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A NOVEL LABORATORY COURSE ON ADVANCED CHEMICAL EXPERIMENTS

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The chemical engineering curriculum in the United States has trained generations of technical experts who have successfully optimized chemical processes and products once they entered the chemical industry. The U.S. chemical industry, however, has entered a critical stage in which it must be able to create new and differentiated value through technical innovations that are essential for long-term survival. This innovation process will require new skills that go far beyond the traditional expertise for the optimization of tasks possessed by young chemical engineers. The innovators must be able to identify new opportunities, explore the boundaries of technology, evaluate critical issues, develop and implement technologies, and communicate effectively with scientists and engineers from other disciplines. Therefore, one of the most important educational tasks of a modern university, in combination with a strong theoretical foundation, is to challenge students in laboratory courses to think, explore, hypothesize, plan, solve, and evaluate.

The typical sequence of laboratory skills development stops short of introducing young engineers to the most critical aspects of experimental work. Chemical engineers usually begin developing their laboratory skills in chemistry courses, where experiments are closely managed. At this early stage in their development, students follow detailed instructions and learn basic principles by observing the results. In the undergraduate engineering laboratory course (the "unit operations lab"), students have more freedom in experimental design but still have well-defined objectives and manipulate equipment someone else has set up.

It is rare, however, for undergraduate students to be taught how to create new experiments. It is also rare for undergraduate students, and hence beginning graduate students, to have an appreciation for the care, planning, design, and testing required to produce equipment that will give reliable and useful results. Even such simple issues as leak testing or adapting analytical devices to new tasks are outside most students' experience. Even more important is an absence of opportunities to learn how the lessons learned from the failure of an approach can be fed back into the empirical process to seed the finally successful idea. All these skills require more creative freedom than is usually allowed in a well-structured laboratory course. In the novel laboratory teaching approach described here, we try to provide students with a learning environment that allows them to develop advanced experimental skills that are necessary for success in research and development environments.

LABORATORY EQUIPMENT

A true opportunity for students to discover and develop experimental skills is expensive in both hardware and the recurring costs associated with providing the freedom to make mistakes. This expense is often a strong deterrent to the development of the laboratory content of chemical engineering curricula. We have been fortunate to have the interest and

commitment of the Dow Chemical Company in this educational investment in future experimentalists. Dow's financial support has made possible this version of the laboratory and the educational opportunities it affords. We note, however, that the concept of combining the research in chemical engineering at the host institution with the experimental expertise of interested faculty and equipment dedicated to support students is a portable one and can provide a vehicle for exporting this approach to laboratory instruction virtually anywhere. At Purdue, equipment has been chosen to allow us to design projects that involve a variety of experimental techniques in which new apparatus can be created. The projects in the early stage of this course are planned so that future generations of students will benefit from the new instrumentation. They may then, for example, modify the existing equipment for their purposes.

The initial focus of the lab development project has been in adsorption, catalysis, and reaction engineering. Instrumentation available in the teaching laboratory includes

- *A Fourier transform infrared spectrometer for molecular identification of adsorbed or gas phase species*
- *A mass spectrometer for chemical analysis of atmospheric streams from reactors or adsorption systems*
- *A scanning force microscope for topographical analysis at the nanometer scale*
- *A surface area and pore structure apparatus for analysis of active porous materials such as catalysts*
- *An atomic adsorption spectrophotometer for elemental analysis of solids*
- *A gas-chromatograph for chemical reaction analysis*

STUDENT BODY, COURSE STRUCTURE, AND STUDENT EVALUATION

Experiments for this course were piloted with two separate groups of honors students in 1994 and 1995. In the spring semester of 1997, the course was offered the second time at Purdue. Over twenty students were interested in this novel course approach, but due to space limitations, only twelve could be admitted to the course. The student body was heterogeneous, consisting of three chemical engineering juniors, one chemical engineering senior, one chemistry senior, four chemical engineering graduate students (first and second year), two chemistry graduate students (first and third year), and one materials engineering graduate student (first year). In contrast to the previous years, the undergraduate students were not honors students, but experimentally interested students with various grade point averages.

The students were divided into three groups of four and each group was given an open-ended project. The project descriptions provided the groups with an overall project objective and a well-defined starting point, but required them to develop and pursue their own research ideas. When the groups were organized, care was taken to obtain an equal distribution of students of different levels, departments, and gender for each group, simulating an industrial research environment. For most students, this course was their first experience in a group where graduate and undergraduate level students from different backgrounds had to work together to achieve a common goal. In the beginning, the

students had to assign the roles of leader, experimentalist, and analyst among the group members. The diversity of the groups clearly added a learning dimension. The course was also successful with undergraduate chemical engineering students only, however. While those groups were honors students, it is our belief that a genuine interest in the hands-on aspects of chemical engineering is more important than the students' grade point averages.

The total time allotted for the course was two three-hour blocks per week. A combination of approximately 80% laboratory time and 20% lecture time was chosen to give the students enough time to gain hands-on experience with designing, building, and testing effective experimental equipment and adapting modern analytical instrumentation for chemical engineering measurements. A conference room was chosen over a classic classroom setting for lectures and for student presentations to facilitate discussions between the students.

The lectures were concentrated in the first six weeks of the semester (two hours of lecture per week) and covered a variety of topics in instrumentation, molecular-level measurements, and computer data acquisition. The diverse backgrounds of the students required implementation of a teaching philosophy that started with basics and built progressively and at a reasonably fast pace to a deeper and more applied level. Lectures and demonstrations were also given in scientific writing, literature search, and oral-presentation skills. A summary of topics covered in the lectures can be found in Table 1.

TABLE 1

Lecture Topics

- How to perform a successful literature search
- Data acquisition and programming of virtual instrumentation
- Vacuum technology
- Flanges and fittings
- Scientific writing
- Scientific oral presentations
- Molecular vibration
- Vibrational spectroscopy—experimental aspects
- Heterogeneous catalysis—an overview
- Adsorption and desorption
- Thermal desorption spectroscopy
- Scanning probe microscopy

There were no quizzes, tests, or final exams for this course. Student evaluation was based on three major factors:

- *Performance in the laboratory (motivation, ideas, group dynamics, results)*
- *Oral presentations (one 15-minute talk for each group member during the semester as well as so-called 5-minute updates every Tuesday morning, showing progress and drawbacks encountered during the last week and encouraging student*

- discussions)*
- *Written reports (each group wrote a total of three reports about their progress during the semester)*

While the evaluation for the first two points was on an individual basis, all group members received the same grade for their reports, motivating them to work closely together. The students were asked to turn in their individual contributions to the papers, not for grading purposes but to help them improve their writing skills and to provide them with individual feedback if necessary.

PROJECTS

One project was given to each of the three student groups. These projects were designed in advance by the instructors around space and instrument-use limitations. Due to the open-ended nature of the projects, it was not possible to plan the whole course in advance—which at a later stage in the semester caused some logistical problems when more than one group wanted to use the same instruments (this problem was solved by offering extra laboratory hours). Due to the open-ended nature of the research projects, it proved to be important to closely monitor students' progress, without imposing the instructors' opinion on their approach. Frequent, open discussions with the group were by far the most effective way to guide their research.

Project 1 involved the synthesis and characterization of the mesoporous materials MCM-41 and ZSM5. The students were motivated to study the effects of process variables on the zeolite properties, such as pore size and acidity. Available instrumentation for this project was a physisorption apparatus, atomic absorption, X-ray diffraction, mass spectroscopy, and nuclear magnetic resonance. This project had no major design component and therefore was able to make use of a wider variety of techniques to characterize the chosen materials.

Project 2 involved the design and construction of an IR transmission cell to perform an IR spectroscopy study on supported catalysts. The students built the reaction cell around a 2 3/4-inch Conflat cross, which was provided to them as a starting point. The students then had to design a sample holder with heaters and thermocouples, a simple gas inlet system with flowmeters, and a gas analysis system using the given mass spectrometer setup. After several attempts and many iterations with the instructors, the final design was built by the chemistry machine shop. During this process, the students learned that it is important to pay attention even to minor details, such as the material used for the screws or how to attach CaF_2 windows to a metal flange. The reaction chosen by the instructor was CO oxidation, and the students opted for a SiO_2 -supported Pt catalyst, which they prepared and characterized with atomic absorption, scanning electron microscopy, and chemisorption. The C-O stretching vibration of CO adsorbed on the Pt particles was observed during adsorption, desorption, and reaction conditions.

Project 3 will be discussed in detail. It included design and construction of an attenuated total reflection (ATR) IR cell to perform liquid-phase IR measurements. The group was

composed of a third-year chemistry graduate student, a first-year chemical engineering graduate student, and two chemical engineering juniors. The objectives of the project were to teach the students how to design and build an optical device from scratch and how to perform IR vibrational spectroscopy.

The students were asked to design and build an ATR cell out of commercially available optical elements. ATR spectroscopy was chosen because it is an effective method for liquid IR sampling, sample liquids are easy to handle, and the overall cell design can be relatively simple. Design constraints given to the students in advance were that the cell must fit into the sample compartment of the available Nicolet 550 FTIR spectrometer. In addition, the instructors had ordered a 450 trapezoidal zinc selenide (ZnSe) crystal of dimensions 50x20x2 mm as the ATR element to avoid major time delays for the students. The ATR cell had to be easily removable from the sample compartment, since Project 2 also made use of the ETIR spectrometer during class periods. A starting budget of \$1,000 was given to the group.

The secondary objective of this project included the implementation of a temperature controller and a mass spectrometer data acquisition module in LAB VIEW®. This addition to the main project was chosen to bridge time gaps while the group was waiting for parts ordered or being machined. Figure 1 shows the original project description given to the students during the first class period.

In the beginning, the students felt this project assignment would be almost impossible to accomplish. None of the students in the group had any research-based experience with IR vibrational spectroscopy or design of optical components. The students set out to find information about IR spectroscopy in general and publications about other ATR cell designs. Their first thought was to find other designs in the scientific literature or manufacturer's catalogs and to simply "copy" one of them. They soon realized that the available information in the literature was sparse and the majority of the descriptions were not useful in designing their own cell.

During this first week, the instructor (who had done a literature research prior to the class) was available for discussions when students needed him, but he did not interfere or direct the information-gathering process. The students realized that they had to start their own thinking process, which required a better understanding of the underlying physical principles.

The group started to postulate design concepts. During this phase, it was important for the instructor to give suggestions while preserving the students' freedom to develop and pursue their own ideas. For example, the students soon found out that it is necessary to focus the IR beam on the entrance slit of the ATR crystal and immediately associated "focus" with "lens." Therefore, their first idea was to use silica or plastic lenses in their design. At this point, the instructor had to alert the students to the fact that lenses for IR wavelengths have to be made out of special materials in order to be transparent.

After the students had been encouraged to look at other IR beam designs and at the FTIR spectrometer in the lab, they came to the conclusion that gold-coated focusing mirrors would be the way to go. Further problems that had to be solved were the construction of the liquid sample holder and the holders for the optical components. Several design iterations and many discussions followed. After three weeks, the students had decided on their final design, which allowed them to stay within the budget. This group finished their design and construction of an ATR cell (shown in Figure 2), the total cost of which was about 30% of commercially available cells!

Students and instructors agreed that the final term paper should be written in the form of a scientific paper, which could be submitted to a scientific journal. After the cell design was finished and the cell was extensively tested with IR grade fluids, the students had eight lab periods left in the semester to perform experiments, which they planned on their own. For most of them, this high degree of freedom was unique compared with previous experiences in other laboratory classes. The group chose to compare various grades of gasoline, which they collected from local gas stations.

We think that the possibility of performing experiments with the equipment they designed and built is important in making this course a satisfying experience for students. Therefore, progress of the students should be monitored closely by the instructors to ensure that design and construction are finished with at least four weeks left in the semester.

EDUCATIONAL ASPECTS

During the semester, the students were closely monitored by an educational researcher from the Division of Chemical Education in the Department of Chemistry at Purdue University. The project evaluation was done using Action Research as the methodology. [1,2] Qualitative Action Research is an informal, formative, interpretive, and experiential model of inquiry in which everyone involved in the study is an active, knowing participant. [3,4] The knowledge sought was “what worked and why,” and “what needs to be changed for the next class?” The educational researcher spent every moment he could with the students and the instructors during the scheduled class time. Data were collected from a variety of sources, including oral and written field notes, video tapes of the presentations and lectures, group lab reports, student written evaluations, student interviews, and conversations with the instructor. Oral data were transcribed for analysis, and inductive analysis [5,6] was used to find emergent patterns in the data. Inductive analysis is a method used in qualitative research that allows meaningful categories and themes to develop from the raw data, such as transcriptions and field notes. Reading, categorizing, and re-categorizing data patterns through the whole evaluation period over time allowed the students’ and instructor’s words, actions, and interests to become clearly organized into knowledge claims that could inform the instructional practice of the course. Four emergent knowledge claims at the writing of this paper are:

- 1. Groups that are mixed by academic experience can work well and benefit everyone.*

The factor that added the highest degree of complexity to the course was the mixture of

undergraduate students and graduate students in each group. The groups were allowed to negotiate for themselves who would be the group leader. A graduate student emerged as the group leader in each group, but the interpretation of what it meant to be a leader varied in each group. Due to the long-term nature of the projects, everyone in each group needed and gained a basic understanding of every aspect of their group's project. The undergraduates attributed much of their self-improvement in their technical writing skills and experimentation skills from working with graduate students as peers in their groups. Due to the collaborative nature of the projects, full participation in every aspect of the group's project was required of each group member, and for the most part undergraduate and graduate students took over equal research responsibilities. One graduate student commented, "It would have been impossible for one of us to have done all the work and come up with all the ideas. I have my own research to do. The undergraduates worked really well with us." Most of the cooperative learning literature is devoted to groups of the same age, grade, or course level. [7] In this course, not only was there a horizontal relationship among the students due to the different areas of study, but also there was a perceived vertical relationship due to academic experience status.

2. The traditional roles of leader, experimentalist, and analyst were initially assigned, but the borderlines between these categories were often crossed.

In order for the groups to function properly, every member of each group needed to develop a basic understanding of every aspect of the group's project. Due to the large nature of the projects, no one person (including the instructor) could have made all of the content, design factor, and experimental design decisions for the group. Each member of the group was required to make two presentations during the semester, a task usually assigned to the group leader in traditional laboratory courses. Therefore, the members of each group became "experts" on a particular instrument, content, or experimental process. Each member of the group was involved in the experimentation and/or analysis of each aspect of the project. Although there was a graduate student in each group who served as a group leader, each described his/her role as more of a group-organizer/facilitator than as a leader who told everyone what to do.

3. The written reports and presentations afforded the students freedom to improve their own knowledge throughout the semester.

The groups were required to turn in three formal written reports during the semester. An exciting observation was made after the first reports were graded and handed back to the students: the students separated the graded reports into sections for each group member to work on; the comments and suggestions written on their reports were used to improve their existing experimental procedures and to improve their technical writing in subsequent papers. The researcher had never observed this in the traditional chemical engineering laboratory classrooms. The students, especially the undergraduates, gave great praise to the Tuesday-morning presentation time, which obviously helped them to improve their own presentation skills. But the students also said that this time helped them improve their "presentation listening/ comprehension" ability. By listening to all of the presentations from every group, the students were exposed to a greater knowledge base than if they had only

heard the presentations from their particular group. Many of the “Five Minute/One Overhead” talks were followed by 10-15 minutes of questions and discussion. Two undergraduates, however, felt intimidated due to the content level of questions and discussion. Their major complaint was that the questions and discussion were sometimes based on knowledge that was neither based on nor developed in the course itself. The remainder of the undergraduates also felt a similar degree of intimidation, but realized that research is not exactly like a classroom; one undergraduate noted, “You have to utilize knowledge from wherever you can.”

The setting of the Tuesday-morning talks was also important. The setting was not a classroom, but a conference room. Instead of sitting in desks, all facing the front, everyone sat in comfortable chairs arranged in a “U.” This provided an atmosphere that was more like a community of researchers rather than a classroom of individuals.

4. The students used their experimental freedom for taking ownership and responsibility for their own knowledge and skills.

In each of the interviews, the students praised the freedom that the design of this course allowed them to have, as compared with traditional laboratory courses they had experienced. This course allowed them to “set their own standards,” “set and achieve their own goals,” and “make mistakes, and change things to fix them.” The students described other laboratory courses as “being told what to do” in order to “give them [the instructors] what they wanted for the points.” Other representative comments can be found in Table 3.

TABLE 3
Student Comments

Assertion 1

- “Having graduate and undergraduate students in the same group was a definite benefit. The diversity was something that we (undergraduate students) never would have experienced otherwise. Working with them allowed us the chance to benefit from their skills. Now I know that there are real people behind those doors in the building. I know more now what they do and how they approach problems.”
- “The course could be divided into undergraduate and graduate students, with the undergraduates working in a more controlled environment. The course went fine, but it would be interesting to see what the graduate students could do if they were by themselves.”

Assertion 2

- “We tried to keep the roles through the first paper, but they just didn’t work out, except for the group leader. That was something that stayed the same.”
- “I wrote up a complete section of the second paper on TPD from the background and theory to our experiments. I was an analyst at the first, but I guess we all kind of... took care of our own areas. The roles didn’t do too much for us.”

- “I have learned lots of lab techniques and benefitted from working with others who are not in my major, learned to see things from a different point of view. As well I have learned things that I hadn’t expected like how to use what I know and combine it with what I don’t know, and what my group members know to solve a problem.”
- “I have increased my problem solving abilities. My analytical thinking has changed as well as how I look at things. See the forest not the trees.”
- “I think one of the best qualities of this course was the flexibility given to the students on the projects. To actually plan, design, construct, and implement a tool is very rewarding. The very fact that the design begins from scratch forces us to understand every part of the process.

Assertion 3

- “The presentations were good. We got to see what other people were doing instead of just hearing about our own project. The other groups were doing some things that related to our experiments.”
- “The discussions and lectures were okay. Sometimes they went a little long and cut into our lab time. That made it difficult some days when you only had a certain number of days on the equipment.”
- “My technical writing has greatly improved. Working with the graduate student helped, and I got to be sort of an ‘expert’ on a process and a piece of equipment.”

Assertion 4

- “In other unit-ops labs they [the instructors] set the standards, or they have been set since time began. Here we set our own standards and deal with problems that come up. It’s up to us to fix them.”
- “I think the freedom and responsibility we had in the course is one of the greatest parts of it. We were able to do what we needed to do to solve the problems to reach our goals.”

CONCLUDING REMARKS

We have reported a novel approach to laboratory teaching for undergraduate and graduate students providing degrees of research freedom atypical for chemical engineering laboratory instruction. Judging from our experience with this course and the student feedback, we can conclude that the approach provides valuable training for every student interested in learning more about experimental work. We expect the course to become an elective course in Purdue’s chemical engineering curriculum. The concept is portable to other universities. The main ingredient is a cluster of interested and experimentally oriented faculty willing to design course projects and seek the optimum level of monitoring to maximize student success in independent work. The scope of projects will, of course, depend on equipment available for student use. As with any new course, the faculty time commitment is largest the first time through when all the projects are new and untested.

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Figure 1: Information given to the students of Group 3 at the beginning of the course.

Design of ATR Cell and LAB VIEW Programming

Keywords:

- *Fourier transform infrared spectroscopy*
- *Attenuated total internal reflectance*
- *Infrared optics*
- *IR sampling of liquids*
- *Virtual instrumentation*
- *LABVIEW instrument interface programming*
- *Mass spectroscopy*

This project is divided into two separate parts, which in the beginning will have to be performed simultaneously. First of all, we are interested in the control of the quality of liquids. For that purpose, we want to use Fourier transform IR vibrational spectroscopy to obtain vibrational spectra of liquids. We will use attenuated total internal reflectance spectroscopy (ATR), a powerful and versatile tool for IR liquid sampling. The group has to design and realize a small ATR cell including the IR optics, which will fit into the sample compartment of the FTIR spectrometer available in the Dow lab. Since more than one group will use the FTIR spectrometer during the semester, the ATR cell has to be portable, and easy installation and removal are important design criteria. The instructor will provide

an ATR crystal for the group as a starting point for developing the ATR cell. Once the cell is completed and tested, you will perform experiments with several liquid samples.

The second task of the project is to learn the basics of the programming system LAB VIEW and to program a control interface for the LEYBOLD mass spectrometer (manuals and basic software tools will be provided). This program will also manage the heating controller for thermal desorption spectroscopy. Both instruments communicate with a Power MAC via a serial port interface. Groups #1 and #2 will depend on the timely “delivery” of this program package, since they will use the interface to take valuable thermal desorption data. Therefore, coordination of the time schedules between all groups is necessary and should be performed by the leaders of each group.