Incorporating Inquiry-Based Projects into the Early Lab Experience

Shannon L. Servoss and Edgar C. Clausen
Ralph E. Martin Department of Chemical Engineering
University of Arkansas

Abstract

In using the traditional approach for teaching laboratory, the instructor provides detailed instructions for completing the lab and, after performing the lab, students report results in either a written or oral report. This approach effectively introduces the techniques and technical writing skills required of all engineers, but the process also has a tendency to become a bit redundant for both the instructor and the students. Chemical Engineering instructors at the University of Arkansas have addressed this problem by introducing an inquiry-based learning approach in the introductory laboratory during the Fall and Spring semesters of the 2011-2012 school year. The inquiry-based laboratory still introduces the students to technical writing and simple measurements, but also creates interest in chemical engineering by allowing students to design experiments on their own. As expected, student feedback revealed some frustration with the vague nature of the inquiry-based learning approach but, overall, the students appreciated the challenge and acknowledged that the problems were interesting and useful.

Introduction

In contrast to the traditional note-taking/homework/test approach to learning, inquiry-based learning involves an active approach to acquiring knowledge, including decisions on how and where to obtain information, discerning relevance and applying the knowledge.[1] The instructor's role in the process includes:[2]

- specifying the objectives of the lesson or exercise,
- making the instructional decisions, such as the size of groups and the method of assigning students to groups,
- clearly explaining the task,
- monitoring the learning process and providing assistance as needed, and
- evaluating the group process and student learning.

Examples of inquiry-based learning applied in engineering education include the development of design problems for the biomedical engineering[3] and environmental engineering[4] classroom, the use of virtual laboratories in place of traditional capstone design classes[5] and the introduction of rather ill-structured problem-based learning (PBL) projects throughout the curriculum.[6, 7]

The purpose of this paper is to describe the inquiry-based approach as practiced in the introductory laboratory course in chemical engineering at the University of Arkansas. The selected inquiry-based projects are presented and discussed, as well as laboratory organization and student feedback from the initial two offerings of the course.

Background

Unlike most Chemical Engineering programs in the Midwest, the University of Arkansas offers a two-hour introductory laboratory course in Chemical Engineering (1 ½ -2 hours per week in the laboratory, and 1-1 ½ hours of drill to discuss report writing and data analysis). With the relatively recent addition of Freshman Engineering, which occupies the freshman year, Chemical Engineering students now take the course, CHEG 1212 - Chemical Engineering Laboratory I, in either the first or second semester of their sophomore year.

Chemical Engineering Lab I has been traditionally used to introduce students to simple measurements common to chemical engineering, as well as technical report writing. In a typical semester, the students work in groups of 3-4 to perform 6-8 of the experiments listed in the first column of Table 1 and then generate memo or short form reports on the results of their experimentation. Memos are 1-2 page reports in memorandum form that have experimental results in the form of tables or graphs appended. Short form reports are full-blown laboratory reports with a very limited review of the literature and an abbreviated discussion of results. Near the end of the semester, the student groups perform an experiment that contains design elements, such as the projects listed in the second column of Table 1.

Table 1. Traditional Experiments Used In Chemical Engineering Lab I

Experiments
• Using density to estimate the concentrations of aqueous solutions • Using viscosity (tube viscometer) to estimate concentrations of aqueous solutions • Measuring viscosity of viscous liquids (falling ball viscometer) as a function of temperature • Measuring concentration with a spectrophotometer • Measuring the thermal conductivity of a solid • Preparing drying curves for the drying of various solids • Measuring concentration with a gas chromatograph • Calibrating pressure gauges with a dead weight tester • Using a sieve shaker to determine the size distribution of a mixture of solids • Using acid-base titration for measuring concentration • Using a pH meter for measuring concentration • Determining the settling rate of solids in a slurry • Filtering solids from slurries • Measuring the Reid vapor pressure
End of Semester Projects
• Comparing pressure-assisted slurry filtration with the plate-and-frame filter press • Aerobic fermentation of sugar to produce cell mass • Determining the effect of variables on the distribution coefficient for extracting acetic acid with organic solvents

Although this traditional approach for teaching laboratory is effective in introducing measurement techniques and the technical writing skills needed in engineering, the course also has a tendency to become routine for both the students and the instructor. As an alternative, Lab

I instructors introduced inquiry-based learning into the laboratory during the Fall and Spring semesters of the 2011-2012 school year. Inquiry-based laboratory still introduces the students to technical writing and the simple measurements required of all chemical engineers, but the new approach also creates interest in chemical engineering by posing an engineering problem for the students to solve within the limitations set forth in the experimental description.

Course Structure

As applied in Chemical Engineering Lab I, inquiry-based learning is a multi-step process for both the instructor and students:

- Step 1. Students in groups of 3-4 are given a brief summary of an engineering problem that includes a list of the materials and supplies that are available for solving the problem
- Step 2. Students collect information from the literature and develop a plan for solving the problem
- Step 3. The student group prepares a pre-lab report for approval prior to initiating lab activity
- Step 4. The student group performs the experiment
- Step 5. Each individual student (or, in some cases, student group) submits a written report, first as a draft and then for final approval

Each of these steps is discussed in detail in the following paragraphs.

Step 1. Problem Statement

Figures 1 and 2 are examples of inquiry-based problem statements that were given to the student groups. Other than the brief problem statement and operating protocol on the use of pertinent equipment, no other background information was provided to the students.

CHEG 1212L

Experiment C: Membrane Separation

Teaching Objective: Students will learn to design experiments and become familiar with proper lab procedures, technical writing, and proper citations.

Materials Provided:

- Membrane system
- Membrane (50 and 100 kDa cut-off)
- Unknown protein sample
- BCA protein quantification kit
- UV spectrophotometer
- General lab supplies

Instructions: Membranes are commonly used to separate the components of a mixture when it is advantageous to not use heat. Membrane separations are particular useful in the food industry, biotechnology, and pharmaceuticals. In this experiment, you will be testing a membrane system for its ability to allow a specific protein pass through the membrane, or permeate the membrane.

Figure 1. Inquiry-Based Problem Statement, Example 1

CHEG 1212L

Experiment D: Gas Chromatography

Teaching Objective: Students will learn to design experiments and become familiar with proper lab procedures, technical writing, and proper citations.

Materials Provided:
- Gas Chromatography Instrument
- Injection syringe
- Ethanol
- Methanol
- Isopropanol
- n-Butanol
- Acetone
- Unknown ethanol sample
- General laboratory supplies

Instructions: Gas chromatography (GC) is used to analyze and quantify mixtures of volatile compounds. You may have seen this technique used (often incorrectly) on your favorite forensics show. In this experiment, GC will be used to analyze mixtures of volatile organic compounds, including samples containing an unknown percentage of ethanol. Your first step will be to determine an appropriate internal standard to use for the experiment. You will then analyze the unknown ethanol sample using the internal standard. **Instructions for sample injection are given on page 2.**

Figure 2. Inquiry-Based Problem Statement, Example 2

Step 2. Information Collection

The student groups were given approximately five days to collect background information and to develop a plan for collecting data to solve each engineering problem. As expected, most of the information collected was from the web (because it is easy), which can be a source of good or bad information. Web use thus becomes a part of the learning process, and determining the validity of web information was discussed in drill. Students who have tried to locate background information and prepare a plan, but are "stuck", are directed to appropriate websites for help.

Step 3. Pre-lab Report

The pre-lab report must contain a brief statement of purpose, a list of required materials, an experimental plan, a job safety assessment form and literature references. A typical pre-lab report, minus the safety assessment forms, is appended. The pre-lab report must have enough detail such that another student group can run the experiment without assistance. After pre-lab preparation, the report is submitted through an on-line course site for approval by the instructor. The instructor provides feedback, and the report may be approved by the instructor, or approved after modification. Students must have pre-lab approval before running the experiment.

Step 4. The Experiment

The lab period of 1 ½ - 2 hours is available for the students to perform the experiment. The lab is performed in the presence of graduate teaching assistants (TAs).

Step 5. Reporting
A draft written report is submitted to the instructor after performing the lab. These reports may be individual reports or group reports, either a memo or short form, at the discretion of the instructor. A final written report is then prepared and submitted after the graded draft is returned by the instructor and revised by the student(s) as needed. Two oral presentations are made by each group during the semester on inquiry-based lab topics selected by the instructor.

Inquiry-Based Project Timetable
Table 2 shows the timetable for assignments and deliverables for the inquiry-based labs. All problem statements, as well as the lab schedule, are available to the students at the beginning of the semester. The pre-lab report is due two days prior to the lab, and must be approved prior to performing the lab. A draft written report is due one week after performing the lab. The final written report is then due one week after the graded draft is returned. Two group oral presentations are made per semester on lab topics selected by the instructor.

Table 2. Timetable for Inquiry-Based Labs

Time	Assignment/Deliverable
Two days prior to lab	Pre-lab report due
Prior to day of lab	Obtain pre-lab report approval
Lab day	Perform experiment in the lab (TAs)
One week after lab	Draft report due
One week after graded draft returned	Final report due
Twice per semester	Oral presentations

Inquiry-Based Experiments
Table 3 shows a list of lab experiments used during the Fall 2011 and Spring 2012 semesters. All of the exercises were inquiry-based because limited direction was given in solving the stated problem. However, some of the experiments required much more effort in preparing an experimental plan than others because of complexity. These more complex assignments are marked with an asterisk (*) in Table 3, and a sampling of these experiments is discussed in more detail in the following section. As is noted in the table, ten experiments were performed in Lab I during the Fall semester, while only five experiments were performed during the Spring semester. This difference occurred when, during the Fall semester, the instructor realized that students needed draft reports and instructor input prior to submitting the final report. Thus, draft reports (with instructor input) were added to all assignments during the Spring semester, which resulted in less, but more meaningful experimentation.

Table 3. Lab I Experiments, 2011-2012

Fall Semester 2011
• Preparation of ice water for a hot day • Preparation of pH paper from household items* • Determining density for solids and liquids, with comparison to literature values* • Determining viscosity for three liquids, with comparison to literature values • Determining settling rates of solids

• Determining the thermal conductivity of solids • Gas chromatography for separating mixtures of alcohols • Sugar extraction from biomass* • Fermentation and CO_2 production* • Protein quantification
Spring Semester 2012
• Preparation of pH indicator from household items* • Gas chromatography with internal standard* • Membrane recovery of BSA protein* • Acetic acid extraction through liquid-liquid extraction* • Preparation of drying curves for the drying of solids

*Complex assignment

Description of Selected Experiments
The sugar extraction experiment involved hydrolyzing the complex carbohydrate in algae and other biomass materials through size reduction and chemical treatment. Students selected the biomass material and hydrolysis conditions to maximize the recovery of fermentable sugars. This experiment was first used in the University of Arkansas Engineering Summer Academy (ESA) for rising 11th and 12th graders.

The purpose of the fermentation experiment was to investigate variables affecting the fermentation of sugars to ethanol using the yeast *Saccharomyces cerevisiae*, such as sugar type and concentration, nutrients concentration, agitation rate and temperature. The students were required to select the experimental conditions. This experiment was an improvement over a relatively simple experiment used in ESA.

The purpose of the inquiry-based gas chromatography (GC) experiment was to select an internal standard for use in the analysis of ethanol/water mixtures, briefly described in Figure 2. GC has traditionally been an integral part of Lab I, but internal standards were not used in GC analysis.

Membrane separation was a protein separation process that was briefly described in Figure 1. Two membranes (50 kDa cutoff and 100 kDa cutoff) were tested for their ability to separate bovine serum albumin (BSA) protein from solution. This experiment grew out of faculty research efforts, and had not been used previously in summer workshops or teaching laboratories.

The acetic acid extraction experiment involved determining the distribution coefficient for the extraction of acetic acid in aqueous solution with ethyl acetate. This experiment was previously used as an end-of-semester Lab I project several years ago.

Grading
The grading scheme for the Fall 2011 and Spring 2012 semesters is described in Table 4. As was explained previously, the differences in grading largely resulted from a reduction in the number of experiments with the addition of draft reports. Ten total reports were required in each semester. Reporting (both written and oral) accounted for 78% of the total grade in the Fall

semester and 68% in the Spring semester. Less tangible items such as the safety quiz, proper use of the lab notebook and professionalism were also part of the grade.

Table 4. Lab I Grading

Category	**Fall 2011**			**Spring 2012**		
	Number of reports	**Points**	**% of total**	**Number of reports**	**Points**	**% of total**
Safety quiz		50	3		50	2
Lab notebook		100	6		150	6
Professionalism		100	5		300	12
Pre-lab reports	10	250	14	5	450	18
Draft reports	0	0	0	5	500	20
Memo reports	3	300	17	2	300	12
Short form reports	3	450	25	3	450	18
Group reports	4	400	22	0*	0	0
Presentations	2	150	8	2	300	12
Semester Total		**1800**			**2500**	

*Group reports were assigned in the Spring semester, but are included in the memo and short form categories.

Course Implementation Results

The following paragraphs present results from three of the inquiry-based experiments, along with student feedback from the Fall 2011 and Spring 2012 semesters.

Membrane Recovery of BSA Protein

The overall purpose of the membrane experiment was to test the effectiveness of 50 kDa and 100 kDa cutoff membranes in retaining BSA protein (measured at 66.5 kDa). One group prepared a 1 mg/mL solution of protein, and then pumped the solution through the membranes while collecting permeate and retentate. A photograph of the experimental apparatus, which was supplied to each student group, is shown in Figure 3. BSA was measured in the permeate and retentate for each run using a BCA Protein Quantification Kit. To compare the membranes, the rejection coefficients were calculated using the equation

$$R = \frac{C_f - C_p}{C_f} \qquad (1)$$

where C_f = concentration of BSA in the feed stream
C_p = concentration of BSA in the permeate stream

Thus, the rejection coefficient is a measure of the fraction of the BSA retained by the membrane. Similarly, the sieving coefficients for each membrane were calculated by the equation

$$S = \frac{C_p}{C_f} \qquad (2)$$

The sieving coefficient is a measure of the fraction of the BSA that passes through the membrane and ends up in the permeate.

Figure 3. Photograph of Experimental Apparatus for BSA Protein Recovery

Table 5 presents the rejection coefficient and sieving coefficient for each membrane. The rejection coefficient was higher for the 50 kDa membrane, which meant that more BSA was retained by the 50 kDa membrane. The sieving coefficient was higher for the 100 kDa membrane, which meant that more BSA flowed through the 100 kDa membrane. Because of the size of the BSA protein (66.5 kDa), it was expected that all of the BSA would filter through the 100 kDa membrane. This did not occur because BSA exists as both a monomer and a dimer.

Table 5. Calculated Results for BSA Recovery

Membrane, kDa	Rejection Coefficient	Sieving Coefficient
50	0.259	0.741
100	0.155	0.845

Gas Chromatography (GC) with Internal Standard

The objective of the GC experiment with internal standard was to improve the analysis of ethanol/water using a flame ionization detector by selecting an internal standard to improve (normalize) the linearity of the calibration. After testing a number of alcohols, one group selected methanol as the internal standard because of its short retention time and strong response by the detector. After selecting the internal standard, the group prepared a number of aqueous solutions containing methanol and ethanol. The standard curves with and without normalization are shown in Figure 4. Because of the presence of the internal standard, the normalized curve had less scatter. As a final task, the normalized curve was used to find the concentration of ethanol in an unknown aqueous solution.

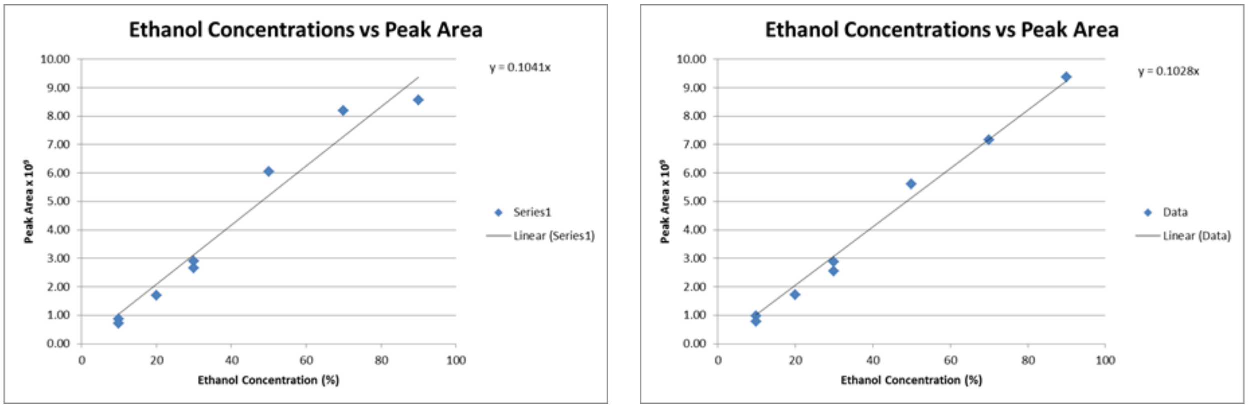

Figure 4. GC Calibration Curves for Ethanol without (left) and with (right) Normalization

Student Feedback

Student feedback on the use of inquiry-based experiments was solicited at the conclusion of both semesters. The comments were generally positive in the Fall semester, but some frustration was expressed with the mechanics of the inquiry-based process. Less frustration was seen in the Spring semester with the addition of draft reports, and the students generally noted that the labs were challenging, interesting and useful.

Comments from Fall 2011:

- The protein quantification assay was very interesting, and may be a technique that I use in the future.
- Overall, I thought most of the experiments were interesting and useful.
- It was really difficult to understand what was expected on each report. I wish we actually learned more about the experiments we were performing instead of being expected to look everything up on our own.
- I understand that we are supposed to design the experiments ourselves but, if we would have had more help, it would have avoided mistakes.
- A little more time could have been spent discussing what exactly was expected in the lab layout, pre-labs and lab reports.
- I think the level of difficulty was just right to make sure that the amount learned in this course matched evenly with the true appeal of the subject, which is what enhances the overall knowledge retained in a course.
- The class was helpful and improved my writing, teamwork and problem solving skills. However, I feel as if some of the experiments could have been substituted with ones that require more experimental design and problem solving.
- This class was excellent and I enjoyed it quite a bit.
- Slightly more clarification and feedback on lab reports would be ideal.
- My favorite experiment was the sugar extraction because I felt it was the most like an actual engineering problem.

Comments from Spring 2012:

- I loved that we were given a goal and that our assignment was to find a way to reach that goal effectively.

- I thought the class was overwhelming at first, but then things worked out for the better.
- This course helped me improve my report writing skills for future courses.

Conclusion

Inquiry-based laboratory experiments were introduced into the introductory laboratory class in Chemical Engineering at the University of Arkansas to spark interest in chemical engineering problem solving. Although many of the students were initially frustrated by the process, inquiry-based labs are now accepted by the students as challenging, useful and interesting assignments. Several lessons were learned about what works well and what does not work well in the inquiry-based process in the laboratory:

- The number of experiments in an inquiry-based laboratory must necessarily be less than in a traditionally laboratory in order to give students time to create and think. Widespread technical coverage is thus sacrificed in favor of problem solving to spark student interest.
- Pre-lab reporting is essential in keeping young students on-track and in developing quality learning experiences.
- Even though draft reports require more instructor grading time, submission of these interim reports improves report quality.
- A major challenge with the inquiry-based approach is the need to continually develop new ideas for experiments so that the experience remains fresh for the students (and the instructor).

The inquiry-based program will continue into the Fall 2013 semester with a new group of experiments.

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Biographical Information

SHANNON L. SERVOSS
Dr. Servoss currently serves as Assistant Professor of Chemical Engineering and holder of the Ralph E. Martin Professorship in Chemical Engineering at the University of Arkansas. Her research interests include biomimetics, biomaterials and protein microarrays.

EDGAR C. CLAUSEN
Dr. Clausen currently serves as Professor, Associate Department Head and the Ray C. Adam Endowed Chair in Chemical Engineering at the University of Arkansas. His research interests include bioprocess engineering, the production of energy and chemicals from biomass and waste, and enhancement of the K-12 educational experience. Professor Clausen is a registered professional engineer in the state of Arkansas.

PRE-LAB REPORT

Title: Gas Chromatography

Objective: Determine an internal standard for ethanol and analyze the percentage by volume of ethanol in an unknown ethanol sample by gas chromatography.

Equipment:

- Gas Chromatography Instrument
- Injection syringe
- Ethanol
- Methanol
- Isopropanol
- n- Butanol
- Acetone
- Distilled Water
- General lab supplies
- Unknown ethanol sample
- 5-flasks

Procedure:

<u>Part A (week 1):</u>

1. Take 10 alternating draws and purges of the sample out and into the syringe.
2. Take 1 slow draw of at least 5 µl of ethanol. Make sure that there is no air in the sample.
3. Slowly discharge ethanol until 0.5 µl is left in syringe.
4. Wipe the needle to remove any excess sample off the outside of the needle.
5. Slowly draw plunger back drawing air into the syringe so that the 0.5 µl of ethanol is at the 2-3 µl mark on the syringe.
6. Click the Green arrow on the computer screen and enter ID information and wait. After, and only after, the computer registered the run and says "start" move to next step.
7. Insert the syringe needle completely into the injection port keeping your forefinger on the plunger head the entire time. Quickly drive the plunger all the way down and abruptly remove the syringe needle from the port.
8. Click on start on the computer screen immediately after injection.
9. Record the retention time, the height of the peak of the sample and the column temperature.
10. Repeat steps 1 - 9 for methanol, isopropanol, n-butanol, and acetone. Run all of the samples 3 times. Make sure your column temperature stays constant.
11. After you have the peaks for all your samples you compare them to determine a good internal standard for ethanol at the tested column temperature. An appropriate internal

standard would be one which has a peak that is close to the one of ethanol but doesn't go higher than ethanol's peak.

12. To be sure the internal standard works, mix 0.5 µl of the internal standard with 0.5µl ethanol to determine that the internal standard is combining well with ethanol.

Part B (week 2):

1. Obtain the ethanol standard solutions of 10%, 30%, 50%, 70%, and 90% by volume. Mix 0.5 mL of each standard with 0.5 mL of internal standard.
2. Process each mixture of ethanol standard and internal standard by following steps 1-9 from part A. Use the computer to determine the peak area for the ethanol and peak area for the internal standard on the chromatogram by integration. There should be a total of five chromatograms analyzed.
3. Obtain the unknown ethanol sample and mix 0.5 mL of this sample with 0.5 mL of internal standard.
4. Process this mixture by following steps 1.9 from part A. Use the computer to determine the peak area for the ethanol and the peak area for the internal standard on the chromatogram by integration.
5. Use Microsoft Excel to plot Ethanol Peak Height/Internal Standard Peak Height vs. Volume % Ethanol. This plot should only include the standard ethanol solutions tested.
6. Use the curve to find a best-fit line for the data. Find the equation for this best fit line using Excel.
7. Take the ethanol peak area for the unknown ethanol solution and divide it by the peak area for the internal standard. Use this value as the y value in the best fit equation and solve for the x value to find the % volume of ethanol for the unknown ethanol solution.

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