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Operation and Control of a Distillation Column as a Tool to Teach the 'Real Problem'*

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This paper examines the benefits provided to undergraduate students by the dynamic operation of a fully automated pilot plant. The experiment was designed to foster a deep understanding of control concepts and to offer the opportunity to deal with real control devices and problems. The students need to identify and set the proper control configurations, tune the control parameters and test the controllability for typical process upsets following a stop and go experimental organization. We believe that this hands-on approach is an indispensable step in the formation of our future engineers.

NOMENCLATURE

K_u ultimate gain
K_{vs} valve capacity
K_c controller gain, [%]
P_u ultimate period, [s]
T temperature [°C]

Greek letters
θ time-constant used in the Ziegler-Nichols open loop [s]
τ time-constant used in the Ziegler-Nichols open loop [s]
τ_D derivative time [s]
τ_I integral time [s]

INTRODUCTION

THE CONTROL of chemical processes is necessary to assure both the quality of the final product as well as the safe operation of the plant. As such, the teaching of control forms an indispensable part in the training of chemical engineers. The recent innovations in teaching methodologies make it important to reconsider the strategies used to teach control at the undergraduate level. In particular, we should think about the fundamental questions involved:

- What is the scope of control in which our students should be educated? (We train many people for the academic area, where the jobs are getting scarcer.)
- What is the message we want to convey to our students? (We tend to focus on continuous processes, excluding batch processes.)
- Is there a single educational style? (We need to balance the expensive experimental work with the necessary theoretical background.)

In this article, we describe a distillation process that is fully controllable and operable from a remote computer through a PLC (Programmable Logic Controller) [1, 2]. The students are required to plan, operate, and present results and conclusions taking into account operational constraints, starting with easy tasks (feed preparation) to more complex (parameter tuning), all within the scheduled time. The principal objective is to acquire the methodology of control and optimization of an automated continuous process. The instructors assume that the operational procedure is known (startup, steady-state operation and shutdown protocols). At the end of the experimental work, the teams are required to deliver a technical report and make a public presentation that includes a question-and-answer session.

COURSE ORGANIZATION

The educational approach was revised at the School of Chemical Engineering (University Rovira i Virgili, Tarragona, Spain) switching the emphasis from instructor-based teaching to student-centered learning [3, 4]. In the area of process control, diverse activities are distributed across the curricula. The students are first introduced to the basic concepts in the compulsory course Process Control and Instrumentation (60 hours) after which they can take the elective course Advanced Control (45 hours). The knowledge gained from these courses is applied in the Laboratory of Process Manufacturing (120 hours) as well as in the Final Year Design Project.

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The main characteristics that need to be taken into account in designing laboratory courses include:

- class size (24 groups of three students);
- intellectual maturity of students (students experienced in team-work);
- student motivation (very high since they freely operate a pilot plant without interference from the instructors);
- learning objectives (dynamics of chemical processes),
- instructor's preferences (Integrated Projects developed throughout each course [5]).

The Laboratory of Process Manufacturing is taken during the fourth year. The course is devoted to process dynamics and operation both experimentally, with a semi-automated batch plant as well as a fully controlled continuous plant, and by process simulation (Hysys.Plant®, PRO-II©). In addition, the course addresses other no less important topics such as plant safety or troubleshooting (insights are needed for a rapid and qualitative interpretation of how each variable influences the system performance).

The laboratory is based on *open-ended problems*, so that the students face situations in which there are no step-by-step detailed guidelines on how to carry out the activities. Non-technical capabilities and abilities such as problem-solving skills, planning, management, teamwork, responsibility, accountability and decision-making are also emphasized. However, a minimum guide is provided in order to standardize the activity and to reduce the time spent on side issues (e.g. familiarization with the experimental set-up). In this way, results from different groups are comparable, full-size lapses are minimized and students can be easily evaluated. When the students are sufficiently familiar with the methodology, the instructors pose challenging questions that help enforce creative thinking and maintain continuous feedback.

MATERIAL AND METHODS

Experimental equipment

The pilot plant was built by ASESA (Barcelona, Spain). The experimental unit has three sections: stock tanks, feed preparation, and a distillation column. The control is carried out using a Honeywell S9100 system (Morristown, NJ, USA), which is connected though a LAN to the main computer located outside the experimental area which runs the central station supervision program (SCAN 3000). The equipment was purchased four years ago ($\approx$100 000 US$), thanks to the special funds provided by the Catalan Government to endow the new teaching facilities of the ETSEQ.

Where possible, the experimental equipment is made of glass (Schott Ibérica, Barcelona, Spain), hence increasing the educational value, as the students can *see* the internals and liquid and vapor distribution in the column. The technical characteristics are:

- The distillation column (8.5 cm internal diameter) is equipped with a thermosiphon reboiler and an air-cooled condenser (AT–44D, Pecomark S.A., Barcelona, Spain). The column has three 45-cm packing sections of Multifil® stainless steel (KnitMesh Limited, Surrey, England).
- The vessel V–1 has a volume of 10 L. The agitation system is a RM–144D (Schott Ibérica).
- Endress + Hauser (Weil am Rhein, Germany) was the provider of the differential pressure transmitters (PMC 731, Deltabar S), differential pressure indicators (PMD 230), level probes and indicators (DC 11/26 TEN, FEC 12) and PT–100 thermometers (TST 220 and TST 240).
- Peristaltic pumps (303 FAC/D, Schott Ibérica) attached to the 300-series pump head (Watson Marlow, Falmouth, England).
- Control valves with pneumatic actuators (Schubert & Salzer, Worcester, UK) were used (Series GS, models 8020, 8021, 8030 and 8044). Solenoid valves are Bacosol from KV Automation Systems (Buckinghamshire, England). The SRI (model 8020) electro-pneumatic positioner was from Eckardt (Stuttgart, Germany).

Analytical methods

The analyses of ethanol and water are carried out in a Hewlett-Packard (Palo Alto, CA) 5890 Series II Plus gas chromatograph, equipped with a TCD detector using helium as the carrier gas. The packed column is a Porapak Q (2 m, 2 mm i.d., 80 mesh, Supelco). The signal is integrated with a Hewlett-Packard 3396 and related to composition through a calibration curve. The curve was provided to the students, given that not enough time is available to include the calibration in the experiment.

Experimental procedure

Students have three sessions each one of four hours to perform all the experimental work (no more time is available due to laboratory time constraints). The schematic P&ID provided (Fig. 1) gives a general idea about the process, but due to the complexity of the pilot plant, students are required to identify the main elements (instruments, pumps, valves and tanks) *in situ* the day before they start the experimental work.

The first step involves the preparation of the feed mixture. An ethanol + water mixture at 50% w/w is prepared in vessel V-1 from tank T-1 (water rich) and tank T-2 (ethanol rich). The product obtained is sent to tank T-3 if the analytical results are satisfactory. In this way, students learn how to perform easy tasks and familiarize themselves with the different control screens and the operational problems regarding any distributed control system (4 hours). The mixture is then fed to the distillation column from one of three possible feed points and the products are collected in T-4 (bottoms) and T-5

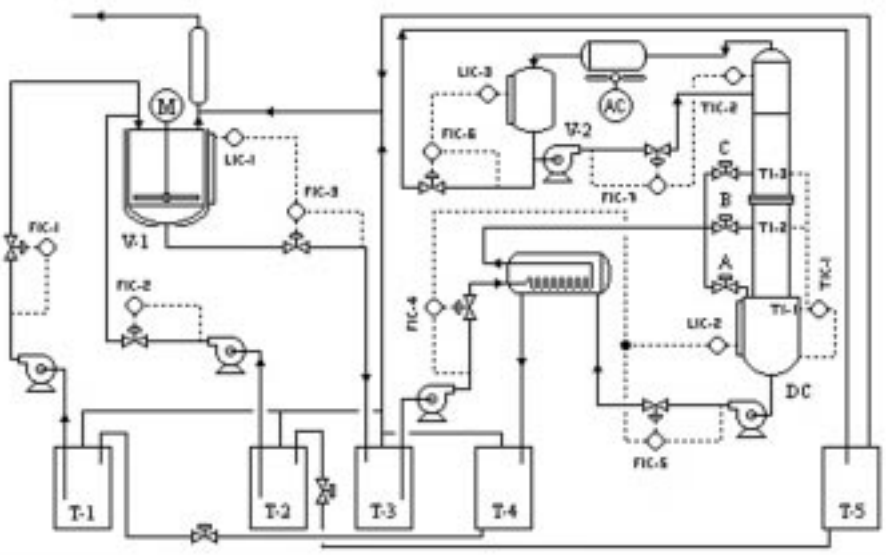

Fig. 1. Schematic diagram of the experimental set-up. AC: air condenser; DC: distillation column; FIC: flow indicator controller; LIC: level indicator controller; TIC temperature indicator controller; M: mixer; T: tank; V: vessel.

(distillate). After checking the composition, the contents are returned to tanks T-1 and T-2, respectively, thus closing the loop. On the control side, the students have to use the different P&IDs from the computer, identifying the control loops, managing the alarm recognition, operating the equipment from the computer and tuning the respective controllers. The second day, total reflux conditions are achieved after approximately 40 minutes of operation. Students must take actions to maintain the process variables stable, although a true steady state is seldom achieved (4 hours).

Ziegler-Nichols tuning

The actuating output, $c(t)$, as a function of time of a proportional + integral + derivative (PID) controller is given by:

$$c(t) = K_c\varepsilon(t) + \frac{K_c}{\tau_I}\int_0^t \varepsilon(t)\,dt + K_c\tau_D\frac{d\varepsilon}{dt} + c_s \quad (1)$$

where K_c is the proportional gain of the controller, τ_I is the integral time, τ_D is the derivative time and c_s is the controller's bias signal when $\varepsilon = 0$. The error, $\varepsilon(t)$, is defined as the difference between the set point and the measured output signal.

Although most of the control loop configurations of the process are fixed, the students need to choose the type of controller that they wish to use between P (proportional), PI (pro portional + integral) or PID (proportional + integral + derivative). Once chosen, the parameters of the controller have to be appropriately tuned. Even though a few students decide to take the direct route of a trial and error approach, the majority opts for the use of the Ziegler-Nichols methodology either with the controller connected (closed-loop) or with the controller disconnected (open-loop) [6–8].

- *Closed-loop:* The integral and derivative actions are deactivated and only proportional action is left on. A step upset is introduced, and the ultimate gain where harmonic oscillations are shown, K_u, is found by a trial and error procedure (Figure 2a). At the same time, the ultimate period of the sustained cycling is measured, P_u. The parameters K_u and P_u are used to estimate the settings for feedback controllers according to Table 1.
- *Open-loop*: As response times of some of the control loops are slow, the students find that in certain cases it becomes unpractical to apply the aforementioned closed-loop algorithm. In this case, the feedback mechanism of the controller is deactivated and the dynamic response of the system to a step change in the manipulated variable is studied (Fig. 2b). For a feedback controller, the values can be calculated according to Table 1 [6] where θ and τ are the time constants and S^* denotes the normalized slope ($S^* = S/\Delta p$, where Δp is the magnitude of the step change that was introduced in the controller output and S is the slope of the response).

RESULTS AND DISCUSSION

Feed preparation

In order to prepare the feed mixture with the desired concentration (50% w/w), the students need to mix the contents of T-1 and T-2 in the correct ratio. This ratio depends on the concentrations of the mixtures in the feed stocks, measured by gas chromatography. The students compute the required flowrates by a simple mass balance, together with the restriction that the individual values need to be lower than the maximum pump flowrates. All valves have a capacity, K_{vs}, of

Table 1. Closed and open-loop Ziegler-Nichols parameters [6]

	Closed–loop Ziegler-Nicholds			Open–loop Ziegler-Nichols		
	P	PI	PID	P	PI	PID
K_c	$K_u/2$	$K_u/2.2$	$K_u/1.7$	$1/\theta S^*$	$0.9/\theta S^*$	$1.2/\theta S^*$
τ_I		$P_u/1.2$	$P_u/2$		3.3θ	2θ
τ_D			$P_u/8$			0.5θ

$0.04\,m^3 \cdot h^{-1}$ (maximum flowrate with a pressure drop of 1.5 m of water) except the bottom product valve (FIC-5) which has a K_{vs} of $4.0\,m^3 \cdot h^{-1}$. Effectively, this valve is clearly over dimensioned and achieves the set point with a small opening, and for practical purposes acts more like an on-off valve rather than a control valve. The students thus typically observe that they are unable to find a reasonable set of parameters that keep the valve acting in a smooth manner. In this way, students experience first hand the importance of valve sizing.

A question often arises about the reason why all the flow meters are located after the pump, and not before. Since the vast majority of times the problems are encountered in pumps and not in pipes, locating the flow meter after the pump helps to detect pumping problems and correct them (e.g. by bypassing with the security pump installed in parallel). Such questions help motivate active discussion within the group members.

Start-up and shut-down protocols

After having prepared the feed mixture, the next objective is to elaborate a procedure for column start-up. Students have to go through a detailed check list:

- Is there enough liquid in the feed tank?
- Are the pumps, the valves and the condenser in automatic or manual operation?
- Are the electric resistances of the reboiler totally submerged?

During distillation start-up, students tend to underestimate the amount of liquid withdrawn from the top to be stored in the reflux drum and to completely neglect the column hold-up. Hence they are often surprised when they find that more feed is required in order to maintain constant the level in the reboiler. In this way, the students learn that hold-up has a significant value that should be taken into consideration.

Students are inclined to focus more on the computer screen and forget about the *real* plant. Several operational problems (leakage, broken connections) cannot be detected by merely looking at the automatic control system and alarms and *in situ* supervision is required. Students also tend to trust unconditionally in the laboratory instruments and they seldom realize that an indicator may not be working properly. To help overcome this preconception, one of the controllers that is found in the computer control screen does not physically exist in the pilot plant. Usually, students state that the instrument is not working properly (the flow meter always has a value of $10\,L \cdot h^{-1}$) and rarely realize by themselves that it is not installed.

A question that students typically pose is the reason why all the tanks are linked together with a pipe connected to the tank headspace. The instructors force the students to reach the answer by themselves using a sequence of questions:

- What will happen if the tanks had different pressures?
- Is there any relationship between pressure and flow?
- What is reverse flow?

In this way, students state that tanks are connected to equalize the pressure of the system and to collect and condense any alcohol in the emissions.

Number of equilibrium stages

Samples are withdrawn at certain points to check the mass balance and compute the number of stages. Usually the students use the McCabe-Thiele method [9] with its assumption about constant molar overflow in the column, although students seldom check it (the latent heats of vaporization are 38 and $40\,kJ \cdot kmol^{-1}$ for ethanol and water respectively). Students find a number of stages of 3.6 ± 0.5, while the supplier information predicts that it should be around 12–15 (Table 2). It is experimentally observed that the liquid flowrate is not sufficient for the column diameter, and also the liquid is poorly distributed over the packing in the current experimental setup. This situation leads to poor wetting and thereby to low efficiencies.

Feed location

Before switching to normal column operation, students have to decide between the three possible locations for the feed. Students are often unsure about the decision and regularly choose the lowest feed location even though it enters directly into the reboiler thus eliminating the stripping section of the column. Given that the system has an azeotrope at high alcohol concentration ($x_{EtOH} \approx 0.78$/mol) as well as a high relative volatility at low alcohol concentration and that the mixture is sub-cooled, the most reasonable location would be in the location that enters above one third of the column packing. These calculations and decisions stress the importance of preliminary work in order to save precious experimental time.

Normal operation

The instructors tell the students that steady state is reached after about fifty minutes of operation. The different team members are thus motivated to organize themselves and carry out additional tasks in parallel with the column operation. In particular, an issue that is often ignored by the students is that the liquid levels, both in the reboiler as well as in the reflux drum, have to be maintained constant

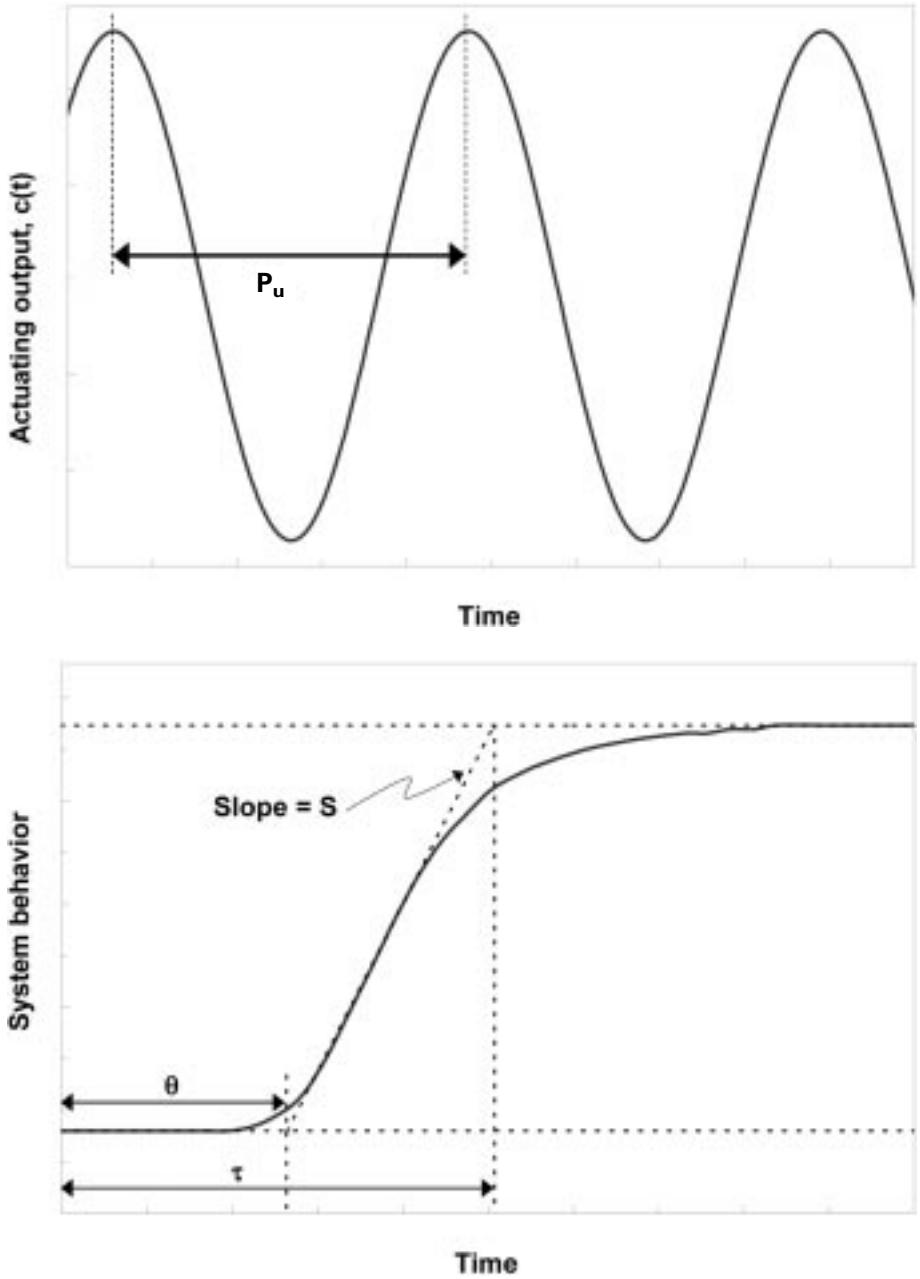

Fig. 2. Theoretical system behavior for a step upset: (a) closed-loop Ziegler Nichols with the ultimate gain (K_u); (b) open-loop Ziegler Nichols.

in order to attain steady-state operation. In addition, the thermosiphon requires that the reboiler level is neither too high nor too low.

The steady state is identified by the attainment of a constant temperature profile in the column. In the actual experimental set-up the middle temperature probe does not work properly and gives a dentate profile. Typically students do not realize by themselves the real cause and the instructor needs to question the team members to help them arrive at the correct conclusion.

Table 2. Experimental results obtained during the last two years

		x_{EtOH}/mol		
	N_{stages}	Feed	Top	Bottom
Mean	3.60	0.4603	0.6817	0.1240
Median	3.00	0.4800	0.6850	0.0705
Standard deviation	1.57	0.1118	0.1004	0.1233

Control hierarchy

One of the most common problems our students encounter is that they are unable to open a certain valve. The detailed equipment information is provided in a different computer screen, and to check the operation of a certain piece of equipment, the up-stream and down-stream specifications must be verified. For example, the valve may be in automatic mode while the pump is

Table 3. Control strategy for the distillation column

Name	Controlled Variable	Manipulated Variable	Remarks
LIC-2	Level	Valve opening,%	Cascaded with FIC-4 or FIC-5
LIC-3	Level	Valve opening,%	Cascaded with FIC-6
TIC-1	Temperature	Power supplied to the reboiler	Cascaded with TT-1, TT-2 or TT-3
TIC-2	Temperature	Valve opening,%	Cascaded with FIC-7
S-1	Feed location	A, B or C, on-off	Block selector

in manual, or there may be an interlock (e.g. even though both the feed valve and pump are in automatic mode, at least one of the feed inlet valves must be open).

Students also experience the difference between limits and alarms. For instance, all tanks have a low-level alarm and a very low-level alarm. In the time lapse between these two signals the operator has to solve the problem. If the very low-level alarm is activated, automatic actions are taken by the PLC (e.g. switch off the reboiler heaters). In this way students have first-hand experience of control structure. The students often need to reflect in order to understand what has happened before continuing with the experiment. Otherwise they may repeat the same action thus activating

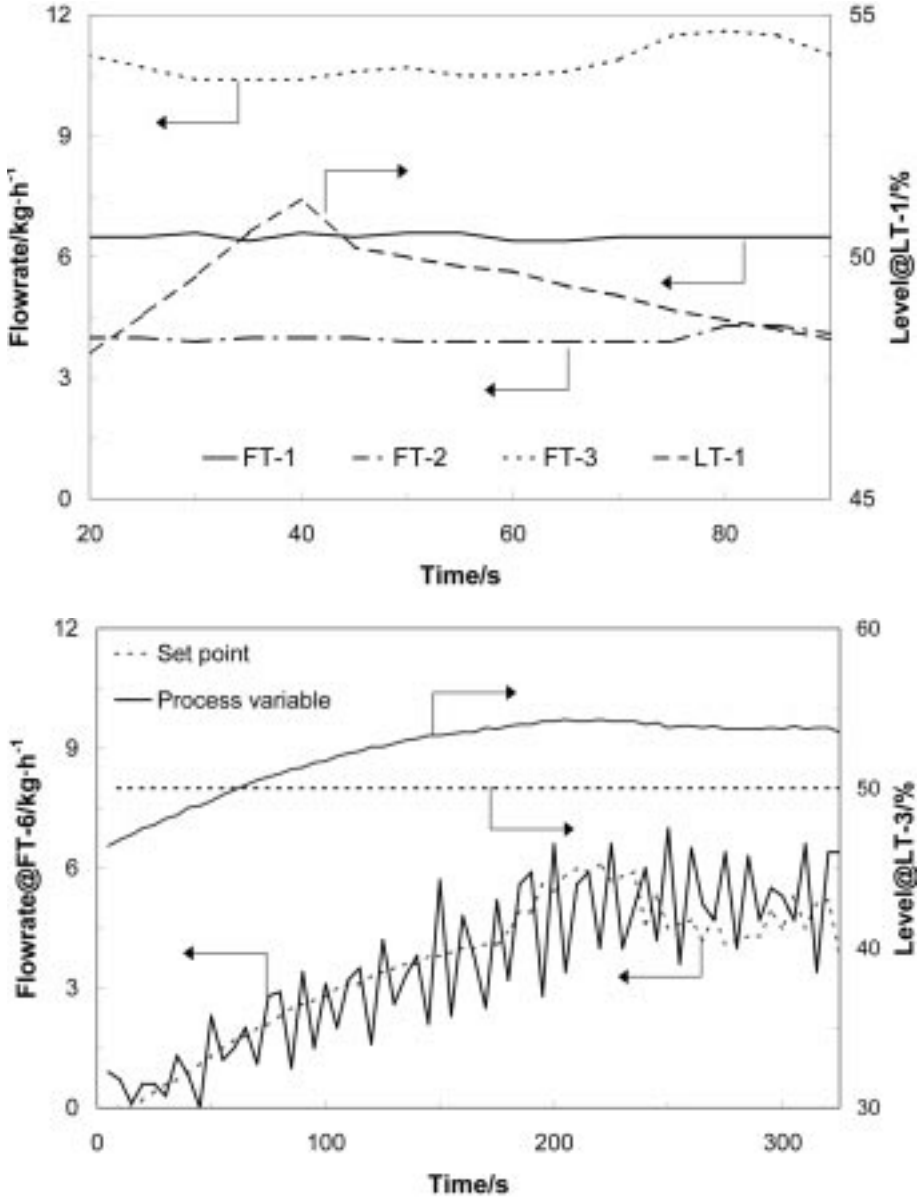

Fig. 3. Level controller: (a) level in the mixture preparation tank (T-1), K_p(FT-1) = 2.5; K_p(FT-2) = 3.0; K_p(FT-3) = 0.7; K_p(LT-1) = 2.0; (b) distillate product flowrate (V-2), K_p(FT-6) = 0.89, τ_I(FT-6) = 3.0; K_p(LT-3) = 3.0, τ_I(LT-3) = 5.0 min.

again the same alarm, with the corresponding waste of experimental time. This stresses the importance of distillation fundamentals.

Control strategy

In order to reach steady-state operation, the column needs to be properly controlled so that the temperatures, levels and flowrates are maintained constant. The software is flexible enough to allow the operator to choose the single-input single-output control strategy, with classical P, PI or PID feedback controllers. The system offers the student the freedom to choose the control strategy (Table 3). Hence, students are able to evaluate the impact of topological changes and have a better understanding of which design is more adequate. As an example Figure 3a shows the level controller in the mixture preparation tank (V-1) for an experiment in which the mass balance closure was found to be reasonable (average error 3.1%). The output flowrate (FIC-3) is performed by gravity and can be controlled either stand-alone (Figure 3a) or cascaded with the tank level controller. Additionally, feed flowrates were controlled (FIC-1 and FIC-2) with standard controllers. On the contrary, Figure 3b shows the results for the level controller in the reflux accumulator drum cascaded with the distillate product flowrate. As can be seen, the process variable tracks the remote set point.

Closed-loop parameter tuning

In order to control the feed flowrate for the column feed preparation, a feedback controllers are used (FIC-01 and FIC-02). In this case, the controller parameters are easily tuned with the closed-loop Ziegler-Nichols procedure, thus allowing the students the possibility of studying the effect of feedback gain on the system performance. In this way, students experience that controllers have a certain lag associated with the process characteristics. According to Fig. 4, the K_u value was 5, while the P_u was 13 s, and the preliminary parameters used for the PI controller were $K_c = 2.27$ and $\tau_I = 2.27$ s. The deadtime of the process is around 20 s. The same technique can be successfully applied to any other flowrate feedback controller of the system.

Open-loop parameter tuning

Some systems are found to have a high inertia and the Ziegler-Nichols closed-loop method is found to be inappropriate. If applied, the harmonic oscillations are very slow and may be affected by the interference of other variables, making impracticable the search of the ultimate gain. In particular, the cascade loop to control the top temperature of the column (the *master* controller that operates the reflux flowrate is TIC-10 and the *slave* is FIC-04) is observed to be very slow. The temperature set point (79°C) was fixed according to the vapor–liquid equilibrium data for the desired overhead concentration. Instead, the open-loop method was selected, and a step upset was introduced into the system by changing the valve action.

Based on the system profile response (Fig. 5), tentative parameters, were found graphically. As expected, a high gain value is found, due to the slow response of this system. The derivative action helps to minimize this effect. A characteristic process lag-time can also be clearly seen in Fig. 5 where there is some time between the impulse and the response.

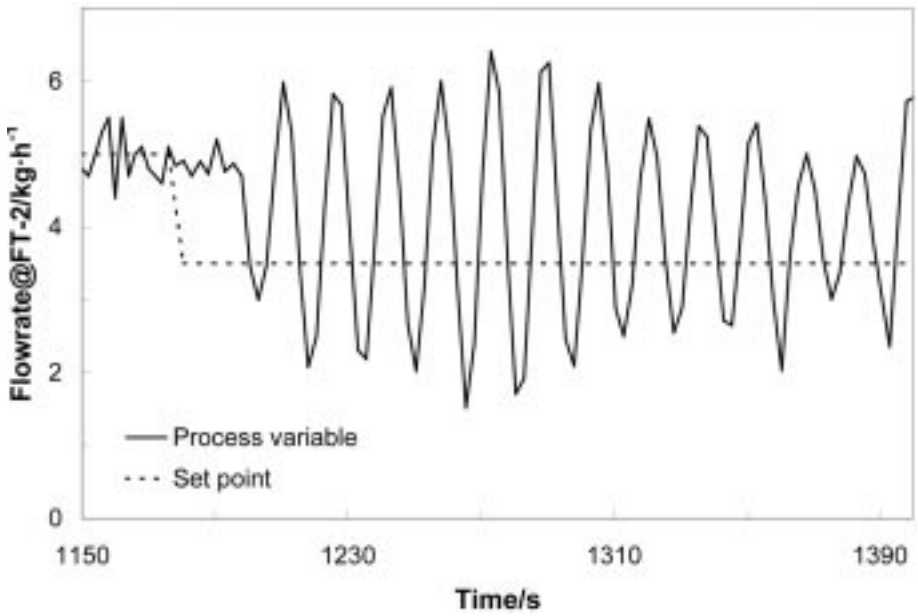

Fig. 4. Feed flowrate tuning by closed-loop Ziegler-Nichols for the FIC-2.

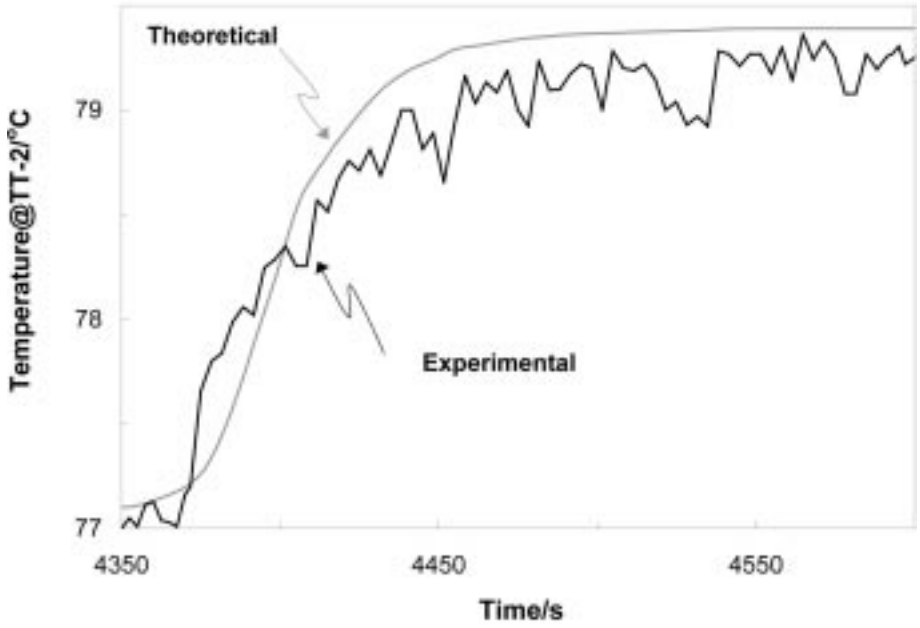

Fig. 5. Open loop Ziegler-Nichols tuning for TT-2 column top temperature, when the reflux valve is closed from 100% to 25%. $\tau = 27$ s, $\theta = 108$ s, Dead time = 14 s, Time lag = 58 s, $K_c = 32\%$, $\tau_I = 30$ s, $\tau_D = 7.2$ s.

Model robustness analysis

At this point all the controllers have been tuned and the students can now observe the incidence on the dynamic behavior of the column for step upsets typically observed in process operation (5–30%, depending on the variable). Students effectively *play* with the parameters of the control loops by testing their behavior and perform a fine-tuning of the system. As expected, the response profiles are not smooth, but rather are dentate. In some cases, the output response is over-damped (Fig. 6), while in others the proportional kickback can be appreciated (Fig. 7), where the response has overshot.

In Fig. 8 a proportional controller action is selected, and therefore very drastic actions follow the system (even without any upset). Moreover, the students observe experimentally that proportional controllers give an offset between the measured variable and the set point. In this equipment it is very frequent to find cross-interactions among different controller loops, and in fact this situation is found in several cases (e.g. column top temperature controller, TIC-2, and reflux drum level controller, LIC-3).

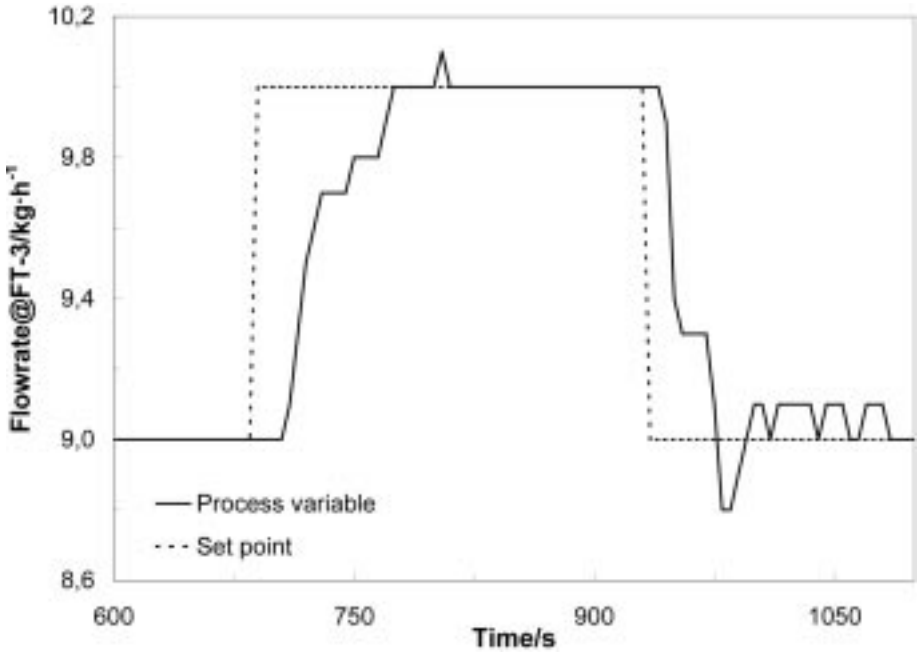

Fig. 6. FIC-03 robustness check for a step upset of 9.0 to 10.0 $kg \cdot h^{-1}$, $K_c = 0.25$.

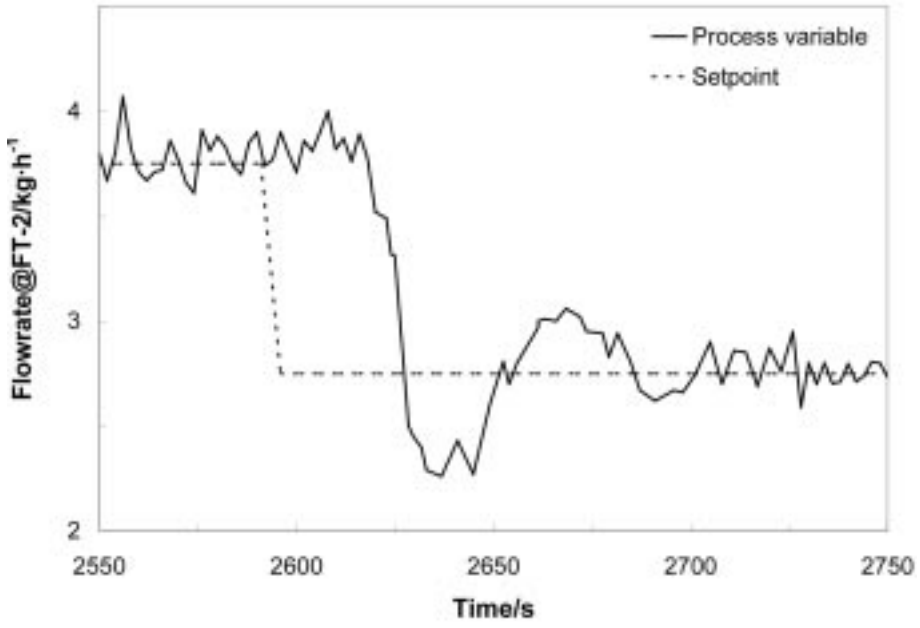

Fig. 7. FT-02 robustness check for a step upset of 3.75 to 2.75 $kg \cdot h^{-1}$, $K_c = 0.4$; $\tau_I = 11$ s.

CONCLUSIONS

The *hands-on* approach is found to be a powerful tool in the teaching of dynamic operation and control. It is used as a fast and effective way to introduce the 'real control problem' where our future engineers learn to handle identification and control design tasks. In the experiment described here, the students have to select the control topology (stand-alone or cascaded), select the appropriate algorithm (P, PI or PID) and tune the parameters (open or closed-loop Ziegler-Nichols). In addition, other no less important topics are addressed such as the severity of alarms or startup and shutdown protocols.

Moreover, the experience gained in this professionally oriented laboratory, in addition to promoting the classical understanding of distillation control, also promotes creative and critical thinking. Simultaneously, the laboratory incorporates aspects that are necessary to achieve the global formation of our future chemical engineers, such as safety, environmental concerns, trouble-shooting or design of procedures. It also includes the no less important *soft* skills used in teamwork, planning and organizing and helps to set the

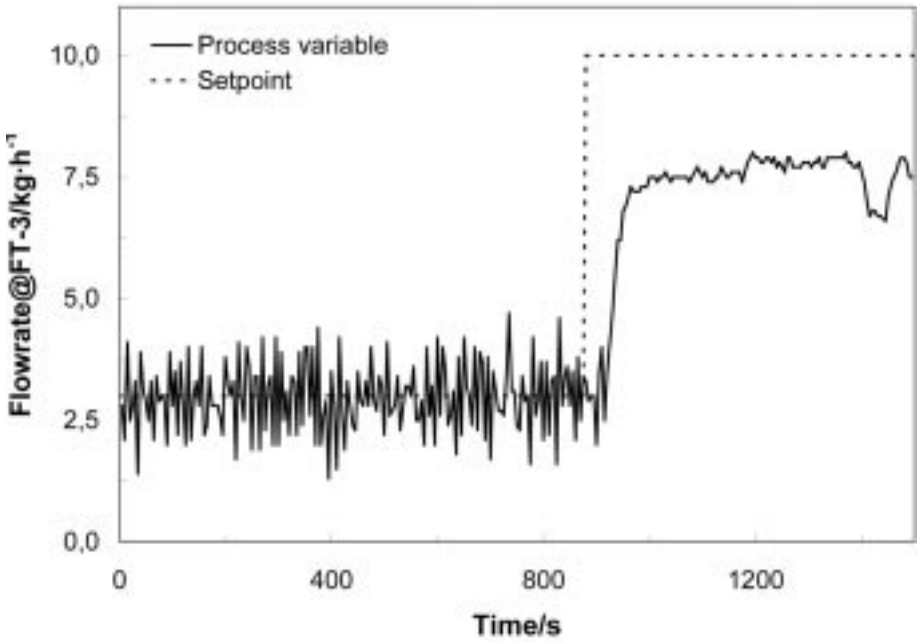

Fig. 8. FIC-03 robustness check for a step upset of 3.0 to 10.0 $kg \cdot h^{-1}$, $K_c = 1.0$.

pattern for our graduates to become successful life-long learners.

The educational objectives of the course were mostly attained. In addition, the course has been very well accepted by our students, testament to which are the many favorable comments that have been received throughout the four years that the laboratory has been running. For instance, one student stated that 'this laboratory was the most interesting that I have ever received, as real industrial equipment are used' or another asserts 'although at the beginning it is difficult to plan the experimental tasks, the lack of information forces us to use our own initiative'. We realise that at the beginning of the experiment, more time is needed than in traditional teaching methodologies, since students often loose their way and need continuous help and guidance from the laboratory instructors'.

Above all, we believe that the approach used here provides a complementary teaching methodology to dynamic operation using computer modeling (in real life, it is not possible to simply *reset* and instead, concrete actions are required to return the equipment to normal operation). These elements can only be reached by way of experience with real pilot plants.

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