Integration of Hardware and Software in a High-Performance Laboratory Reactor

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SYNOPSIS

The assembly of a stirred tank reactor capable of precise reaction temperature control is described. The unit allows easy on-line determination of the thermal effect and calculation of heat transfer coefficients and heat capacities. Construction elements are standard, low cost, and high quality. Temperature control is achieved by a heating-cooling loop integrated to the reactor jacket and a cascade control procedure. An important feature of the equipment is the integration of the data acquisition system with the supervisory control system that allows the capability of an advanced strategy for process control and data acquisition. The capability of the reaction system is demonstrated for the microemulsion polymerization of vinyl acetate, by the use of batch and isothermal operational modes. The course of the reaction is followed on-line by the temperature variation of the jacket. Procedures are described for the calibration of the unit. The reactor developed is versatile, is easy to configure and extend, is low cost, and can compete advantageously with commercial reactor units such as the Mettler RC1. © 1996 John Wiley & Sons, Inc.

INTRODUCTION

An important category within the study of reacting systems is data acquisition for the scale-up and design of the industrial unit.[1-3] The typical job includes testing different reaction mechanisms, determination of kinetic parameters, heats of reaction, heat transfer coefficients, operational policies, etc. The data acquired must be of acceptable precision so that the study must include the appropriate strategy for instrumentation, data acquisition, and control of the operation of the reactor. For a stirred tank reactor, it is possible to integrate a complete system whose mechanical components (reactor, heat exchanger, and mixer), hardware, and software are of standard production and low cost, allowing highly flexible operation modes, instrumentation, and control. This communication describes the building of such a unit and illustrates its operation with the microemulsion polymerization of vinyl acetate.[4-6]

BACKGROUND

The instrumentation and control of chemical reactors have been the subjects of many studies.[7-9] On the subject of temperature control, in particular, general techniques have been developed for achieving a very precise control of the parameter, as well as providing information on the reaction path when significant heat effects are present. Two of the most common techniques are the use of a heating-cooling loop for the heat exchange and cascade control of the reactor temperature.

In the heating-cooling loop, the heat transfer medium (for heating or cooling) is recirculated in a closed-loop fashion. Compared with the nonrecirculating procedure, the heating-cooling loop has the following advantages:

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1. High flow through the jacket, keeping the temperature of the heating-cooling medium more uniform and eliminating the hot/cold spots at the entrance and exit of the fluid. In addition, a high flow favors the heat transfer coefficient.
2. Constant flow rate, eliminating thus the variation in the time constant of the heating medium and the subsequent modification of the parameters of the temperature controller when the heating-cooling load changes.
3. There is no discontinuity in the transition from heating to cooling, leading to a more precise control and simplification of the heat balance calculations.

The cascade control method consists of two temperature controllers operating in series. The principal (master) loop controls the reactor temperature, fixing the set point to the secondary (slave) loop, which controls the jacket temperature by means of the cooler and the heater. This method has several advantages over the direct mode (the reactor temperature controller regulating directly the heat transfer equipment):

1. The control of the jacket temperature corrects the perturbations that occur in the heating-cooling loop without affecting the reactor temperature. Such perturbations may be due to changes in the temperature of the surroundings and/or the cooling medium, changes in the flow, and poor operation of the end actuators.
2. Faster response.
3. The nonlinear relationship between temperature and heating medium flow is transferred from the control loop of the reactor temperature to the slave loop, where it is less critical.

The data acquisition and control strategy may be carried out in different ways; nevertheless, a very common industrial procedure is the coupling of the data acquisition and control system with the supervisory control system, as exemplified by the distributed control system. In this arrangement, the sensors and actuators are connected directly to the data acquisition and control system, which is a specialized microcomputer that carries on the functions of signal conditioning, their digitization, and logic and control function calculations. In this way, multiple conventional controllers are substituted by one unit capable of performing direct control by the use of powerful algorithms and strategies. The supervisory system constitutes the interphase between the operator and the process (through the data acquisition and control system). Its principal functions are:

1. Interphasing the operator with the process. The operator interacts with the process by changing the state of the controllers, modifying parameters, activating operation sequences, observing the state of the variables by means of graphs or mimics, etc.
2. Utilization of personal computer (PC) resources: hard disk variable recording, printing reports, execution of supervisory control routines, etc.
3. Communication with one or more data acquisition and control systems.
4. Ability to carry direct control as well as simulations [using standard algorithms such as proportional–integral–derivative (PID) and its variations or nonconventional algorithms].

In spite of its clear advantages, the arrangement described is not used commonly in laboratory reactors and it seems appropriate to spread the information about its applications. The capabilities of the reactor will be illustrated with the microemulsion polymerization of vinyl acetate, which is presently being investigated in our laboratories.[6] Part of the experimental program relates to the batch isothermal polymerization mode, where the reagents' concentration and temperature are modified. We will describe here only the operational features of the reactor and the results of the calibration methods.

SYSTEM DESCRIPTION

Figure 1 describes the general setup. The main elements of the equipment are as follows.

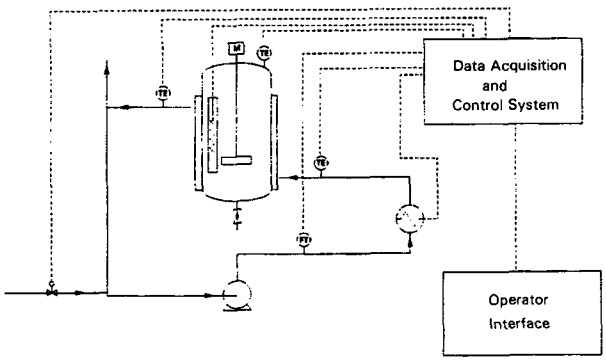

Figure 1 Instrumentation and control of the reactor.

Reactor

The reactor is a cylindrical glass vessel of 100, 150, or 1,000 mL capacity, provided with a jacket for heating or cooling (Kontes cylindrical reaction flask). The cover has three openings for connection to other subsystems, introduction of sensors, input and output of materials, and agitator shaft. The temperature inside the reactor is measured with a type T thermocouple. A low-power (50 W) resistance cartridge is provided for calibration purposes (measurement of heat transfer coefficients and heat capacity).

Agitation

Two exchangeable units are provided, one of high torque (GKH, model HS240) and the other of low torque (Yamato Scientific, LABO stirrer). Both have manual adjustment of the reference velocity and internal control of the same. It is also possible to provide magnetic stirring plates to the 100 and 150 mL reactors.

Heating and Cooling Loop

The heating and cooling loop is made of standard carbon steel pipe, and the heat transfer medium (tap water) is circulated by means of a recirculating pump. Heating is provided by three resistors, each of 1,000 W capacity and activated by electromechanical relays. A solenoid valve allows water flow into the loop during cooling. The temperature of the water entering and leaving the jacket is measured with type *T* thermocouples. The recycle line is provided with a turbine-type flow transmitter (George Fisher, model Signet 8500).

Data Acquisition and Control System

The data acquisition and control system is based on a microprocessor (Analog Devices, model uMAC1060) with a capacity of 32 I/O analog and 24 I/O digital signals. The hardware is a microprocessor (Intel 80188) with 128K main memory, A/D and D/A convertors, and subsystems for conditioning the analog and digital signals. The software is a real-time operating system, *C* compiler, and subroutine library for configuration, data acquisition, control, and mathematical operations. The processor executes a user program where the input is updated, optional calculations are performed (such as calibration and control routines), and the output is updated. An important task is communication with the supervisory system that takes place by interrupts dictated by the latter.

Supervisory System

The supervisory system is composed of a commercial software package (Iconics, GENESIS) executed in a PC. The package is composed of a configurator of control strategies and an executive system that performs the strategy during the operation of the reactor.

Our reactor has important advantages over similar commercial apparatus, such as Mettler's RC1: (1) it is easily adaptable to reactor size; (2) it can operate in continuous mode; (3) there is complete freedom regarding data acquisition and control strategies; and (4) the operator's interface is richer and more expressive and can be designed by the operator himself.

EXPERIMENTAL METHODS

The reactor is capable of various modes of operation with respect to the temperature control: isothermal, adiabatic, programmed profile, etc. However, we will only illustrate the isothermal operation.

Isothermal Operation of the Reactor

A cascade control strategy was utilized, as illustrated in Figure 2, where the master loop was the control of the reactor temperature, which actuated on the jacket temperature controller. The slave loop was the control of the jacket temperature, which regulated heat input when the actuation was larger than 50% or cooling water input when the actuation was less than 50%. An algorithm, executed by the supervisory system, converts the signal from continuous to discrete, as required by the relays and the solenoid valve. The supervisory system also executed the algorithms of the cascade control (master and slave loops). Control tuning was realized with system simulations and the ultimate gain method,[10]

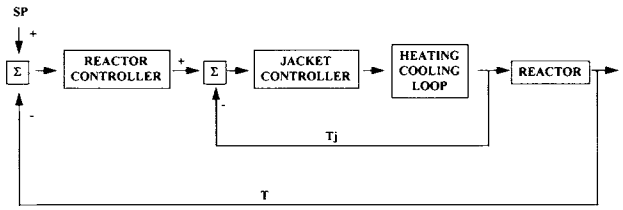

Figure 2 Cascade control arrangement.

which consists of a test where the gain of the controller is increased just until the system becomes oscillatory in response to a perturbation-like addition of cold solution.

Calculation of Heat Transfer Coefficients

In the absence of chemical reaction, the energy balance is (Table I):

$$U_R A_R (T_j - T) + Q_c - Q_P = d(m_s c_{ps} T)/dt \quad (1)$$

During isothermal operation, two states are possible, depending on whether the calibration cartridge is on or off:

$$U_R A_R (T_j - T) - Q_P = 0 \quad (2)$$

$$U_R A_R (T_{jcal} - T) + Q_c - Q_P = 0 \quad (3)$$

Eliminating Q_P from eqs. (2) and (3), we get

$$U_R A_R = Q_c/(T_j - T_{jcal}) \quad (4)$$

The data obtained in this type of test also permit the calculation of the $U_c A_c$ value of the heating-cooling loop, which will be useful for simulation studies of the system. For the latter, the energy balance of the loop gives

$$P - U_C A_C (T_j - T_{surr}) - U_R A_R (T_j - T) = 0 \quad (5)$$

$$P_{cal} - U_C A_C (T_{jcal} - T_{surr}) - U_R A_R (T_{jcal} - T) = 0 \quad (6)$$

$$U_C A_C = [(P_{cal} - P)/(T_{jcal} - T)] - U_R A_R \quad (7)$$

Calculation of the Heat Capacity of the System

If we assume that the initial temperature of the reactor is different from that of the stationary state and that the jacket temperature, the heat losses Q_p, and the value of $m_s c_{Ps}$ are constant, eq. (1) yields

$$T = T_{ss} + (T_0 - T_{ss}) \exp(-t/\tau) \quad (8)$$

where $\tau = m_s c_{Ps}/U_R A_R$. A simple method for determining the value of τ is to determine the response time needed for attaining 63.2% of the stationary value.[11] Substitution of the value of $U_R A_R$ calculated previously from eq. (4) yields the value of the heat capacity of the system.

Table I Nomenclature

A	Heat transfer area
c_P	Heat capacity
m	Mass
P	Power delivered by the heater
Q_P	Heat losses
Q_c	Energy delivered by the calibration cartridge
S_P	Set point
Σ	Sum of signals
T	Reactor temperature
t	Time
τ	Time constant
U	Overall heat transfer coefficient
Indices	
0	Initial conditions
c	Heating-cooling circuit
cal	Value of the variable when the cartridge is active
j	Jacket
R	Reactor
s	System and its contents
ss	Stationary state
surr	Surroundings

Polymerization

The aqueous reaction mixture contained 4 wt % of vinyl acetate (Aldrich), initiator (V50; Wako, 0.3 wt % of the vinyl acetate weight), and surfactant [dodecyltrimethylammonium bromide (DTAB), Aldrich; in a 5 : 95 weight : volume ratio with water]. The total reaction volume was 100 mL, operation was at 333.15 K, and agitation was provided by a magnetic stirrer. Previous to reaction, the vinyl acetate (4 g) was distilled and purged with argon to eliminate dissolved oxygen. The surfactant micelle solution was prepared by adding to the reactor 95 mL of water and the required amount of DTAB, under mixing. Heating was started, and when the desired temperature was reached, agitation was suspended momentarily while the reactor contents were sparged with argon to eliminate oxygen. Vinyl acetate was added, followed by the required weight of initiator (0.12 g). Samples were taken periodically to determine the extent of reaction. The total time of reaction was 60 min.

RESULTS AND DISCUSSION

Isothermal Operation

First, a run was made to tune up the control loops by use of the ultimate gain method, as described in

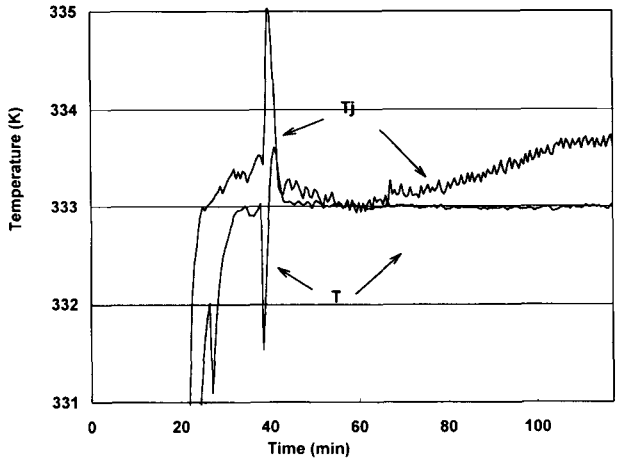

Figure 3 Temperature evolution in a typical polymerization run.

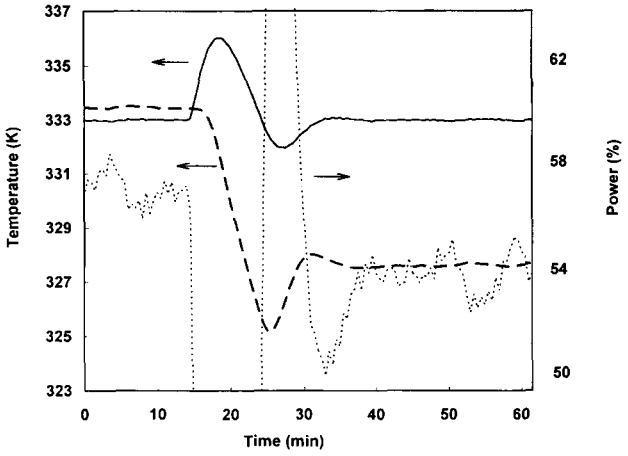

Figure 4 Responses in a calibration test; (straight line) T, (dashed line) T_j, (dotted line) P.

the Experimental Methods section. The perturbation was considered to be the addition of reagents. The parameters obtained for the proportional band of the slave and master loops were 8 and 55%, respectively, and 0 and 1.1 min^{-1} for the integral effect. Figure 3 describes the variation of reaction temperature with time; it is observed that during the course of the reaction, this parameter remains within a band of ±0.05 K. The fluctuations during the first 5 min are due to the control response to the addition of initiator. From this figure, we can also appreciate the way the system is detecting the heat released by the reaction: at the beginning, there is a period of low heat generation (jacket temperature relatively high), then comes a period of maximum heat release (jacket temperature reaches its minimum level), followed by a stage of asymptotic approach to zero heat generation. In general terms, this is the behavior observed during this type of polymerization.[4-6] The method also allows in-line tracking of the reaction, by integrating the heat generated. This possibility is being studied for implementation in the following stage of development of the reactor.

Determination of Heat Transfer Coefficients

Figure 4 shows the response of the system to a calibration run. Substituting the values of the stationary state (T_j = 333.60 K, T_{jcal} = 327.70 K, T = 333.60 K, P = 138 W, P_{cal} = 84 W, Q_c = 25 W) in eqs. (4) and (7), we obtain U_RA_R = 4.24 W K^{-1} and U_CA_C = 4.91 W K^{-1}. These values are of the same order of magnitude as the ones estimated from tabulated data[12] for the heat transfer coefficients in reactors and the Nusselt correlation for free convection[13] for the heat transfer coefficient in the heating-cooling loop: U_RA_R = 2.8 W K^{-1} and U_CA_C = 3 W K^{-1}.

Determination of the Heat Capacity of the System

Figure 5 describes a run for the determination of the value of mc_P of the system. Application of the graphical method described previously gives $t_{63.2\%}$ = $t_{330.56\,K}$ = τ = 2.1 min, so that after substitution of the value of U_RA_R, m_sc_{Ps} = 534 J · K^{-1}. This value is similar to the corresponding one of the contents alone, which for this run is known precisely (417 J K^{-1}).

CONCLUSIONS

The experimental runs prove that the equipment is well provided for controlling the temperature during

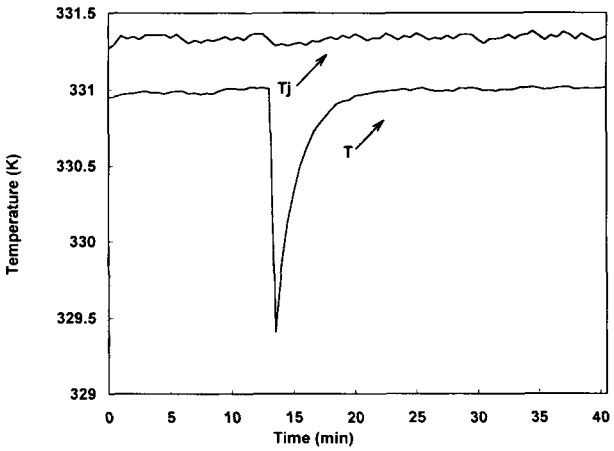

Figure 5 Response at initial conditions.

the microemulsion polymerization of vinyl acetate. In addition, it provides in-line reaction follow-up, as well as the acquisition of basic data for the design and scale-up of the reactor (heat transfer coefficients and heat capacity of the system).

The description of the construction elements demonstrates that it is possible to integrate a laboratory reactor of high performance and versatility which can be adapted to different operating modes (batch, semicontinuous, and continuous), different capacities and agitation regimes, and different strategies for data acquisition and control. Temperature control is very precise; in isothermal operation, the temperature variations are smaller than the ones usually allowed (± 0.1 K). The control system is easy to configure; it requires conventional programming, but most of its configuration is based on the selection of algorithmic blocks containing a variety of preprogrammed functions. The reactor can be easily expanded; there is no limit to the number of sensors and actuators that can be added. In addition, and very important, it is inexpensive to build: the cost of commercially available units for similar purposes (e.g., Mettler RC1) starts at about $200,000, whereas the one described here can be implemented at a cost of $15,000.

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