ChE *laboratory*

A HOLISTIC UNIT OPERATIONS LABORATORY

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n the coming century, chemical engineers will face many new challenges. The needs of the chemical industry are progressively moving from process-oriented engineering to product-based engineering, and the new environment requires that chemical engineers address a broader body of knowledge and collaborate with other specialists.^[1] Hence, industry expects to hire graduates capable of applying their understanding without further training, of finding creative solutions, and of communicating the outcomes. Technical competence is no longer sufficient if it is not combined with non-technical abilities such as problem solving, management, leadership, teamwork, decision making, and ethical responsibility.^[2] This has been recognized by the Accreditation Board for Engineering and Technology (ABET)^[3] which has specified that engineers should demonstrate not only a broad scientific base but also a set of skills linked to social capabilities.

As a result, the paradigm of engineering is shifting from *hard* engineering to *soft* engineering, although technical aspects are still the core. This shift involves dealing with issues such as more efficient teaching methodologies, different learning styles, new learning materials, and the revision of course syllabi, which must evolve to fit the new paradigm of education by switching the emphasis from instructor-based teaching to student-centered learning.^[4]

Since real problems do not recognize disciplinary boundaries, the unit operations laboratory could easily be a suitable place for a holistic approach to chemical engineering.^[5-14] In addition to the classical understanding of unit operations, a professionally oriented chemical engineering laboratory could provide creative and critical thinking, the ability to design experiments, and the capacity to analyze data and draw reasonable conclusions. Simultaneously, the laboratory should incorporate aspects that are necessary to achieve a global education of the chemical engineer, such as safety and environmental concerns, commercial relevance, troubleshooting, and design of procedures. A similar laboratory with structured experiments was recently proposed.^[15]

In response to these expectations, the School of Chemical Engineering of the Rovira i Virgili University (URV) has a laboratory that addresses soft skills and requires rigorous understanding of the basic operations. The course is based on a constructivist approach, and students learn by forming their own interpretation of open-ended experiments. The instructor's role is to guide the students and prevent misconceptions, rather than to transmit formal knowledge to passive students.

COURSE STRUCTURE

The chemical engineering degree at URV takes five years to complete. Each course is divided into two fifteen-week semesters. The courses are run using a credit system in which one credit is equivalent to ten hours of lectures. The complete degree requires students to obtain 405 credits.

The unit operations laboratory is a nine-credit course given during the second semester of the third year. By this time the students have taken the basic subjects, several fundamental

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The laboratory . . . simulates a professional environment in which students must design experimental procedures to meet customers' demands. [It]also addresses other no-lessimportant topics such as safety, legal regulations, economy, troubleshooting, and the environment.

laboratories, and a few other major subjects, so they can interpret the basic concepts underlying unit operations. The course is devoted to classical unit operations (except heat transfer) and includes water and wastewater treatment.

The planning and execution of the experimental work and the subsequent interpretation and presentation of the results is the essence of the course. The students (approximately 60 per year) are organized into teams, usually of three to four

members. Random teams are preferable since this promotes a mixture of learning styles and the development of interpersonal skills. The instructional objectives are

- To test real equipment, manage possible upsets, and solve operational troubles
- To design procedures for start-up, steady-state operation, and shutdown
- To identify key variables during normal operations
- Insights of unit operation Report editing Project management Select key variables Coordinator Leadership Planning experiments Confidence and Plan timing consistency of results Write the report Scheduling **Team working** Literature search Supervision Analyzer Calculations Practical aspects Error analysis Analytical methods Data collection Deal with hard and soft specifications

Figure 1. Team roles and organization in the laboratory.

- To search for, consult, and interpret technical documents
- To process data and check the mass and heat balances, physical properties, thermodynamics, transport phenomena, and chemical reaction
- To develop decision-making criteria depending on product specifications, environmental constraints, legal regulations, safety, and economic reasons
- To consider the importance of errors in the validation of the results obtained
- To formulate hypotheses and simplifications to facilitate the analysis and modeling of unit operations
- To optimize the operating conditions according to the experimental results
- To present effective oral and written results and conclusions

The laboratory course was devised around the following structure:

ter
They are supervised by one faculty member and one assistant lecturer
The experimental equipment in the laboratory is divided

The students have access to the laboratory for one three-

hour session, four days per week throughout the semes-

- The experimental equipment in the laboratory is divided into three different blocks
- Students spend four days completing the classical unit

operations experiments (distillation, absorption, liquidliquid extraction, and a set of reactors); three days for the water treatment modules (reverse osmosis and ion exchanger); one day for the wastewater treatment plants (flocculation-sedimentation, aerobicactivated sludge, and anaerobic fluidized bed). Each group must perform two experiments from the first block, one from the second, and the whole of the third block.

Thus, the maximum involvement of the students in experimental work is forty-two hours. They spend the rest of the time planning, analyzing results, and reporting.

The methodology is to imitate a professional environment in which decisions have to be taken, responsibilities assumed, mutual confidence experienced, and tasks programmed and distributed. The tasks are randomly assigned to each team. During the course, each team member must perform, at least once, the role of coordinator, operator, and analyzer (see Figure 1).

COURSE DEVELOPMENT

Once the problems are assigned, students are provided with a simple scheme of the equipment. In turn, we expect them to find all the relevant information needed to design and conduct the experiments and analyze the data obtained. Instruc-

Spring 2002

tors merely act as supervisors and assist teams if they fall into a dead-end situation or if potential safety risks are detected. As the laboratory slogan says, "Good judgment comes from experience. Experience comes from bad judgment."

The course progresses through a six-step procedure that must be satisfactorily completed:

- 1) Experiment preparation and preliminary report tutoring
- 2) Two sessions of experimental work (just one for block 3, with no possible extension)
- 3) Intermediate report and new planning tutoring
- 4) Two additional experimental sessions (only one for block 2)
- 5) Technical report
- 6) Oral presentation and question-and-answer session

This schedule enables better monitoring of the performance and evolution of students and also provides continuous feedback. The preliminary report must contain practical procedures (start-up, routines for steady-state operation, and shutdown protocols). After students have been tutored, the planning is accepted on the basis of the experimental aspects, time management, and analytical methods. Therefore, faculty efforts are especially important at the beginning of the course to prevent inaccurate procedures. As students become increasingly familiar with the methodology, instructors focus their attention by posing challenging questions that encourage creative thinking. Once the plan has been accepted, the team must reserve the equipment for two laboratory sessions. All experimental data, tasks, and incidents must be detailed in the laboratory notebook for reproducibility. The notebook is checked periodically and graded at the end of the course.

After the first part of the experimentation, each team must check the coherency of the data (mass and energy balance) and draw preliminary conclusions. At this point, a progress report must be written to compare the results with the model predictions and to discuss the goals reached. Frequently, partial results induce changes in the subsequent experimental plan and students are forced to make decision criteria for themselves.

At the end of the second period, the teams must deliver a technical report of each experiment. The final report is expected to contain all the valuable information needed to justify the conclusions in a concise and clear manner. In any case, students are asked to report the confidence interval of the results and to provide explanations for the behavior observed and any possible deviations from the theoretical models. Usually, the results and discussion are presented in the same section. Finally, the students must propose a solution in a few lines.

COURSE EVALUATION

The evaluation is mainly based on the oral presentation of *152*

the final report for each assignment. Each member of the team is required to give an oral presentation of one randomly chosen assignment. As all members of the team are fully accountable for all the assignments, they do not know in advance which one they have to present. After the presentation, the student is questioned about the statements (from the report and/or the presentation), the procedures and the conclusions, as well as any unclear parts of the discussion. Three faculty members judge the quality of the presentation and the overall knowledge of the problem.

Table 1 shows the grading scheme used to obtain the students' final qualification. As can be seen, the final grade depends on their knowledge of unit operations, their performance in the laboratory, and their personal skills. There is a good balance (45% versus 55%) between personal skills and collective skills, but an individual factor assigned to each student can increase or decrease the final grade by 10%. With this factor, we attempt to account for the greater or lesser involvement of a particular student in the group performance. This involvement is easily detected in the group's daily work. The examination at the end of each experiment permits students to learn from their own experience and mistakes. On average, 12% of the students fail at the first attempt and after additional work just 5% do not qualify. It should be pointed out that the pre-laboratory and intermediate reports are a crucial part of the learning procedure, so they are used basically to collect information about course dynamics and as a firsthand source of feedback.

LABORATORY EXPERIMENTS

Nine different experimental set-ups are currently available in the laboratory. The present structure of the laboratory and the assignments are the result of the evolution toward design of a zero-waste disposal laboratory. Table 2 shows a list of the problems addressed during a typical course. Usually, 3 to 4 new problems are posed every course (*i.e.*, the Murphree efficiency at the distillation column or the HETP at the ex-

TABLE 1 Grading Scheme	
Course Component	Grade Allocation
Individual Assignment	±10%
Procedures Methodology: planning, methods, group dynamics Reproducibility: experiment description in notebook	10% 10%
• Final Report Editing: structure, distribution, composition, numbering, v Readability: composition, grammar, conciseness, neatness Results: goodness of data, proper discussion, appropriate s	visual impact 5% 5% solution 20%
 Oral Presentation Editing: visual impact, relevant slides, content Performance: preparation, timing, tone, contact with audie Question session: fundamentals, experimental, results, eva 	5% ence 5% aluation 40%

traction column), so the assignment for each experimental set-up is not unique. All the problems are depicted as potential real-life cases that are not necessarily limited to the chemical process industry. This encourages multidisciplinarity and forces students to the knowledge of related areas such as environmental engineering.

The laboratory is based on open-ended problems. In contrast, in the classroom courses on unit operations, students solve close-ended problems in a tight environment. In the laboratory, however, the students face situations in which they

TABLE 2 Problem Statement for Each Experiment

Block 1

- Distillation A client asks for the best economic conditions for operating a continuous distillation column. The column is fed with an ethanol-water mixture containing 60% w/w of ethanol and a flowrate up to 25 L/h. The product composition must achieve 90% of the azeotropic. The reboiler and pre-heater power are 2 and 0.3 kW, respectively. The feed cost is \$0.5/L and the product is sold at \$2/L. The power cost is 0.1 \$/kWh.
- Absorption A customer has to decrease the ammonia content in a waste air stream (3.2 m³/h) from 15% v/v to less than 1%. An absorption tower is available where the ammonia could be absorbed with water. The availability of water is limited.
- 3. Liquid-Liquid Extraction An industrialist needs to purify 5 L/h of a binary mixture (45:55 w/w) containing MIBK and acetic acid (HAc). the MIBK recovered must retain a maximum of 2% HAc. Two technologies, liquid-liquid extraction with water and conventional distillation, have to be checked. Operating conditions must be optimized.
- 4. Reactors A small industry produces an aqueous stream contaminated with ethyl acetate (20 g/L). The acetate content must be reduced to 3 g/L or less before disposal. Hydrolysis using sodium hydroxide (NaOH) is proposed as treatment. Unlimited 0.2 mol/L NaOH solution is available at low cost. The customer asks for the reactor type and the operating conditions to comply with legislation.

Block 2

- 5. Reverse Osmosis In an isolated farm, a saline source is used (1.25 g_{NaCl} L) to purify water. The farm needs 100 L/h of water with conductivity up to 50 µS/cm. A second-hand reverse osmosis module is available without technical characteristics. Operating conditions must be set so that water is produced with minimum energy expenditure.
- 6. Ion Exchanger A laboratory received a new piece of equipment. The product specification is 150 μS/cm. The conductivity of the crude water, which is freely available, is close to 1 mS/cm. The water production cycle must attempt to maximize the pure water yield.

Block 3

- 7. Flocculation/Sedimentation The acidic effluent of a galvanic plant must be treated. The plant generates 5 m³/h of water with 1000 ppm of copper. A preliminary design and scale-up of the treatment plant must be made using the data collected from the 100 L/h physical-chemical treatment plant.
- 8. Sewage Treatment Plant The Mayor of a city on the Mediterranean coast (with a population of 100,000) is aware that the urban sewage is more refractory than expected and cannot be biologically treated. The sludge plant needs to be re-engineered, so preliminary scale-up from laboratory data (2 L/h) must be carried out.
- 9. Denitrification Plant A modern farmer has implemented a sophisticated hydroponics system, but the purged water (5 m³/day) does not comply with environmental law. Biological denitrification is proposed as treatment. Experimental data can be retrieved from a 0.1 L/h lab-scale equipment. Scale-up must be done.

encounter build-up equipment, but no precise step-by-step guidelines. Thus, they need to understand the principles of unit operations, since mathematical models are of no use for a rapid qualitative interpretation of how each variable influences the unit operation performance. For instance, the reboiler power or the top condenser duty are seldom set in distillation design. On the contrary, they are usually calculated using the reflux ratio required for a particular separation. It is noteworthy that the main disturbance for students is that the reboiler power is fixed in a *real* distillation column, so that its capacity and the reflux are constrained.

One of the characteristics of this laboratory is the minimum waste production, where students experience aspects covered by several elective subjects (i.e., environmental engineering). At the moment, waste production has been reduced almost completely at no significant additional cost. In fact, the only waste that cannot be treated in situ is the precipitate from the flocculation-sedimentation plant (copper hydroxide and calcium sulfate), which is sent to a qualified waste-treatment company. The other wastes are either reused or treated. For instance, the water-ethanol mixture used in distillation is reused throughout the course in the same equipment. Extraction requires more complex treatment. Refined methyl-isobutyl ketone (MIBK) is directly reused. After extraction, however, the acetic acid and water mixture still contains a certain amount of MIBK, which is recovered by distillation. Moreover, since distillation of the acetic acid-water mixture is difficult, the MIBK-free mixture is reused as a feed for the activated sludge plant. Notice that the principal environmental impact produced by the laboratory is due to the life-cycle impact of the electrical power used.

STUDENT PERFORMANCE AND FEEDBACK

When problems are assigned, students who are not familiar with problem-solving schemes often miss the point, and continuous assessment is required. Hence, instructors act more as counselors, redirecting students efforts, than as formal teachers of structured knowledge. Once students realize that there is no single solution or approach to each problem, they connect up the disparate pieces as a whole and develop their problem-solving skills exponentially.

The next critical point is when students prepare the intermediate report. They tend to make a list of results and do not estimate errors, check the robustness of the experiment, or explain deviations. Discrepancies and unexpected results are ways of identifying and correcting mistakes. The final reports are generally well-structured and carefully edited, and above all, the discussion of the results explains the dependence with the process variables and makes comparisons with model predictions.

At the end of the course, students are required to anonymously answer a feedback questionnaire. Table 3 summarizes the answers from the last course, which are similar to those of previous years. In general terms, the responses show that the course is well-accepted and they are particularly favorable for those statements about what students perceive they had learned/improved. This demonstrates that the educational objectives of the course were mostly attained.

It should be pointed out that students feel much more comfortable with this kind of teaching, although they demand more supervision. We should focus our efforts on providing students with training based on creative thinking, critical criteria, and problem-solving skills rather than providing them with a better understanding of unit operations, which they are capable of learning for themselves. Overall, the students' main objection was the amount of time they had to devote to the course, which was greater than the time scheduled. We should point out, however, that the extra time was spent on planning, data analysis, and reporting, since self-motivation often led students to go beyond the requirements of the course.

Many favorable comments have been received during the four years that the laboratory course has been running, which encourages us to continue pioneering the application of new educational methodologies in Spain. In the near future, this course will be part of an even more ambitious one-projectper-year strategy to stress holistic education in the chemical engineering undergraduate program.

CONCLUSIONS

We have designed a unit operations laboratory course with the main objective of providing chemical engineering undergraduate students with creative thinking skills, criteria for decision making, problem-solving and communication skills, and the capability to monitor and operate unit operations. The laboratory, therefore, simulates a professional environment in which students must design experimental procedures to meet the customers' demands. The course also addresses other no-less-important topics such as safety, legal regulations, economy, troubleshooting, and the environment.

Faculty members act as mere advisors, so students are not subjected to passive teaching. Student skills are developed through open-ended problems and by posing Socratic questions that enhance critical thinking. Obviously, we do not expect students to magically develop their entire individual potential within this laboratory, but as the course advances, most of the students become capable of designing experiments, analyzing results, and suggesting solutions. Simultaneously, they improve their self-confidence and learn to make attractive presentations. Faculty members must provide motivation when students fail and continuous assessment is needed if students are to make headway. The laboratory procedure (preliminary report, two-day experiments, intermediate report, and two additional experimental days) forces students to adopt a very useful stop-and-go procedure.

The benefits of the course largely make up for the tremen-154

TABLE 3 Results of the Feedback Questionnaire

(Class Size, 60 students: Score, 0-strongly disagree, 5=strongly agree)

		Ave.	St.
	Question	L)ev.
1.	After the informative session I understood the course methodolog	y 3.8	1.0
2.	The laboratory schedule was well-programmed and coordinated	3.1	1.2
3.	The problems matched my academic background	3.6	1.0
4.	The laboratory exigency fitted my previous formation	3.5	1.1
5.	Facilities and infrastructure were suitable	3.1	1.1
6.	The duration of the course was appropriate	2.8	1.4
7.	In this course, I improved		
	A. My basic knowledge of the unit operations	4.0	1.2
	B. My management and organizational abilities	3.4	1.0
	C. My report-writing skills	3.1	1.1
	D. My oral-presentation skills	3.6	1.0
	E. My documents/information-search skills	3.4	1.0
8.	All holistic aspects were taken into account in the final grading	3.8	2.3
9.	The team performed reasonably well	4.7	1.9
10.	I prefer this stye of teaching to a pre-set lab methodology	3.4	1.5
11.	Instructors were always available	3.8	1.0
12.	Instructors made sure that the experimental objectives were clear	2.9	1.7
13.	Instructors supervised the team performance sufficiently	2.9	1.9

dous effort required. The driving force for all of us is the same as for the students—the excitement of learning by doing.

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