent) or 250 μL syringe (for the solute). Both syringes use non-coring needles to pierce a rubber septum capping the ebulliometer inlet. After the pure solvent is heated and reaches steady state, the experimental boiling temperature is recorded and compared to the temperature predicted by the Antoine equation at the ambient pressure. Typically, the difference between the measured and calculated boiling temperatures is within 0.05 to 0.1 ºC, which provides students with confidence that their measurements are correct before proceeding further with the addition of solute.

Students typically do four to five solute injections to determine the change in temperature as a function of composition as shown in Figure 2a. Binary phase diagrams (*Txy*) for the van Laar, Margules, and Wilson models are shown in Figure 2b for the methanol + methyl acetate system. The predicted azeotrope points agree well with literature data[16] as shown in the Figure 2b inset.

This experiment teaches students how to assess multiple hazards, measure and model multicomponent phase behavior, and compare their model predictions to literature results. The modeling component builds upon fundamentals the students learn during their junior year in CPE512, and the Aspen Plus simulation provides a "real-world" design problem for students to solve and develop creative solutions based on experimental measurements.

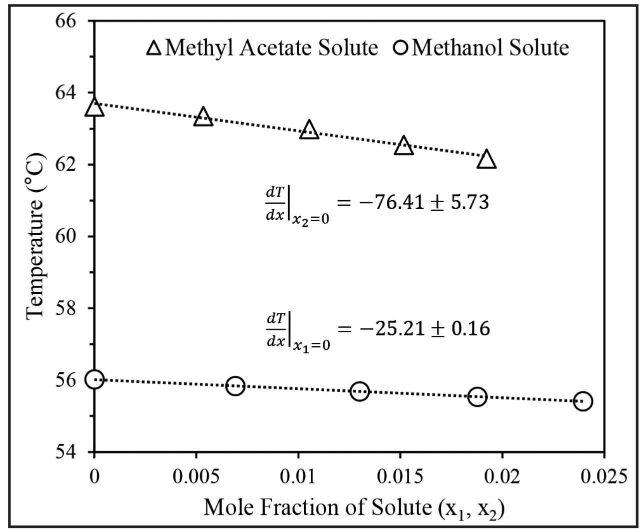


Figure 2a. *VLE experimental results for the change in temperature with composition in the infinite dilution regions for Methanol (1) - Methyl Acetate (2) system. Errors in the T and x directions are smaller than the symbols shown.*

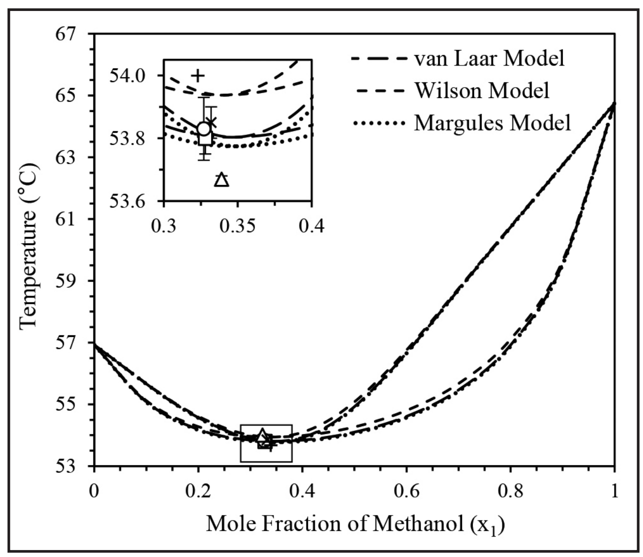
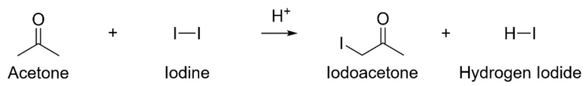


Figure 2b. *T-x-y Diagram of the methanol (1) - methyl acetate (2) system to predict azeotropic behavior from experimental infinite dilution parameters. Azeotrope points from literature are represented by symbols* □[17], ○[18], +[19], Δ[20], *and* ×[21].

KIN EXPERIMENT

Reaction kinetics are an integral part of reactor design and necessary for optimizing chemical production in industry. The goal of the KIN experiment is to determine the rate law, reaction orders, Arrhenius frequency factor, and activation energy for the iodination of acetone. Students also practice simulating a batch reactor using Aspen Plus to compare their measured experimental data with model predictions. In addition, students can scale up the process model to a continuously stirred tank reactor (CSTR) and perform a sensitivity analysis to determine which variable has the greatest impact on the rate of reaction. Students are taught basic concepts of spectrophotometry and rates of reaction in General Chemistry II (CHEM135) and learn how to experimentally determine reaction orders and Arrhenius rate constants using the initial rates method (IRM) in Chemical Engineering Kinetics and Reactor Design (CPE524). The University of Delaware Department of Chemical and Biomolecular Engineering operates a similar KIN experiment in their UOL designed by the corresponding author.[22]

The iodination of acetone reaction shown in Equation (2) provides a simple method to measure reactant concentration, as aqueous iodine has a strong brown color that can be monitored by spectrometry, while all the other reactants and products are colorless. Calibration equations to convert absorbance to iodine concentration have been developed by the laboratory instructors and are provided to the students.



(2)

In this study reactants acetone (Ac) and iodine (I_2) are combined with hydrochloric acid (HCl) catalyst to produce iodoacetone.[23] Students analyze the effect of varying reactant concentration and reaction temperature on the reaction rate. The reaction mechanism for the iodination of acetone is represented by three elementary steps, as shown in Equations (3-5).

$$\mathrm{Ac} + \mathrm{H_3O^+} \overset{k_1}{\leftrightarrow} \mathrm{AcH^+} + \mathrm{H_2O} \qquad \text{(equilibrium step)} \tag{3}$$

$$\mathrm{AcH^+} + \mathrm{H_2O} \overset{k_2}{\rightarrow} \mathrm{Ac^*} + \mathrm{H_3O^+} \qquad \text{(slow step)} \tag{4}$$

$$\mathrm{Ac^*} + \mathrm{I_2} \overset{k_3}{\rightarrow} \mathrm{AcI} + \mathrm{HI} \qquad \text{(fast step)} \tag{5}$$

The first step is an equilibrium reaction between Ac and the acid (such as HCl) to form the protonated acetone intermediate (AcH^+). The second reaction step proceeds more slowly and is the rate-determining step, producing an enol intermediate (Ac^*). The final step is a fast reaction between the intermediate and iodine (or bromine) to produce iodoacetone (or bromoacetone). From this mechanism the overall expression for the reaction can be represented, as shown in Equation (6), where rate is first order in acetone and HCl and zero order in the halogen (iodine or bromine).

$$r = k[Ac]^{\alpha=1}[I_2]^{\beta=0}[HCl]^{\gamma=1} \tag{6}$$

Students determine the experimental orders of reaction in the lab using the Initial Rates Method (IRM) by changing the concentration of one component and keeping the concentration of the other components and temperature constant.[24] For example, to solve for the order of Ac in the reaction, the concentrations for I_2 and HCl as well as the reactor temperature are held constant, so the new rate expression can be defined as shown in Equation (7) where $\mathrm{K} = k[I_2]^{\beta}[HCl]^{\gamma}$.

$$r = \mathrm{K}\,[Ac]^{\alpha} \tag{7}$$

Students plot experimental data using a linearized form of Equation (7), shown in Equation (8), to determine the order of each reactant.

$$\ln(r) = \alpha(\ln[Ac]) + \ln(\mathrm{K}) \tag{8}$$

Students typically perform 12-14 experiments with three runs each varying Ac, I_2, and HCl concentrations to determine the reaction orders. They perform three to five runs with a fixed reactant concentration and different reactor temperatures to determine the Arrhenius frequency factor and activation energy. Students also analyze how experimental errors in concentration and temperature lead to uncertainty in reaction orders and activation energy, and compare experimental results with literature and Aspen simulations.

Similar to the VLE experiment, students utilize part of the pre-lab tour to analyze hazards, prepare PFDs and P&IDs, and think through "what-if" scenarios. Since HCl is a corrosive chemical, the KIN experiment requires students to wear splash goggles, lab coats, and Neoprene gloves. In addition, students begin experimentation during the pre-lab tour, typically holding the concentration of reactants constant and performing four to five runs at different temperatures to determine Arrhenius parameters that are used for their data analysis during the pre-lab meeting with the instructor.

As shown in Figure 3, the apparatus contains two 1000 mL Pyrex® jacketed glass reactors (one on each side of the rack) agitated by ServoDyne mixers operating at 300 revolutions per minute (RPM). Experiments are conducted in batch mode with reactants added manually through a port at the top of the vessel. The four reactor ports contain a type K thermocouple to monitor temperature, a sampling line to circulate reactor contents for absorbance readings, and two capped injector ports. A Cole-Palmer Masterflex L/S Compact Drive peristaltic pump continuously circulates the reactor solution through a flow cuvette installed in a Jenway 6320D spectrophotometer for absorbance measurements. Reactor temperature is maintained by a Solid State Cooling Systems isothermal bath. After the reaction is complete, the remaining solution in the reactor is drained into a waste container for disposal. Data from the thermocouple and spectrometer are collected on a laptop computer running LabVIEW.

***Figure 3**. KIN experimental apparatus: One-liter, jacketed glass reactor (A) with temperature-controlled recirculator (B). An overhead agitator (C) mixes the reactor with the agitation speed set by a motor controller (D). A peristaltic pump (E) circulates reactor contents to the in-line spectrophotometer (F). Absorbance and the reactor temperature from the thermocouple (G) are recorded and displayed on the laptop (H). Waste is drained into containers (I).*

In preparation for each run, students use Ohaus Scout scales to weigh reactants before adding them to the reactor. The initial experiment recommended by the lab instructor during the pre-lab tour requires students to calculate how to prepare a solution containing 1.0 M Ac, 0.01 M HCl, and 0.001 M I_2 run at a temperature of 30.0°C. Subsequent runs vary either concentration or temperature, and data are compared to the initial experiment. Iodine is added to the reactor last to read the absorbance in the very early stages of reaction initiation. Since the reactor is not insulated, heat is lost through the outer wall, and students must be diligent to control the isothermal bath to maintain the desired reactor temperature.

Students convert absorbance data to iodine concentration using calibration information provided by the instructors and determine the initial rate of reaction. Using the IRM with linearized and non-linearized forms of the rate equation, they determine the orders of the reaction and parameters of the Arrhenius equation. The linearized rate equation with the IRM simplifies the analysis, but measurements and additions must be done carefully, with consistent technique, to minimize errors in results and ensure the other independent variables remain constant. In addition to the linearized analysis techniques, students use a Non-Linear Regression Method (NLRM) to simultaneously fit all experimental runs to determine reactant rate orders, activation energy, and frequency factor. For the NLRM, students use either MATLAB®, POLYMATH™, or Excel Solver for their data analysis.

An example of the iodine concentration as a function of time and temperature is shown in Figure 4a. The rate of reaction for each temperature can be determined once the rate constants are known and is used to determine the activation energy (*Ea*) and Arrhenius frequency factor (*A*). Data used for determining the reaction order for acetone are shown in Figure 4b, which is close to the theoretical order of $\alpha = 1.0$ described earlier. Students then use Aspen Plus to compare experimental results (iodine concentration versus time) with a simulated batch reactor (RBATCH) as well as conduct a sensitivity analysis using a CSTR reactor to determine which experimental variables and parameters have the largest effect on the production of iodoacetone.

The KIN experiment typically takes the entire four-hour lab to complete, with 10-15 minutes for each of the 12-15 runs and 15 minutes for preparation, shutdown, and waste disposal. Reaching and maintaining isothermal temperatures can also take a substantial amount of time. Groups are encouraged to do as many runs as possible to minimize error in the regression models and to replicate any questionable data.

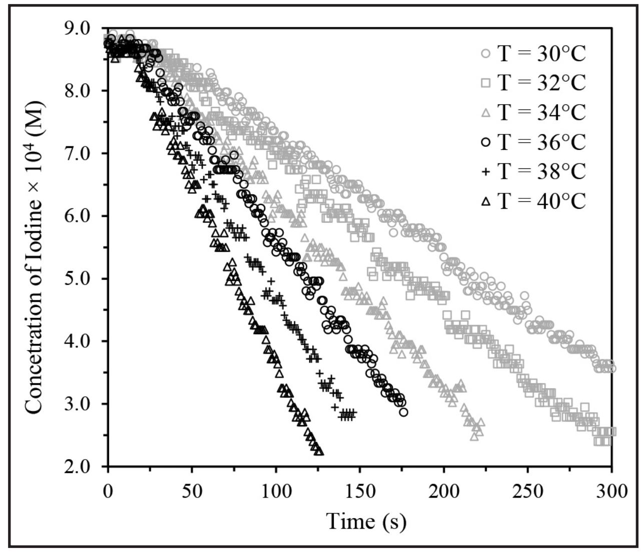


Figure 4a*. The effect of changing the reactor temperature from 30 °C to 40 °C for the KIN experiment. Reaction Conditions: [HCl] = 0.01 M, [Ac] = 2.0 M, and [I_2] = 0.001 M.*

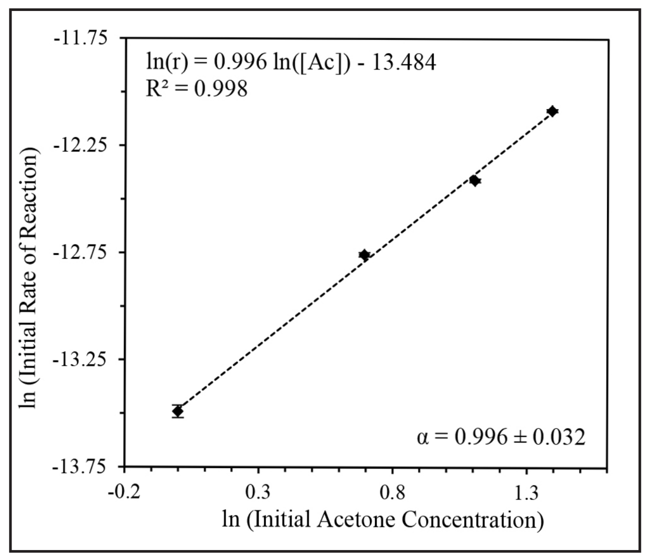


Figure 4b*. IRM to determine acetone reaction order (α) using change in iodine concentration as a function of time (i.e. rate) with reaction temperature held at 30.0°C. Errors in the natural logarithm of acetone concentration are smaller than symbols shown.*

FLU EXPERIMENT

The goal of the FLU experiment is to determine friction factors for an incompressible fluid (i.e. water) flowing through pipes with different diameters and roughness, as well as determine loss coefficients for valves and fittings. Students initially learn about incompressible flow, different flow regimes, and friction factors in Momentum Transfer (CPE511). There are 11 pipes/tubes in the lab with inside diameters ranging from 7.2 – 26.8 mm (0.28 – 1.057 inch) made of three materials (steel, copper, and PVC), with an additional smaller diameter steel tube (4.3 mm, 0.16 inch) and an electropolished steel tube (10.2 mm, 0.403 inch). Three types of 19.05 mm (0.75

inch) valves are also available for students to investigate: a gate valve, a ball valve, and a globe valve. Students choose a minimum of six pipes (two diameters of each of three construction materials) and one valve for their experimental study and are required to investigate the flow behavior in all three flow regimes (turbulent, transitional, and laminar). The flow regimes are defined by different ranges in Reynolds number (turbulent $Re > 4000$, translational $4000 > Re > 2100$, and laminar $Re < 2100$) and are investigated by varying the volumetric flow rate. Reynolds number incorporates the physical properties of the fluid (e.g. density, ρ ; viscosity, μ; diameter, D; and velocity, v) and conditions and conditions for the flow in the system as shown in Equation (9).

$$Re = \frac{\rho(T)\ D\ v}{\mu(T)} \tag{9}$$

Students begin their analysis with a simplification of the First Law of Thermodynamics to apply Bernoulli's mechanical energy balance, Equation (10), for the flow through a pipe.

$$\Delta\left(gz + \frac{v^2}{2} + \frac{P}{\rho}\right) + \mathcal{F} = \frac{\dot{W}_s}{\dot{m}} \tag{10}$$

Pressure drop and volumetric flow rate data are used in the mechanical energy balance to determine the friction loss coefficient, $\mathcal{F}$, which is used with the Darcy-Weisbach equation, Equation (11), to calculate friction factor, f.

$$\mathcal{F} = 4f\left(\frac{\Delta x}{D}\right)\left(\frac{v^2}{2}\right) \tag{11}$$

The apparatus uses two 208 VAC 3-phase WEG electric motors to pump water from a large holding tank into horizontally arranged pipes. A hand-held thermometer monitors water temperature in the tank and is used to estimate the density and viscosity of the water with 8.25% chlorine bleach added as an antimicrobial. The apparatus has ten locations for pipe attachments, five on each side of the apparatus. Pipes are easily removed to allow students flexibility in designing their experimental plan as well as to provide the option of expanding the experiment for different pipes/tubes in the future. Ball valves at the end of each line are used to isolate flow through the desired pipe of interest. Pressure drop across the pipe is measured by an OMEGA PX409 differential pressure transducer that is connected to the pipe of study by quick-disconnect fittings. A laptop computer with LabVIEW software controls the pump speed using a Schneider Electric Altivar ATV320 variable frequency drive and records differential pressure measurements and volumetric flow rate obtained from OMEGA flowrate sensors. A picture of the apparatus is shown in Figure 5.

Chlorine bleach (8.25%) is added to the water to prevent algal growth in the piping and instrumentation, and students are required to identify any hazards associated with its use. In addition, students must evaluate electrical, slip/trip/fall, and ergonomic hazards. Students are required to immediately clean up any spills to avoid slips/trips/falls and prevent electrical hazards. Similar to the other experiments, students utilize part of the pre-lab tour to analyze safety hazards, prepare PFDs and P&IDs, and think through "what-if" scenarios. Students are expected to compare experimental results to two or three literature correlations for friction factor and at least one set of literature values for the valve loss coefficient. As an additional design problem for the experiment, students are asked to estimate the total power consumption of the pump (assuming a constant pump efficiency) for a complete circuit of the fluid in the apparatus.

In preparation for this experiment, groups must calculate desired flow rate ranges to test based on the expected Reynolds number for flow through the pipe. Using samples of the pipes, students measure inner diameters and estimate roughness of each pipe using a roughness gauge. Lengths between the two pressure taps on each pipe are also found using a tape measure. It is recommended that students collect at least 12 data points per pipe (four in each flow regime), but more data points can significantly reduce uncertainty and help in identifying trends in the data.

Friction factors are plotted against Reynolds number to reproduce Moody diagrams and to compare to literature correlations. An example of experimental behavior of the Darcy friction

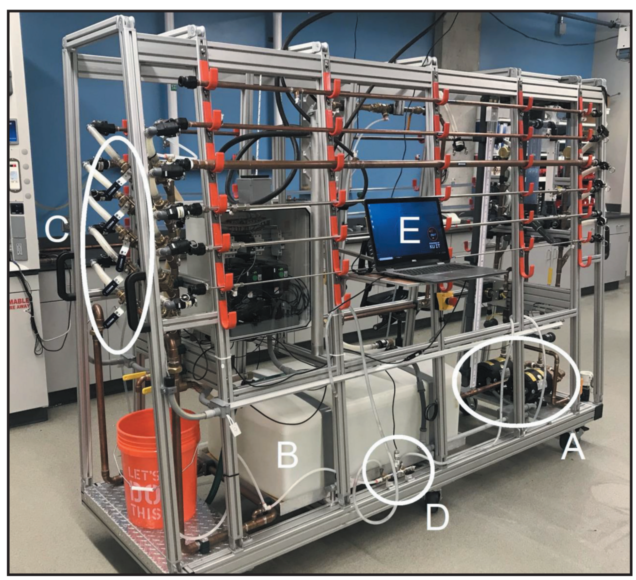


***Figure 5**. FLU experimental apparatus: Electric centrifugal pumps (A) transport water from the tank (B) through the five pipe attachments with flow isolation controlled by ball valves (C). A differential pressure transducer (D) measures pressure drop between two taps located on the pipes. A laptop computer (E) displays and records data from the pressure transducer and flowrate sensors (located above motors, not shown in figure).*

factor with changing Reynolds number for a 7.58 mm (0.299 inch) diameter steel pipe is shown in Figure 6. Students also use the pressure drop and volumetric flow rate data to determine equivalent lengths and loss coefficients for a valve opened to 25%, 50%, 75% and 100% to verify the friction loss behavior.

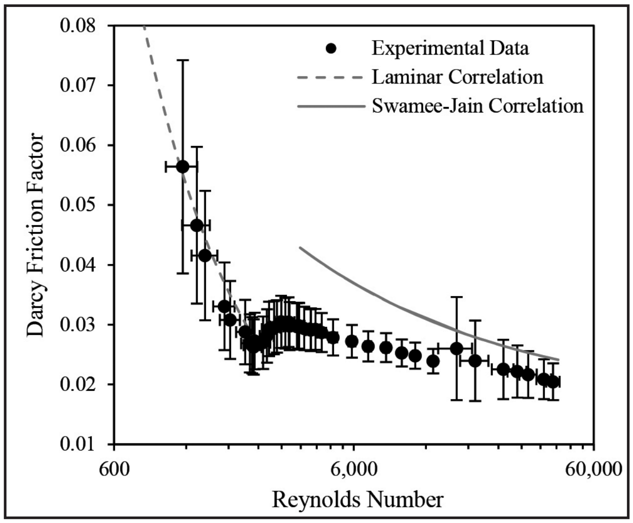


Figure 6*. FLU experimental results for a 7.58 mm (0.299 inch) ID and 2.02 m (79.5 inch) long steel tube showing different flow regimes.*

This experiment reinforces topics taught in CPE511, such as the first law of thermodynamics, Bernoulli's equation, K-factors and the equivalent length method, flow regimes (turbulent, transitional, and laminar), Reynolds number, and friction factor equations. Students also determine which friction factor model statistically fits best with the experimental data.

A unique feature for all of the experiments (VLE, KIN, FLU) used in the CPE616 course is that each one is mounted on wheels and can be easily moved to effectively utilize limited lab space and to transport the apparatus to the classroom for display and instruction in the junior level core chemical engineering courses. A key feature for all of the experiments is an "Emergency Stop" button that, if depressed, will safely shut down the experiment (i.e. turn off motors and heaters) but will continue to monitor variables such as temperature and pressure for observation.

STUDENT FEEDBACK

An informal survey was conducted to evaluate student opinions about the new laboratory experiments and course structure. Responses were received from 75 of the 89 students (84%) enrolled over five sections of the course in Fall 2018. Students were generally positive on the inclusion of multiple instructors teaching each section, with 57% explicitly stating they enjoyed the new experiments and lab structure with only one student specifying a preference toward having a single instructor overseeing all experiments. Respondents noted benefits including that instructors were able to specialize on a single experiment as well as providing continuity and an opportunity for students to interact with a greater number of faculty members. Students specifically mentioned that the new lab structure "fosters a good relationship with the lab instructors" and "makes the CPE courses feel more connected with the junior level core chemical engineering courses."

The most common concern for students was consistency between multiple instructors, particularly in grading (48% of respondents). Though numerous students noted fairness in grading should improve with the new format, students' primary concern regarded each instructor having different expectations and preferences for the written report. This concern was addressed by preparing a detailed rubric for reports and presentations that is consistent for all instructors and available upon request. An additional concern for students was receiving scores and grades on reports before beginning the next experiment. This concern is addressed by instructors returning graded reports within one week of submission, posting scores on Blackboard after the completion of each experiment, and being transparent with students on the quality of their work.

Faculty continue to make improvements to CPE616, both in the instruction and the experiments, based on student recommendations and feedback provided through course evaluation forms and peer evaluation forms. A recent example of modifications made for the Fall 2019 semester was to increase the course credit hours from three to four by adding another lecture session for additional instruction time and providing additional time for groups to collect preliminary data during the pre-lab tour. Though these changes are still quite recent, groups appear more comfortable with writing reports, making oral presentations, and operating the equipment with up to eight hours of time for completion of each experiment.

Modifications to the experiments from one year to the next have included changing the components for the VLE experiment, substituting bromine for iodine in the KIN experiment, and supplying new pipes and valves for the FLU experiment. In addition, a series of Aspen Plus video tutorials has been created and provided online for students to learn at their own pace. The videos are freely available to other instructors on our website (www.shiflettresearch.com). Future plans for the UOL include constructing a pilot-scale distillation column in a high-bay area, adding a control room, and building a new portable rack-mounted controls experiment for analyzing liquid level, temperature, and pH control.

CONCLUSIONS

In CPE616, students apply theory taught in previous courses (specifically thermodynamics, kinetics, and momentum transfer), practice safe operation, identify areas of risk and hazards, design and conduct experiments to achieve objectives, analyze and interpret data, calculate experimental uncertainty and discuss its effect on the overall results, and communicate results through written reports and oral presentations. For many students the UOL is the first course where many of these topics are covered. Most often the major challenge that groups face is developing soft skills such as learning how to work efficiently as a team, writing a group report, preparing a group presentation, and effective time management.

The new course structure effectively doubles the number of students who can be accommodated each semester with limited lab space and includes three in-depth experiments that cover the core fundamentals in chemical engineering. Experiments were designed in-house that efficiently use the lab space, are portable, and can be brought into the classroom. The data are high quality and reproducible with literature sources. Feedback from students has been positive and supports the continuation of this lab structure model.

A rigorous chemical engineering lab course that supports the fundamentals taught in previous chemical engineering core courses such as thermodynamics, fluid mechanics, and kinetics is an essential part of the undergraduate curriculum. Departments should provide significant resources to these UOL courses so that students not only apply the theory from the classroom but can also demonstrate what they have learned in the laboratory. The two-semester UOL course sequence at the University of Kansas prepares chemical engineering students for industry and academia with important skills such as application of theory with experiments, effective report writing and oral communication, time management, leadership, and how to identify and minimize safety hazards.

DEDICATION AND ACKNOWLEDGMENTS

The corresponding author (MBS) would like to dedicate this article to the memory of Professor Prasad Dhurjati who taught at the University of Delaware in the Department of Chemical and Biomolecular Engineering from 1982 to 2020. Prasad inspired the structure of our new laboratory model, loved working with students, and will be sincerely missed for his wisdom and friendship. The authors also thank Mr. George Whitmyre at Whitmyre Equipment Company (Elkton, MD) for assisting us with the design and construction of the three experimental apparatus.

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