

# Unit Operations Laboratory Using Ill-Posed Problems\*

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*The unit operations laboratory is designed to engage students' interest, as it requires them to tackle practical assignments connected to prior knowledge. The course is organized around ill-defined open-ended problems. Students have to identify the problem, formulate hypotheses, search for information, conduct experiments, extract conclusions, and determine the best set of operating parameters to fulfil the objectives. The course requires extensive use of social skills (teamwork, decision-making, task management and oral and written communication). The 'stop and go' method of organization was found to be very helpful in re-directing experimental work and as a source of continuous feedback.*

## INTRODUCTION

GIVEN THE amazing advances in technology, and the decrease in the number of students enrolling for technical and engineering studies, teaching methodologies must switch emphasis from instructor-based teaching to student-centered learning [1], thus enhancing student interest and teaching effectiveness. Other objectives would also be achieved, such as stimulating reflection, motivating low achievers, facilitating distance learning and providing continuing education.

Laboratory experiments play a critical role in bridging the gap between theory and practice, and offer the opportunity to deal with real equipment. Since real problems do not recognize disciplinary boundaries, the Chemical Engineering degree's Unit Operations Laboratory is an appropriate environment for the problem-based learning (PBL) approach. Problems were carefully selected and designed to develop critical knowledge, problem-solving ability, communication skills, self-directed learning strategies and teamwork [2]. The process reproduces the systematic troubleshooting encountered in life and careers, where creative solutions have to be developed without further training.

## COURSE DESCRIPTION

### *Organizing the course*

The Unit Operations Laboratory is a 90-hour course given during the two semesters of the third year. By this time, students have taken the general subjects in basic and engineering sciences (mathematics, physics, chemistry, thermodynamics, transport phenomena, separation processes, reactors),

several introductory laboratories (chemistry and chemical engineering) and a few complementary topics (environmental science, statistics, programming). Consequently, they can interpret the basic concepts underlying the unit operations.

The students are organized in teams of three that meet up to three times each week for a three-hour period for the duration of the course. Each group has to perform four different activities (Table 1), randomly assigned. During the course, each team member must perform, at least once, the role of coordinator, operator and analyzer, respectively, as described in Fig. 1.

### *Syllabus*

The objectives of the course are:

- to solve experimental problems related to mass and heat balances, thermodynamics, transport phenomena and chemical reactions;
- to design procedures for start-up, steady state operation and shutdown, identify key variables for normal operation and manage possible upsets;
- to realistically plan the execution of tasks for a limited period, thus experiencing the power (and pitfalls) of collaborative work;
- to search, consult and interpret technical literature;
- to formulate hypotheses and simplifications to facilitate the analysis and modeling of unit operations;
- to optimize the operating conditions, based on experimental evidence, and consider the relevance of uncertainties in the results; and
- to develop decision criteria according to product specifications, environmental constraints, legal regulations, safety and economic criteria.

### *Role assignment*

In PBL, the traditional roles of teacher and student are significantly changed. The students increasingly assume responsibility, thus promoting

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Table 1. Problem statement for each experiment

**Distillation.** A client asks for the best economic conditions to operate a continuous distillation column. The column is fed with an ethanol–water mixture containing 60% wt of ethanol and a flowrate of up to  $25 \text{ L} \cdot \text{h}^{-1}$ . The product composition must achieve 90% of the azeotrope composition. The reboiler and pre-heater duty are 2 and  $0.3 \text{ kW}$ , respectively. The fed costs about  $0.5 \$ \cdot \text{L}^{-1}$  whereas the product is sold at  $2 \$ \cdot \text{L}^{-1}$ . The power cost is  $0.1 \$ \cdot \text{kw}^{-1} \cdot \text{h}^{-1}$ .

**Absorption.** A customer needs to decrease the ammoniac content from 15% v/v to up to 1%. An absorption tower is available, where the ammoniac could be absorbed with water, with a limited availability. The gas feed flowrate is  $3.2 \text{ m}^3 \cdot \text{h}^{-1}$ .

**Liquid-Liquid Extraction.** An industrial company wants  $5 \text{ L} \cdot \text{h}^{-1}$  of a binary mixture (45:55 w/w) containing methyl-isobutylketone (MIBK) and acetic acid (HAc) purified. The recovered MIBK must retain a maximum of 2% HAc. Liquid-liquid extraction using water and distillation have to be compared.

**Sewage treatment plant.** The mayor of a Mediterranean city (100,000 population) is aware that the urban sewage is more refractory to being biologically treated than expected. Re-engineering of the current plant is needed, so the mayor asks for a preliminary scale-up from the pilot plant data ( $2 \text{ L} \cdot \text{h}^{-1}$ ).

**Denitrification plant.** A farmer has implemented a sophisticated hydroponics system, but the resulting water does not meet the environmental law concerning nitrogen content. Biological denitrification is proposed as a treatment. Experimental data can be retrieved from  $0.1 \text{ L} \cdot \text{h}^{-1}$  laboratory equipment. Scale-up must be done to treat  $5 \text{ m}^3 \cdot \text{day}^{-1}$ .

a sense of empowerment and enhancing motivation. In turn, the role of faculty staff is to guide the students in order to prevent misconceptions, rather than to transmit formal knowledge to passive students. In the laboratory, students freely operate

the equipment without interference from the instructors unless potential safety risks are detected or the teams reach a stalemate. As the course advances, Socratic questions are posed to enhance critical thinking.

#### Laboratory procedure

The course consists of a six-step procedure that must be satisfactorily completed:

- **Experiment preparation and preliminary report tutoring.** The team is responsible for collecting background information and becoming familiar with the experimental apparatus. Team members must define start-up, steady-state operation and shut-down protocols.
- **Two sessions of experimental work.** Students select the variables to be tested, the samples to be withdrawn and the analytical techniques to be used, with an emphasis on time management.
- **Progress report and new planning tutoring.** The report contains the data and a comparison of the results with model predictions, and checks that the objectives are fulfilled.
- **Additional two sessions of experimental work.** Frequently, intermediate results necessitate changes in the planning.
- **Final report.** Teams process the data (mass and energy balance, confidence of the results) and draw conclusions.
- **Oral presentation.** This is followed by a question-and-answer session.

#### Course evaluation

The evaluation is mainly based on the oral presentation. The evaluation exhibits a good balance between individual (45%) and group marks (55%). It should be noted that the pre-



Fig. 1. Team roles and organization.

laboratory and intermediate reports are a crucial part of the learning procedure, and are used to collect information about course dynamics and feedback. Each objective is secured in a closed-loop strategy, whereby the results of the tutoring are used at the end of each experiment. Hence, the examination encourages students to learn from their own mistakes.

## LABORATORY EXPERIMENTS

### *Course development*

Nine assignments are offered (partially described in Table 1), all of which are related to different unit operations. Students are provided with a simple scheme of the equipment and a brief description of the main elements. All the problems are depicted as potential real-life cases that are not necessarily limited to the chemical processing industry, promoting closely connected areas that highlight the multidisciplinary nature of chemical engineering.

In the laboratory, we try to ensure that the students face situations with the laboratory equipment and follow clear objectives, but without detailed step-by-step guidelines [3–4]. This requires them to understand the principles of unit operations, since mathematical models are not always useful for a rapid qualitative interpretation of how each variable influences the unit performance.

When problems are assigned, the students often express shock and dismay ('I cannot believe it! He has just given a few lines that are supposed to state a problem!'). Some students try to reach the solution through the application of traditional models, but they soon become aware that working as individuals is not the correct way to fulfil the activity objectives and that teamwork is required.

The beginning of the course is a very intense time. Students often miss the point and can become discouraged if instructors do not assess them. The preliminary reports can be especially frustrating, as it is then that they realize that the message seems to be: 'we will set these variables, we will analyze these streams, we will measure these data and, if this does not work, we will start again'. First, the students must overcome the misconception that a unique solution and approach exists for each problem. The second belief that has to be challenged is that teachers and textbooks are the only authority for gaining knowledge. In addition, students have to realize that the interdependence of several areas requires them to consider different criteria. When students discover that they are able to connect disparate elements into a whole, their problem-solving skills are developed exponentially and students gain in confidence. Students also frequently pursue assumptions that need to be challenged, such as 'the equipment is not working properly' or 'we are obviously proceeding in the wrong way' or even 'we are doing our best but something is still

wrong'. Instructors need to explain that the real world does not always work like that and that discrepancies or unexpected results are one of the necessary clues to identifying and correcting errors. Some examples are given in the following.

### *Which substance elutes first?*

Students know that the gas chromatography column is filled with a non-polar component. We provide them with the calibration curve, but they have to infer the elution order for the components from their physical properties.

### *VLE: assumptions and validity*

In distillation, the feed stage is usually assessed by experiment. According to the vapor-liquid equilibrium of the mixture, the feed stage must be fixed to the lower column inlet, independently of the objectives. This case stresses the importance of preliminary work, as this can save valuable experiment time.

As we do not provide the theoretical number of stages, the first experiment is performed at total reflux to estimate this value. Students can check efficiency results by comparing with typical ranges. Most students assume that the McCabe-Thiele assumptions are fulfilled and perform the necessary calculations, but just a few of them check this first.

Students often operate with a number of preconceptions. For instance, the reboiler power supplied is seldom set in the distillation design. On the contrary, it is usually calculated according to the reflux ratio necessary to achieve a certain goal. It is interesting to note that the thing that most concerns the students is the fact that the reboiler power is set in build-up distillation units and, consequently, the feed flowrate and the feasible reflux ratio are limited within a certain range.

### *Conflicting absorption effects*

In the ammonia absorption tower, a parabolic-like temperature profile is experimentally found. This is because the top section is controlled by chemical absorption (exothermic), while the air humidification (endothermic) prevails at the bottom. To help low achievers and to check for misconceptions, the following question is put to the students: how does the profile change if the ambient air is affected by a rainy day?

### *Transport properties matter*

Students can easily perform the graphical procedure to calculate the minimum entrainer required in an extraction unit, but they have problems detecting the utility of the air pulse, because the density and solubility differences seem to be high enough. Students discover, by experiment, that, without the air pulse, separation is not achieved. In this way, the tendency to avoid the use of transport properties in design is overcome. In particular, the importance of viscosity and surface tension to the extraction process is reinforced.

*Shaken not stirred*

In flocculation, students tend to simplify the problem and focus only on the solubility. If the experimental and theoretical values are compared, it becomes clear that particle dragging occurs. Some groups check the phase diagram and prove that, for very high pH values, the solid is re-dissolved because a stable complex is formed. Thus, they discover that the pH operating value has to be maintained within a certain range. Another recurrent element is that students tend to vigorously agitate a solution that has an appreciable solid tart, thus trying to dissolve it, while only slight agitation is sufficient to achieve the desired aggregation.

*Misfunctioning instruments*

Students tend to trust that laboratory instruments are working properly. The pH-meter installed in the sewage treatment plant has not been working properly since 1998. When students check the measured value (11), they conclude that the equipment is not working in the optimum operating conditions (4–6). When we remind them about the logarithm scale, they realize that an alternative analytical method is required. As we do not provide them with any other method, they have to find their own creative solution (i.e. measure pH from pure water).

**CONCLUSIONS**

The Unit Operations Laboratory, organized through professional assignments, improves the students' ability to monitor unit operations [5]. Faculty members act as mere advisers and consultants, so students are not subjected to passive teaching. Undergraduate students are actively involved, which enhances understanding and improves retention, proficiency in problem-solving, self-directed learning, decision-making and team participation (cooperation rather than competition). Obviously, students do not magically develop their entire individual potential whilst attending this laboratory, but we expect (and see) some degree of progress.

The laboratory procedure (preliminary report, two-day experiment, intermediate report and two additional experiment days) forces students to undergo a very useful stop-and-go process.

According to the feedback questionnaire, the course generally scored high averages from the students [6]. It should be noted that students are very comfortable with this kind of teaching, although more effort is required in terms of supervision. Overall, the students' main objection was the excessive time demanded by the course, which they claimed was much more than scheduled.

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