An Overview of Portuguese Chemical Engineering Undergraduate Laboratories

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Abstract

The UNESCO's World Declaration on Higher Education for the Twenty-First Century, Vision and Action (1998) states in its paragraph "Innovative educational approaches: critical thinking and creativity": "[…] New pedagogical and didactical approaches should be accessible and promoted in order to facilitate the acquisition of skills, competencies and abilities for communication, creative and critical analysis, independent thinking and team work […]".

The importance of teaching laboratories for students to acquire competencies and abilities especially in creative and critical analysis and teamwork is recognized. On the other hand it is generally recognized in all the EU countries that undergraduate labs are generally badly equipped, badly taught, badly organized and high budget consumers. To make things worse, the traditional education labs consume lots of reactants, produce lots of environmentally aggressive effluents, consume lots of students' time and have a modest output.

Under this framework what should be changed? Would it be easy to do? Or are we condemned to live with bad labs and bad lab classes?

Isolated efforts of laboratory professors and head of departments have been tried within each Portuguese University. Now it's time to involve all partners, head of departments, course directors, laboratory professors and students, of all schools, and make an effort to conquer a quantitative improvement of the undergraduate laboratories and dynamize an inter-school collaboration. The first step in this direction will be done, precisely, at this congress.

Five experiences on main five Portuguese Universities (FEUP, IST, UA, UM, UC and UNL) are described, giving a picture of the effort being played in these institutions to improve the experimental education.

Chemical Engineering Laboratories III at FEUP

Adélio Mendes*; Fernão D. Magalhães and Miguel Madeira

The general objectives defined for Chemical Engineering Laboratories III, taught during the first semester of the fourth year of a five year Chemical Engineering course, are: i) to provide a practical perspective on the theoretical concepts taught in the reaction engineering and advanced separations processes courses, ii) to develop students' conception, execution and operation capabilities and iii) to develop their written and oral communication skills, team work and work discipline competencies.

This laboratory course started six years ago from scratch. It was a great opportunity to re-think its philosophy and to introduce new concepts. Firstly, the objectives were clearly stated. Then, after a careful bibliography review, a set of experimental setups was designed, assembled and tested. Four principles guided us during the selection/development of the experiments: 1) Intrinsic safety; 2) Low price and low operation cost; 3) Environmental safety and 4) High didactic content. In addition to the in-lab work, field trips are organized in close contact with the neighboring industry. Emphasis is placed on process units related to the scope of the course.

A complete lab manual was written, including not only the description of each experimental work, but also introductory texts on the field trips, the student's evaluation criteria, topics on data treatment, etc.

Description

In a typical semester, students have 12 sessions, 3 hours long. They work in groups of 2, arranged in classes of 5 to 6 groups. The first session is dedicated to introducing the course organization, security issues and operation of measurement instruments and other devices. A handbook is provided to each student at this time. The handbook contains the course organization, topics on statistical data treatment, description of the field trips, and, for each experimental work, it considers: i) Objectives; ii) Theoretical introduction; iii) Description of the experimental procedure; iv) Example of experimental results; v) Safety items; vi) Waste deposition; vii) Bibliography, among other items.

Each group has to perform 7 experiments of a total of 14 experiments available:

<u>Ideal reactors,</u>

1 – Kinetics and activation energy measurement of a liquid homogeneous phase reaction; i) crystal violet + sodium hydroxide; ii) ethyl acetate + sodium hydroxide; iii) crystal violet + sodium hydroxide;

2 – Flow characterization of an open reactor using a tracer; i) Stirred reactor; ii) Tubular packed reactor with glass spheres; iii) Tubular reactor;
3 – Steady state conversion of an open reactor; i) Stirred reactor – crystal violet + sodium hydroxide; ii) Tubular packed reactor with glass spheres – ethyl acetate + sodium hydroxide; iii) Tubular reactor – crystal violet + sodium hydroxide;

<u>Catalytic reactors,</u>

4 – Study of the sucrose inversion on an ion exchange resin packed flow reactor and on an immobilized invertase packed flow reactor;

<u>Non-conventional separations processes,</u>

6i – Study of an ion exchange resin;
6ii – Study of the carbon dioxide adsorption on an activated carbon packed column;
7 – Study of the permeability of a hollow fiber module towards helium, nitrogen and argon;

<u>Powder technology,</u>

8 – Particulate solids storage, pneumatic transport, separation and mixing;

The lab lessons start with a multiple-choice quiz. The purpose of this small quiz (5 questions) is to evaluate whether students are sufficiently prepared to attend the lab session. The students themselves suggested this quiz, in order to improve their performance during the lab examination. Many times students lack the theoretical background associated to the experimental work they are performing. We found that these lab classes are a perfect opportunity to clarify some fuzzy concepts on residence time distribution, adsorption wave fronts, heterogeneous catalytic reactions, etc. After explaining students that in a short future they will be asked to be problem solvers and that their job depends on their performance, they become more motivated.

Computer data acquisition has been implemented on all experiments. We tried to always use measuring instruments equipped with a RS-232 port, which provides an economic way to transfer data to/from a computer. The user interfaces are simple and intuitive to use, allowing students to visualize graphically, in real time, the time dependence of the measured variables. At the end of each lesson, students give the professor a copy of their results (an A4 page containing their name and class identification and the file name where the results were stored). They are asked to present a short report of their work on the next lesson. A template is provided so they only have to fill in the experimental results, make the necessary calculations and write the corresponding discussion and conclusions.

Students can view a well-organized laboratory, with reliable and didactic experiments, where environmental issues are respected, as a model for their future work life. Also, the fact that we succeeded to sell one of our experiments to Armfield Ltd, the largest international company selling experiments for undergraduate laboratories, and to publish three papers related to these experiments in international education journals (CEE and JCE), provides an additional incentive to students.

The Chemical Engineering Undergraduate Laboratories at IST – an Overview

Carlos Henriques*

Some years ago, in 1994/1995, an important modification takes place in the undergraduate laboratories organisation on the Chemical Engineering Department of IST: the “umbilical cord” between laboratory courses and the corresponding theoretical courses of the graduation *curriculum* has been cut: laboratories organisation has become autonomous, new responsible professors has been nominated by the head of the department.

The Integrated Laboratories – A small balance

The main idea of the group in charge of the implementation of the Integrated Laboratories [1] was to avoid a decrease of both the quality and the exigency level of the student’s work and evaluation, due to the introduced modifications. It’s clear today that this goal was globally achieved but with an important drawback: a significant number of students performed their lab work, during a given semester, before the corresponding theoretical subject was presented. Nevertheless, the essential of the subjects was maintained as laboratory’s work and a high exigency level was kept: four semesters laboratory courses were organised during the “common trunk” period and other four courses were organised for the different branches (Processes and Industry, Biotechnology and Applied Chemistry).

[1] – Relatório da Comissão de Implementação dos Laboratórios Integrados, DEQ – IST, Junho 1994

In the particular case of Process and Industry branch, a very small level of integration was really achieved, mainly due to the intrinsic characteristics of the included subjects, to the "chronic" lack of funds and also to some inertia of the system.

One of the main modifications was the introduction of a module corresponding to the process simulator ASPEN teaching, dedicated to the students of the 4th year, 2nd semester. This constituted a very important improvement, mainly to be applied in the Chemical Engineering Project work, to be performed during graduation 5th year.

The new *curriculum* on Chemical Engineering graduation – the labs modules

In the LEQ-IST web site [2], in what the objectives and curriculum structure of the chemical engineering degree is concerned, one can read that "in laboratory courses, the objectives of thematic integration, organisation and team work planning must be privileged" as well as "informatics' use, including modelling and simulation, must be reinforced". Furthermore "students must be acquainted with the use of professional-like process simulators from the 3rd semester (LEQ I), in order to be prepared to use it in the Project course" in the degree's last year. The "reinforcement of student's critical analysis, the development of their abilities for communication, as well as the contribution to increase the skills to approach problems with a chemical engineer attitude " is also foreseen.

An example: the LEQ III course (3rd year, 2nd semester)

The Chemical Engineering Laboratories III (LEQ III) corresponds, to each student's group, to a 12 sessions course, 4 hours long each one and includes the following four subjects:

- Mass transfer (diffusivity of a gaseous mixture, mass transfer in a wetted wall column, mass transfer coefficient by the electrochemical limiting current technique);
- Multiphase systems operations (elutriation, fluidisation, pressure drop in a packed column, filtration);
- Ideal homogeneous reactors (batch, CSTR and tubular reactors for the halogenation of acetone);
- ASPEN (simulation of a homogeneous ideal reactor).

The students are divided in groups of three elements each, in classes of six to eight groups.

A support handbook is given to each group, were each experiment is presented. This includes the objectives of the work, a small theoretical introduction, a detailed description of each experiment, the expected main type of results discussion, as well as some bibliographic references. At the end of the session, each group gives the professor a file with their results. Final evaluation includes, for each subject, the analysis of a report of the performed work, that students present during or at the end of the semester.

Chemical Engineering undergraduate Laboratories at UA

Inês Portugal*; F. Avelino Silva, Carlos M. Silva e João Coutinho

Chemical Engineering (ChE) at Universidade de Aveiro started in 1995/1996, within the Chemistry Department. Experimental teaching classes were defined as independent courses that should illustrate the major ChE topics and promote knowledge integration. Furthermore competencies such as team-work, written and oral communication skills, time-management strategies, etc. should be developed.

During the 2nd and 3rd years undergraduate ChE students have Chemistry Labs, covering subjects such as Organic, Inorganic, Analytical and Physical Chemistry. During the 3rd year students have their first ChE laboratories covering Transport Phenomena (at present they have 4h/week x 6 weeks/semester; in the near future they will have 3h/week during two semesters). During the 4th year students have 6h/week of ChE Labs, during two semesters, covering Chemical Reaction Engineering, Process Separation, Process Dynamics and Control. These labs have been running since 98/99, with an increasing number of students and so we might say they're "under construction".

Description

In a typical semester, students have 12-13 sessions, 6 hours long. The first session is dedicated to introducing the course organization and security issues, a brief introduction of each experiment and experimental apparatus, a brief presentation of available calculus software. Students are organized in groups of 2 or 3, in classes of 5 groups.

Each group performs 5 experiments/semester. Each experiment occupies two Lab sessions. In the first week students perform the experimental work and if possible start to organize the experimental data in the available computers; in the second week they meet in the lab to discuss results/calculus with the teacher; the final report must be handed in before the next Lab session. At the end of the semester each group gives an Oral presentation to the rest of the class, while another group is appointed to ask 2 or 3 questions.

[2] – http://dequim.ist.utl.pt/objectivos_leq.html

The Lab handbook, provided to the students in the beginning of the semester, contains information about course organization and evaluation, topics on writing of technical reports, the description of each experiment and some suggestions for data analysis. One or two related scientific papers per experiment are also provided.

At present, the available experiments are:

1st semester

1- SEDIMENTATION - Batch sedimentation tests and design of a continuous sedimentation unit.

2- FILTRATION - Constant pressure filtration, determination of cake compressibility factors and design of a large scale filter.

3- DISTILLATION - Batch distillation of a binary mixture in a packed column; determination of the number of equivalent plates and equivalent height.

4- HOMOGENEOUS REACTION - Determination of steady-state conversion and residence time distribution curve in a continuous reactor. Comparison of predicted conversion (using published kinetics and the RTD curve) with the experimental result.

5- HEAT TRANSFER - Determination of the global heat transfer coefficient in a jacketed stirred tank reactor.

2nd semester

6- ADSORPTION - Determination of the adsorption isotherms and breakthrough curve in a fixed bed column.

7- EXTRACTION - Hydrodynamic study of a liquid-liquid extraction column.

8- DISTILLATION - Distillation of a binary mixture in a perforated plate column.

9- HETEROGENEOUS REACTION - Resin catalyzed hydrolysis in a batch reactor; determination of reaction rate as a function of particle diameter; estimation of the effective diffusivity. Design of a continuous packed bed reactor.

10- CONTROL - Level control in tanks using an industrial PID.

Conclusions

In general students say these labs are demanding and time consuming, but they acknowledge their importance. Students are obliged to work all year round, not just for the exam. Some of the experiments explore topics that haven't been covered in previous courses; this requires an extra effort from the students but prepares them for lifelong learning. Furthermore, they become more alert and motivated in parallel theoretical courses.

Chemical Technology Laboratories (CTL) at the Department of Biological Engineering of the University of Minho

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Introduction

The CTL course started 12 years ago when the first students of the Biological Engineering degree attained the third year (2nd semester). The practical works are mainly focused on chemical reaction engineering and separation processes, but transfer phenomena and calorimetry are also included in the programme. There are at the moment 10 available works making use of different analytical techniques:

1. Determination of the order and the activation energy of the hydrolysis of the Terbutyl Chloride. *Analytical technique: potentiometry-pH.*
2. Determination of the order and the activation energy of the reaction between violet crystal and sodium hydroxide. *Analytical technique: spectrophotometry*
3. Flow characterization and modelling through tracer methodology. Aplication of the 'Dispersion Model' to a packed column. *Analytical technique: condutimetry. Mathematical tool: Solver – Excel.*
4. Flow characterization and modelling through tracer methodology. Aplication of the 'Tanks in Series Model' to a cascade of 4 stirred tanks. *Analytical technique: spectrophotometry. Mathematical tool: Solver – Excel.*
5. Flow characterization and modelling through tracer methodology. Aplication to a tubular tank, a stirred tank and to a series of a tubular and a stirred tank. *Analytical technique: Condutimetry.*
6. Design of a stirred reactor from batch data. Experimental validation. *Analytical Techniques: spectrophotometry and potentiometry-pH.*
7. Rayleigh distillation. *Analytical technique: refractometry*
8. Filtration at constant pressure. *Analytical technique: gravimetry.*

9. Oxygen transfer from air to liquid media: Influence of aeration and stirring power on kla. *Analytical technique: potentiometry- O_2.*
10. Calorimetry: determination of reaction enthalpies (Terbutyl Chloride+water).

The classes have an average of 15 students working in groups of 3. Initially each work was designed to be completed in 2 classes of 6 hours each. The analytical instrumentation was connected to analogical recorders, but, in the last two years efforts are being made in order to upgrade the works with new instruments equipped with RS232 ports for on-line data acquisition. Presently each class last 6 hours and during the semester six works are performed. Each group has to meet the professor individually during 15 minutes for oral evaluation and discussion of safety and waste disposal procedures that are compulsory defined by the students and verified by the supervisor before starting each work.

A SWOT (Strengths, Weaknesses, Opportunities, Threats) analysis

Strengths

It is difficult to recognize the strong and the weak points. Students were asked about them and this analysis is in part result from their feedback. In general the protocols are not restrictive and on purpose, the theoretical basis is not completely explained in there. A previous study and search is essential, because all students will be orally evaluated before starting each work. Students have some degrees of freedom as a way to stimulate their creativity and initiative in the lab work. The number of runs or experimental points is not pre-defined. Students think and decide with the constant support of the professor who should be all the time at the lab. The aim is not only to acquire a practical perspective on the theory learned before, but also to solve practical problems that can arise at any time in the lab. The analytical instrumentation should be calibrated by the students. In the protocols there are no examples of experimental data because students will be limited to get something similar. From our experience this would produce bad results in terms of apprenticeship. Reports are discussed with each group in the next class after they returned them to the professor. This is referred by the students as one of the most positive points because they can correct on time the errors that otherwise will be repeated over all the works.

Weaknesses

In the last two years internal new rules of the academic administration allowed students from the second year to attend some classes of the third year. This caused severe problems in the CTL classes because the theoretical background was not previously acquired. For instance there were students performing a residence time distribution practical work without knowing the definition of hydraulic retention time. Other important weakness is the low weight of CTL in terms of credits. Students, even the best ones, do not invest too much effort on the CTL classes due to the little importance in the final classification.

Opportunities

This attempt to joint experiences and discuss with partners from other universities is welcome for us. The new directives to reduce engineer courses from 5 to 4 years will force to focus on the essential and will give the opportunity to re-think all the philosophy of the CTL classes.

Threats

Apart the above explained weaknesses, the more important threat in all lab classes is the constant attempt of students to have success with the minimum possible work. Not all, but most of them are only interested in finish as fast as possible the experimental work and if possible to get some good data and good reports from other students. This is a bad practice and some mechanisms should be promoted in order to limit it. One possibility is to change as much as possible the aim of the works or the works themselves, to make objectives questions to be answered in each report and to be creative.

Chemical Engineering Laboratories at FCT/UC

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In Coimbra the Chemical engineering course is organised in ten semesters. In the 1st and 2nd years the undergraduate students have Chemistry Labs covering General, Analytical, and Organic Chemistry. This work is performed at the Chemistry Department. All the subsequent teaching is carried out in the Chemical Engineering Department.

The disciplines of Chemical Engineering Laboratories I, II, III and IV were introduced in the 5th, 6th, 7th and 8th semesters of the present plan of the Chemical Engineering course in 1984/85. Laboratory experiments concerning the contents taught in the theoretical classes in the same semester of the course are grouped in each respective discipline. The objective of these disciplines is to provide the means to experiment and understand the chemical engineering fundamental concepts, improving the students ability to learn, and at the same time develop oral, written and team work skills. The structure of these disciplines is intended to optimise the human and laboratory resources available at the Chemical Engineering Department. Optional disciplines included in the 4th and 5th years of the course have their own laboratories. In the last year (5th) students take a discipline intended to work as an introduction to Chemical Engineering Research.

Description

Chemical Engineering Laboratories I and II are both included in the 3rd curricular year. In these disciplines the students perform 5 experiments, 6 h/week, in non-sequential weeks. The weeks between the lab sessions are used by the students to elaborate a report and discuss it with the professor, make oral presentations of three of the experiments and discuss the data treatment of the last one. This accounts for a total of 10-12 sessions (10 groups per session) per semester, including the first session dedicated to a brief introduction of each experimental apparatus. In Chemical Engineering Laboratories I and II the experiments comprise the following subjects:

<u>Fluid Dynamics</u>

- Discharge of a tank through an orifice / exit pipe;
- Experimental determination of the flow rate in open channels – Discharge through an orifice / Venturi meter;
- Experimental determination of head losses and friction factors for water flow in pipes with fittings and valves;
- Experimental determination of critical Reynolds number;
- Experimental validation of Stokes law;
- Fluid rheology / Viscosity of newtonian liquids;
- Chemical Thermodynamics;
- Partial Molar Volumes for the system ethanol-water;
- Experimental determination of a ternary diagram (equilibrium curve and tie-lines): system acetic acid- water-chloroform.

<u>Instrumentation</u>

- Calibration of Thermocouple and Resistance thermometer.

<u>Heat and Mass Transfer</u>

- Thermal conductivity of solids / Unsteady-state heat transfer by conduction;
- Heat transfer by convection;
- Diffusion coefficients in liquids / Diffusion coefficient of n-hexane in air.

<u>Unit Operations</u>

- Project of a continuous sedimentation unit from batch laboratory experiments;
- Flocculation of a clay suspension;
- Filtration of a bleached pulp suspension;
- Experimental determination of the characteristic curve of a centrifugal pump (influence of pipe diameter).

<u>Kinetics</u>

- Experimental determination of kinetic parameters of liquid homogeneous reactions: potassium persulphate + potassium iodine; ethyl acetate + sodium hydroxide.

In the Chemical Engineering Laboratories III and IV, during the 4th year, the students perform in alternate weeks, 4 experiments, 3 hr/week. In each session are present 4-5 groups. The time between lab sessions is used for the elaboration of two small reports, which include mainly data treatment, and two complete reports. The students make an oral presentation of one of these reports and discuss the other with the professor. In the majority of the experimental apparatus it is possible to perform on-line data acquisition. The available experiments are included under the following topics:

Unit Operations

- Batch distillation of a binary mixture in a pilot-scale packed column
- Continuous distillation of a binary mixture in a perforated plate pilot-scale column.
- Liquid-liquid extraction in a pilot-scale packed column
- Experimental determination of heat-transfer coefficients for double-pipe heat exchangers.
- Heating liquids in tank storage.

Process Dynamics and Control

- Dynamical response of a second-order system (two interacting tanks);
- Level control in interacting tanks using a PID controller.

Chemical Reactors

- Determination of steady-state conversion in a battery of continuous stirred tank reactors;
- Study of gas-solid fluidisation regimes in a ballotini bed column;
- Determination of residence time distribution curves in a battery of continuous stirred tank reactors and in a fixed-bed tubular reactor;
- Study of the operation of an adsorption column.

Conclusion

During the lab session the professor addresses several questions to the students in order to improve their capacity to critically evaluate the results obtained and to access their preparation of the experiment. The students find these disciplines very time consuming. The fundamental concepts are taught at the same time in the theoretical classes. The recent installation of the Chemical Engineering Department in "Polo II" of the UC provided the rare occasion for repairing the old experimental apparatus and increase the number of available experiments by including new ones. This provides the means to offer within each of the Chemical Engineering Laboratories disciplines different experiments in each year.

Laboratories of Chemical Engineering at FCT/Universidade Nova de Lisboa

Academic staff of FCT/UNL

Experimental learning is a major objective of the Chemical Engineering course at FCT/Universidade Nova de Lisboa. During the first two years the students have a significant training on Chemistry and Physics laboratories, while during the three last years of their studies they are involved in different Chemical Engineering laboratories. During the two last years, 40% of their classes are spent in the laboratory.

The laboratory classes are organized under different categories: 1 – Demonstration Laboratories, associated with specific disciplines; 2 – Computation Laboratories; 3 – Research Project and; 4 – Design Project.

The Demonstration Laboratories are organized in close interaction with individual disciplines, or group of disciplines, and they intend to promote the student's understanding of fundamental concepts introduced in the theoretical classes. The groups of disciplines that include Demonstration Laboratories are: Chemical Reaction Engineering, Mass Transfer and Separation Processes, Biochemical Engineering, and Process Control and Automation.

The Chemical Reaction Engineering laboratories involve the following experimental sessions: determination of kinetic parameters of chemical reactions (overall order of reaction, partial orders and activation energy); experimental determination of the residence-time distribution (RTD) and modeling by the association of ideal reactors; prediction of the conversion for a given reaction, by using the segregation model and the maximum mixedness model; determination of Thiele modulus and effectiveness factor values for different pellets of Pd/C catalysts; preparation and characterization of polymeric materials.

The laboratories of Mass Transfer and Separation Processes include the study of different operation units, namely: liquid-liquid extraction equilibria and kinetics in a stirred cell, heat and mass transfer in a cooling tower, adsorption in an activated carbon packed-bed column, and micro/ultrafiltration of defined suspensions in a tubular membrane module.

The Biochemical Engineering laboratories involve an integrated study of a fermentation process: determination of K_La values under different operating conditions; kinetics of cell growth, substrate consumption and product formation; strategies for bio-process monitoring and control.

The Process Control and Automation laboratories include experimental work on: dynamic response of a second-order system consisting of two non-interacting or interacting tanks and; tuning of a PID controller for liquid-level control of two interacting tanks.

The Computation Laboratories are organized in association with individual disciplines. Essentially they involve problem solving and process design. We are currently training our students on the use of computational tools for problem solving at meso and macro scales. These include a general high-level computing language (MATLAB), a Computational Fluid Dynamics package (shareware version of PHOENICS), a DAE solver and optimizer (gPROMS), and a steady-state process-oriented spreadsheet (ASPEN). It is expected that, in the near future, the computational tools offered to the students will be enlarged to include to include molecular simulation.

In the Research Project (1 semester, 15 weeks, 10h/week) each students develops, individually, his own research in a given topic under the supervision of the academic staff. It is intended to develop the ability of the students to plan their experimental work, to build their own (relatively simple) experimental set-up, and to interpret the data acquired. The students have to prepare a final report and to present a seminar to the class and the academic staff.

Under the Design Project laboratory (1 semester, 15 weeks, 7h/week) the students have to implement a selected patented process (chemical reaction, product recovery and characterization) to be designed.

Recently, a 200 m^2 pilot-plant area has been equipped with pilot-scale units, which are used for industrial demonstration projects and for teaching purposes (undergraduate teaching and monographic courses). These include a chemical reactor fully equipped, a heat exchanger, a cooling tower, a batch distillation column and a micro/ultrafiltration capillary membrane unit. Computer data monitoring and control has been installed on all units allowing the students to acquire information on real-time.