

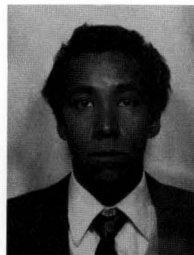
# USE OF A MODERN POLYMERIZATION PILOT-PLANT FOR UNDERGRADUATE CONTROL PROJECTS

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Most chemical engineering processes exhibit considerable deviations from ideality since complex physico-chemical phenomena are involved which are difficult to describe quantitatively. It is not unusual, therefore, that the popular approaches to the design of automatic control systems may result in controllers that fall short of their design specifications and that a significant amount of time must be spent in subsequent implementation and fine-tuning stages.

Many of these "unmodeled" phenomena, which are not likely to be observed in a bench-scale laboratory, may readily manifest themselves in an environment that more closely resembles an industrial-scale operation, such as a pilot-plant set-up. Testing control algorithms in a pilot-plant rather than only at the simulation level can help the control engineer to at least attempt to take into consideration these phenomena and to address important implementation issues before a control system is attempted on-line on the particular application. A substantial reduction of the implementation time, on-line tuning of the controller, and a more suitable control system design may then be possible.

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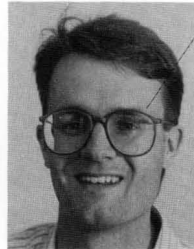


To illustrate some of the general statements made above, the most representative steps of a series of undergraduate control projects are presented in this paper. These control projects are part of the senior undergraduate design course. They can be done on an individual or group (2-3 students) basis and their duration can be between four and eight months.

In these projects, the undergraduates are encouraged to experiment and even to redesign a flexible reactor set-up in a modern computer-controlled polymerization pilot-plant. Our objective at the outset was to "challenge" the students with something more than a simulation exercise. For the purpose of this paper and for the sake of brevity, three popular control strategies, namely, a PID controller, a Smith predictor, and a Dahlin controller (for details see Stephanopoulos<sup>(1)</sup>) are tuned, evaluated, and applied for temperature control of a polymerization pilot-plant reactor. In addition, a commercially available real-time expert system shell was used to code and implement a rule-based PI-type control algorithm.



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**William R. Cluett** received his BSc (1981) from Queen's University, Canada, and his PhD (1986) at the University of Alberta, Canada, both in chemical engineering. He joined the department of chemical engineering at the University of Toronto in 1986. His interests lie in robust control, adaptive control, and applications to industrial processes.

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The design and implementation effort using the expert shell was compared with the one required for the previous control strategies.

These control projects were very beneficial from an educational/training point, both to the students and to the faculty members. Our observations are briefly discussed in the following sections.

## TEMPERATURE CONTROL IN POLYMERIZATION REACTORS

Polymerization reactions are exothermic in nature; the amount of heat released during monomer conversion to polymer is considerable. Temperature variations greatly affect the kinetics of polymerization processes, and through the kinetics they have a strong impact on the way the produced polymer is structured and thus, on its physical properties and quality characteristics. Therefore, control of reactor temperature is critical. If the polymerization temperature is allowed to increase, monomer conversion increases and more polymer is produced. Hence, the polymerizing mixture becomes more viscous and heat removal becomes more difficult. This may easily lead to disastrous reactor runaways. Reactor temperature must, therefore, be kept within the limits that allow one to carry out a safe polymerization, *i.e.*, within the system's heat-removal capabilities<sup>[2-6]</sup>.

## THE PILOT-PLANT REACTOR

Figure 1a shows a schematic of the pilot-plant reactor layout, and Figure 1b is a photograph of the whole set-up. The pilot scale stainless steel reactor consists of a vessel with a volume of 5 l, surrounded by a jacket. Oil is pumped through the jacket to heat or cool the vessel as required. The reactor is mounted on a hydraulic lift stand and has a removable top head which is held stationary while the vessel is lowered or raised on the stand to open or close the reactor. A variable-speed turbine agitator is used for stirring the reactor contents. Auxiliary lines (*e.g.*, vacuum, feed, nitrogen, *etc.*) enter and leave the top of the reactor.

The heat transfer system consists of a water/oil heat exchanger with two 9 kw electric heaters connected in series and a 1 hp circulation pump. One of the two electric heaters is automatically shut off when the temperature of the oil is above 50 C. The oil returning from the jacket is pumped through the heat exchanger. A thermocouple at the exit of the second heater (TT2) measures the delivered oil tem-

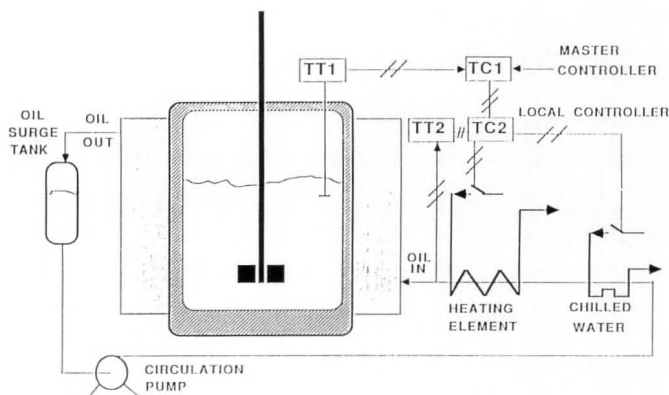


FIGURE 1a. Schematic of the pilot-plant reactor.

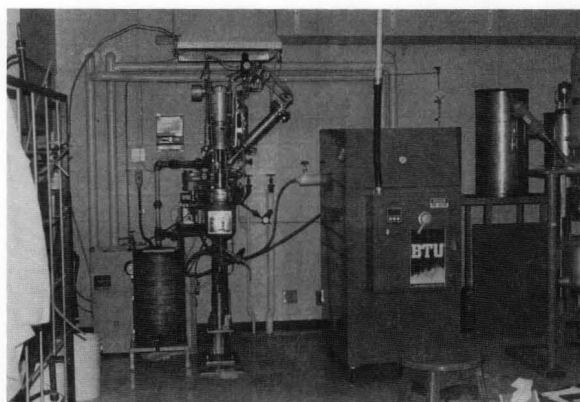


FIGURE 1b. Photograph of the pilot-plant set-up.

perature. This measurement is sent to a local controller (TC2), which turns on or off one or both heaters and opens or closes the solenoid valve that allows the cold water to flow through the heat exchanger. The function of this internal local controller is to maintain the oil delivery temperature at a desired value. From the heat exchange unit, the oil is sent to the reactor's jacket. A by-pass pipe connecting the oil output with the oil input to the unit is provided in order to prevent an excessive pressure buildup from the oil pump.

Three type-J insulated thermocouples constitute the set-up's sensor system. The first (TT1), inserted through the stationary head, measures the temperature of the reactor contents. The other two measure the oil temperature at the jacket inlet (TT2) and outlet (not shown). The thermocouple signals are sent to an IBM PS/2 60 computer via an OPTOMUX OPTO-22 data acquisition system.

The control configuration consists of the local internal controller (TC2) in the heat transfer system connected in cascade with the master control algorithm (TC1) in the process computer. The master

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controller receives the measured reactor's temperature and calculates the value of the oil delivery temperature that will maintain the reactor's temperature as closely as possible to a desired operating point. The calculated value is sent to the heat-transfer system where it becomes the set-point for the internal controller. The internal controller is simply an on/off control system and can be calibrated, activated, or deactivated via the unit's control panel keypad.

## ANALYSIS AND DESIGN OF THE CONTROL SYSTEM

Several monomer systems and modes of reactor operation were tried during the design projects. The tests included "dry runs" with water or solvent, solution polymerization of methyl methacrylate (MMA) with varying fractions of solvent (40%-60%), emulsion terpolymerizations of styrene-butyl acrylate-acrylic acid, "soap-free" emulsion polymerizations of styrene, and low conversion (less than 40%) bulk copolymerizations of styrene-butyl acrylate; the reactor modes employed were batch and semi-batch isothermal and temperature programming cases. For the sake of brevity, the examples that will be used here will be drawn from the solution MMA homopolymerization tests. However, the control algorithms performed in a similar way for all different monomer systems and modes of operation.

To perform the system identification, the reactor was filled with solvent (about 4 l), and its temperature was raised to 50 C. Toluene was used as the solvent. Steps of 20 C were applied to the reactor temperature set-point, and a first order plus dead-time model was fitted to the open-loop response. The identified transfer function follows:

$$G_p(s) = e^{-t_d s} / \tau_p s + 1 \quad (1)$$

with  $t_d = 70$  s, and  $\tau_p = 500$  s.

Based on open-loop dynamics, a sampling time of four seconds was chosen to approximate continuous-time control.

The three algorithms considered are very popular in industry. The Smith predictor, usually applied

in conjunction with PI or PID control, provides dead-time compensation, while the Dahlin controller forces the closed-loop to behave in a desired first-order plus dead-time fashion.

Recently, increasing attention has been given to the use of expert systems for process control applications. In one of the design projects, a commercial expert system shell (RTES) was used to code a simple rule-based PI-type controller for the control problem under consideration. This algorithm included heuristics to compensate for dead-time and for changes in the operating point. The results were compared with those from the Dahlin controller, the Smith predictor, and the conventional PID controller. For all controllers, the performance criteria were minimum oscillation about the reactor's set-point and minimum response time. At steady-state, an error band of 0.5 C was allowed. These criteria were chosen so as to minimize variations in the polymerization rate, which in turn affect conversion (productivity) and product quality.

## EXPERIMENTAL PROCEDURE

A nominal operating point of 50 C was chosen. During the development and tuning stages, the reactor, with about 4 l of solvent, was heated to the operating point. Once steady state was achieved, a change of set-point from 50 C to 70 C was applied. Once the reactor stabilized at the new operating condition, a set-point change from 70 C to 50 C was issued. To test for disturbance rejection, 300 ml of toluene at room temperature was then injected into the reactor. The controllers were fine-tuned by trial and error until the closed-loop responses met the performance specifications.

As a final test, a "real" polymerization was carried out using the different control laws. 1.4 l of MMA, 2.5 l of toluene, and 33 g of initiator were mixed into the reactor. The reactor contents were brought from room temperature to the nominal operating point (50 C), and the polymerization was allowed to proceed for two and a half hours (Stage A). After this period, the reactor's temperature set-point was increased to 60 C and maintained at this value for another hour (Stage B). Finally, 5 g of

initiator (AIBN) dissolved in 300 ml of toluene at room temperature was injected into the reactor, representing a disturbance. The reaction was allowed to continue for one more hour (Stage C), and then the polymerization was stopped and the reactor was cleaned.

## ANALYSIS OF THE RESULTS

The results that follow are representative of an extensive set collected in the pilot-plant during the design projects. The aim is to show how the use of the pilot-plant helped in the design, implementation, tuning, and evaluation of several control algorithms. The results also show the progression in the students' steps/thoughts.

In general, according to the students' observations, the use of the pilot-plant was of great value in mimicking a "real-world" process with respect to:

- the unmodeled dynamics introduced by the use of a first-order plus dead-time model to approximate the process (bearing in mind that the process was a polymerization system which exhibits highly non-linear phenomena, such as a reaction exotherm and an increase in viscosity ["gel effect"] as the reaction proceeds)
- the physical limitations and operational characteristics of the equipment constituting the pilot-plant. This was particularly important for the heat exchange unit whose on/off internal controller and variable time-delay associated with driving the oil to the desired set-point (control manipulation) made the closed-loop system prone to an oscillatory behaviour
- the training point of view. The students had the chance to enlarge their knowledge with typical polymerization processes, systems that are sufficiently complex to present a challenge to the undergraduates, and furthermore, systems largely unstudied in the undergraduate curriculum. Thus, this combination of "undergraduate-level control algorithms" with "complex processes" made the undergraduate design projects much more meaningful.

First, the development and comparative analysis of the controllers for the reactor full of solvent is presented. Next, two of the controllers with the more satisfactory performance are applied to the solution polymerization of MMA. Finally, the results of the rule-based controller are discussed.

Figure 2 shows the closed-loop response of the reactor using the velocity form of a PID controller. Although the controller brings the process to steady state, one can observe an overshoot of approximate 8 C and a settling time of more than one hour.

The control objectives are, therefore, far from being satisfied.

To improve the PID controller performance, upper and lower limits were imposed on the oil temperature set-point, and the internal controller settings in the heat-transfer unit were readjusted following discussions with the manufacturer. The closed-loop response with the new bounds and the finely-tuned PID controller parameters is shown in Figure 3. One can see that the controller stabilizes the process in approximately thirty minutes, with no overshoot. The response was considered satisfactory, and this controller was chosen as the base case for performance evaluation.

Figure 4 shows the step-response from 50 C to 70 C of the Smith predictor and the Dahlin control-

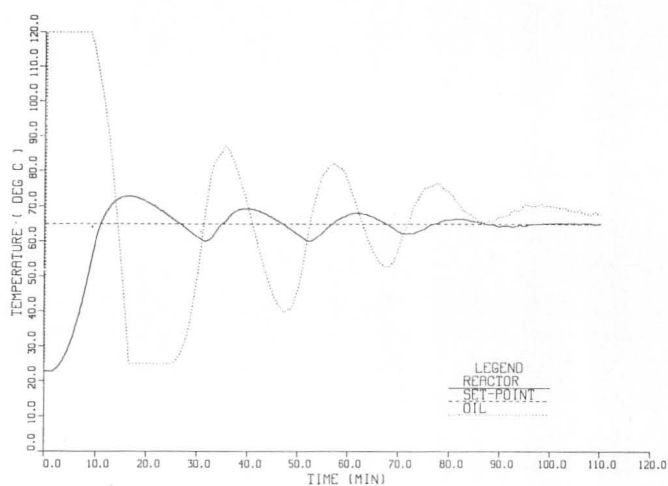


FIGURE 2. Closed-loop Response (Toluene, PID controller).

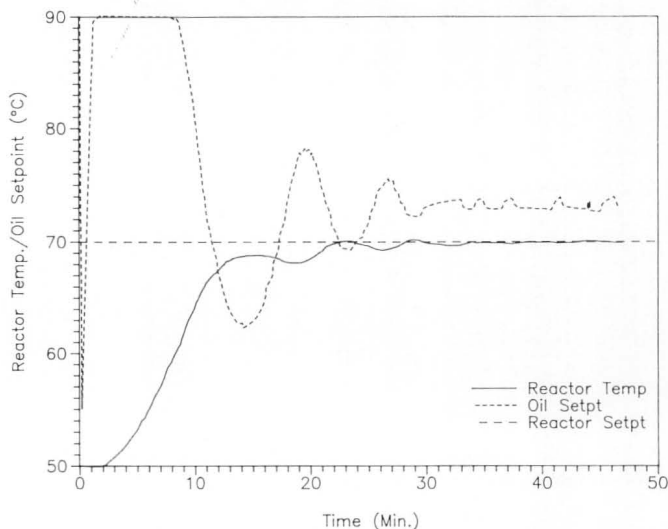


FIGURE 3. Closed-loop response (toluene, bounded finely-tuned PID controller).

ler as compared to the PID. One can see that the Smith predictor gives a smoother step-response than the other two controllers. Figure 5 shows the closed-loop response to a "cold-disturbance" for the three controllers under consideration. In this case, the performance of the Dahlin controller was better than those of the Smith predictor and the PID controller.

Figures 6 and 7 show the reactor temperature record for the solution polymerization of MMA under PID and Smith predictor control, respectively. Although both strategies gave acceptable control, the Smith predictor generated smoother control manipulations, which provided a more acceptable behaviour of the oil temperature variations.

Finally, based on the previous observations, a

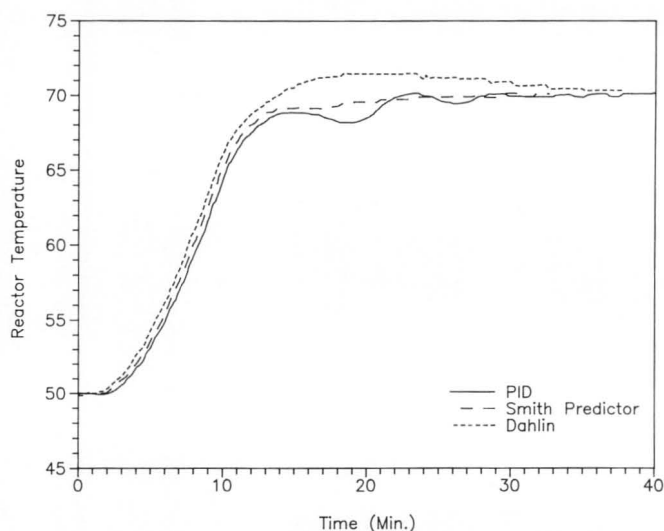


FIGURE 4. Closed-loop response (toluene, PID, Smith predictor and Dahlin controllers).

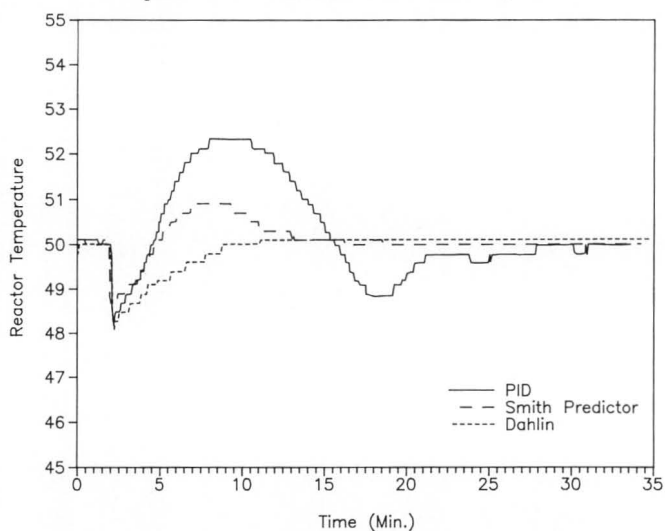


FIGURE 5. Disturbance rejection (toluene, PID, Smith predictor and Dahlin controllers).

rule-based PI-type controller was designed to compensate for the process dead-time and to provide smooth step response and good disturbance rejection. From the previous process experience, three heuristics were defined and implemented:

- No control action until the reactor temperature responds to a previous manipulation within a tolerance band of  $\pm 0.5^\circ\text{C}$  (to compensate for dead-time).
- The initial manipulation to a set-point change in the reactor's temperature is to set the oil set-point equal to the reactor set-point.
- No PI control action until the temperature of the inflowing oil is within a  $1^\circ\text{C}$  band of the oil set-point generated by the control algorithm. This heuristic is used in order to reduce the oscillating behaviour due to the high capacitance of the process (volume of reaction mixture

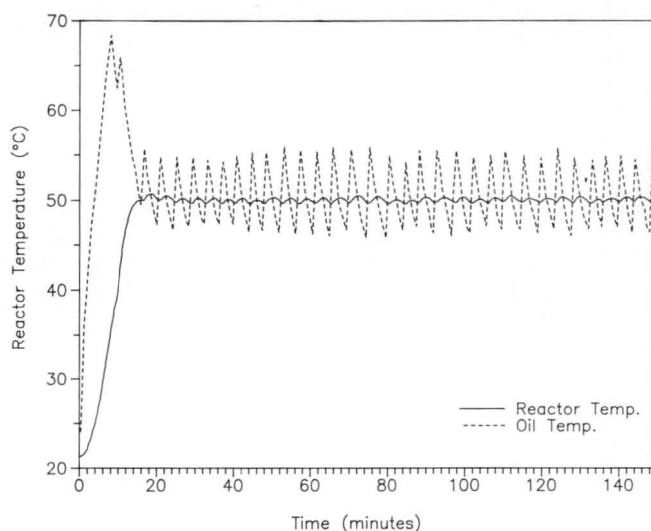


FIGURE 6a. MMA solution polymerization (PID control, stage A).

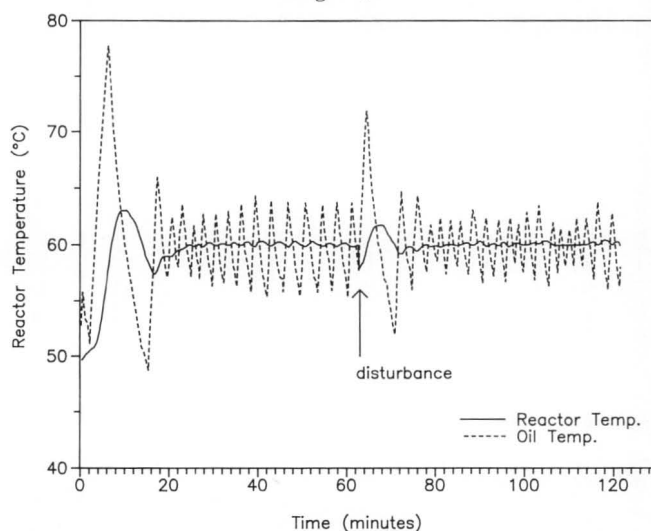


FIGURE 6b. MMA solution polymerization (PID control, stages B and C).

to be heated/cooled) in combination with the process dead-time.

The behaviour of the rule-based controller is shown in Figure 8 for the solvent test. Comparison with Figure 3 reveals that it was possible to approximate the performance of the PID controller with that of the rule-based controller.

## CONCLUDING REMARKS

Working in a modern pilot-plant allowed the students to gain experience in handling large volumes of hazardous materials, process start-up and shut-down, equipment failures, operational variations, scaling-up, equipment cleaning, and run-time scheduling.

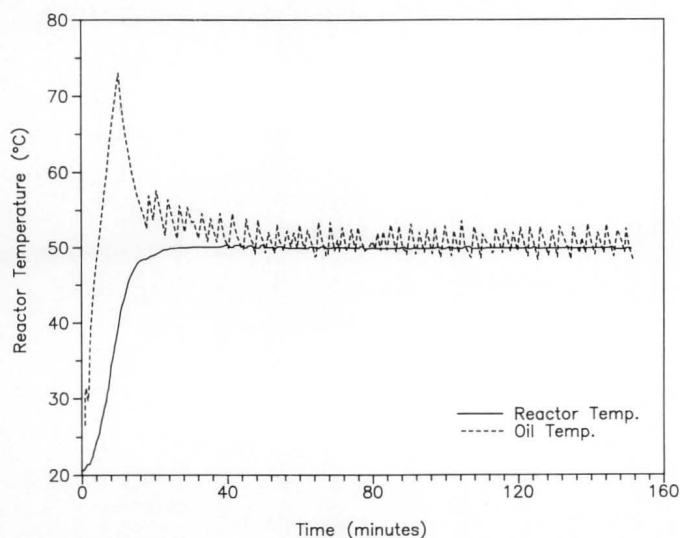


FIGURE 7a. MMA solution polymerization (Smith predictor, Stage A).

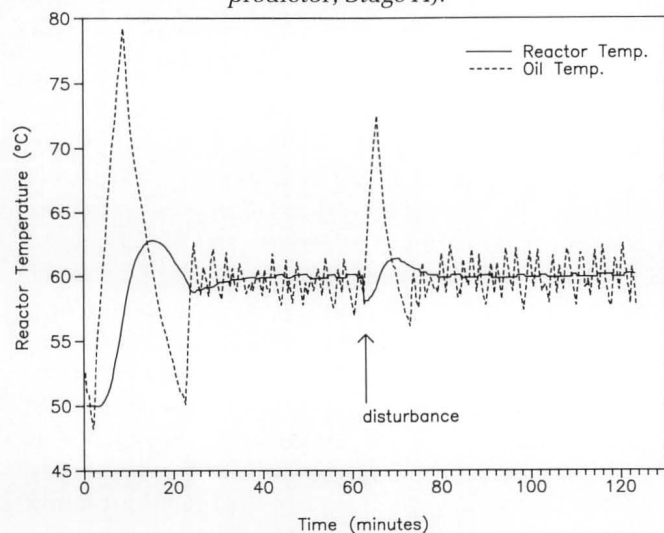


FIGURE 7b. MMA solution polymerization (Smith predictor, Stages B and C).

These complexities not only made the design projects more meaningful from a technical point of view, but also offered the students invaluable experience in another important aspect which is largely untouched in undergraduate education—the aspect of project/group management. By setting more difficult objectives in a virtually open-ended project, and by involving the undergraduates in something outside of their "familiar turf" of departmental laboratories, the whole project became a "group effort." Different groups had to rely on the good or bad performance of other groups in order to accomplish their objectives, a fact that resulted in everyone who was involved being interested in what others were doing. Cooperation with a group, coordination among different groups, and time-planning and management became the key features.

The operation required the participation of many groups of undergraduate students, graduate students, faculty members, technicians, and research engineers, *i.e.*, groups with different levels of expertise. As one group of students put it, "...all of us benefited from this learning experience tremendously. The projects gave us the opportunity to supervise and to be supervised, to plan and to organize, and to work together in a large team...." And another group put it, "...after these projects we appreciated much better what one means by systems in series or in parallel, interacting or non-interacting, and by the rate-controlling step...."

Currently, we are trying to introduce new projects involving the implementation of more advanced control techniques, such as minimum variance (constrained and unconstrained), predictive and/or adaptive. Continued on page 60.

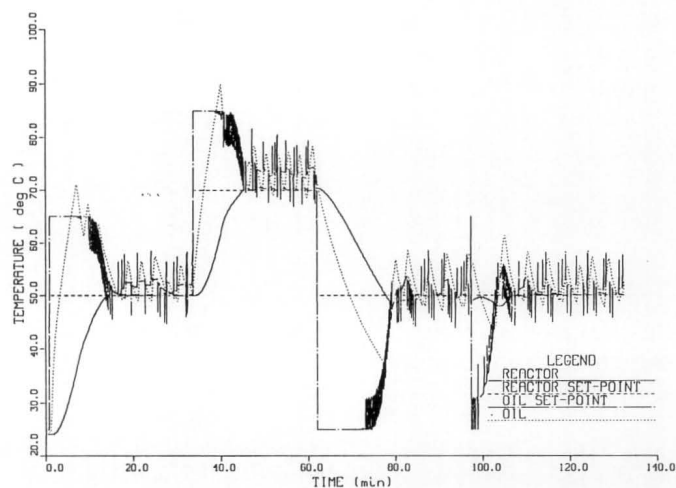


FIGURE 8. Closed-loop response (Toluene, bounded rule-based controller).

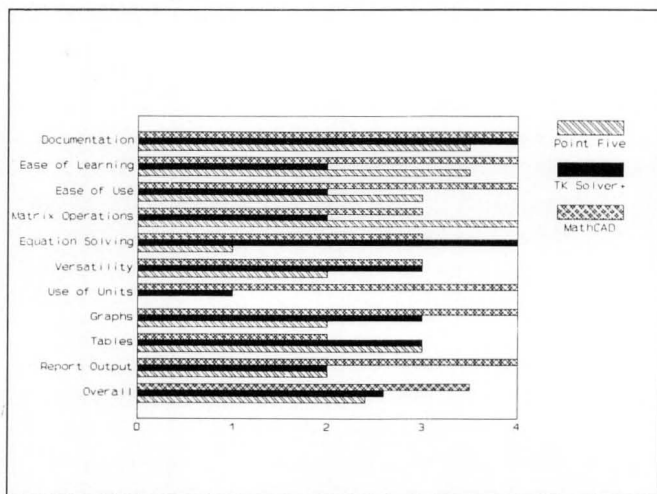


Figure 9

**FIGURE 9.** Comparison of Point Five, TK Solver Plus, and MathCAD. Scale: 0=unsatisfactory, 1=poor, 2=fair, 3=good, 4=excellent.

ability of its reports. We were able to efficiently perform matrix operations, create user functions, and solve series of simultaneous equations, without having to know many programming techniques. We enjoyed the feature of creating and editing graphs without having to leave the scratchpad every time we wanted to examine a plot – a feature especially helpful when trying to find roots. But one of the most impressive features is that the printed results from MathCAD are very readable. The free-format use of units and graphic abilities make it easier for the user and non-user to read and understand a MathCAD file.

In an attempt to give some impression of the individual strengths and weaknesses of these various packages, we rated them on the accompanying documentation, ease of learning, ease of use, matrix operations, equation solving capability, versatility, use of units, generation of graphs, generation of tables, readability of output, and overall impression on a scale from 0 (unsatisfactory) to 4 (excellent). While each of the programs that we evaluated has particular strengths and weaknesses, as indicated in Figure 9, MathCAD was our selection as the program best suited for general use in the chemical engineering curriculum.

#### ACKNOWLEDGEMENT

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#### REFERENCES

1. MathCAD, MathSoft, Inc., One Kendall Square, Building 200, Cambridge, MA 02139
2. Point Five, Pacific Coast Software Inc., 887 NW Grant Ave., Corvallis, OR 97330
3. TK Solver Plus, Universal Technical Systems, Inc., 1220

- Rock Street, Rockford, IL 61101
4. Matlab, The Math Works, Inc., 20 N. Main Street, Suite 250, Sherborn, MA 01770
5. GAUSS, Aptech Systems, Inc., 26250 196th Place SE, Kent, WA 98042
6. Macsyma, Symbolics Inc., 8 New England Executive Park East, Burlington, MA 01803
7. Eureka, Borland International, 4585 Scotts Valley Drive, Scotts Valley, CA 95066
8. FORMULA/ONE, Soft-Sense, 12 Rockaway Lane, Arlington, MA 02174
9. Shacham, M., and M.B. Cutlip, *Chem. Eng. Ed.*, **22**(1), 18 (1988)
10. Heller, M., *Personal Engineering & Instrumentation News*, **5**(9), 39 (Sept. 1988)
11. Smith, A.L., *Academic Computing*, **2**(3), 36 (1987)
12. Himmelblau, D.M., *Basic Principles and Calculations in Chemical Engineering*, 4th ed., Prentice-Hall, Inc., Englewood Cliffs, NJ, p. 591 (1982)
13. McCabe, W.L., J.C. Smith, and P. Harriott, *Unit Operations of Chemical Engineering*, McGraw-Hill Book Company, New York, p. 85 (1985)
14. Fogler, H.S., *Elements of Chemical Reaction Engineering*, Prentice-Hall, Inc., Englewood Cliffs, NJ, p. 164 (1986)
15. Fogler, H.S., *Elements of Chemical Reaction Engineering*, Prentice-Hall, Inc., Englewood Cliffs, NJ, p. 572 (1986)
16. Edgar, T.F., and D.M. Himmelblau, *Optimization of Chemical Processes*, McGraw-Hill Book Company, New York, p. 208 (1988) □

## POLYMERIZATION PILOT-PLANT

Continued from page 39.

tive. Our efforts reflect our belief that it is time to introduce more sophisticated control algorithms (which, at present, are usually encountered at the graduate-student level) at the undergraduate level, combined with more complicated real processes.

#### ACKNOWLEDGEMENTS

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#### REFERENCES

1. Stephanopoulos, G., *Chemical Process Control: An Introduction to Theory and Practice*, Prentice Hall, New Jersey (1984)
2. Hoogendoorn, K., and R. Shaw, "Control of Polymerization Processes," *Proc. IFAC PRP-4 Automation*, Chent Belgium, Pergamon Press, p. 623 (1980)
3. MacGregor, J.F., A. Penlidis, and A.E. Hamielec, "Control of Polymerization Reactors: A Review," *Poly. Proc. Eng.*, **2**, 179 (1984)
4. Richards, J.R., and P.D. Schnelle, Jr., "Perspectives on Industrial Reactor Control," *Chem. Eng. Prog.*, **84**, 32 (1988)
5. Davidson, R.S., "An Intelligent Temperature Controller for Jacketed Reactors," *Am. Cont. Conf.*, **2**, 1380; *CEP*, **82**, 18 (1986)
6. Kern, A.G., "Simplify Batch Temperature Control," *Chem. Eng.*, pg 61, March 28 (1988) □