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CONSTRUCTION AND USE OF COMPUTER CONTROLLED LABORATORY BATCH REACTOR

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Abstract

Two connected computer controlled batch model reactor of flexible hardware and software system have been developed. The system supports the education and the development of chemical technologies, being appropriate for controlling and simulating a wide range of processes as well as for measurement of the heat effects. Model and acid-base experiments were carried out for testing the operation of the systems.

Keywords: laboratory reactor, process control, reaction calorimeter, simulation of acid-base reaction.

1. Introduction

The computer controlled batch reactors are increasingly applied in chemical technologies, especially for multi-purpose plants, due to better product quality, reproducibility, and safety achieved by this way. Some examples:

A novel strategy for molecular weight distribution control of polyethylene terephthalate during the polymerisation process has been successfully implemented by WANG (1993) in an industrial polyester plant.

BASILIA – CINAR (1992) described the development and performance of a model object based expert control system that provides fault-tolerant control of a fixed-bed CO oxidation reactor.

A mathematical model of a polymerisation process in the batch reactor is derived by ADACHI (1993) considering reaction rate, mass and energy balances. Kinetic reaction parameters are estimated from experimental data and parameters of energy balances are obtained from reactor operating conditions.

The higher level utilisation of the advantages offered by the controlled industrial reactors, however, is hindered by the lack of appropriate model equipment. The research and development engineers elaborate the new technologies using the conventional laboratory tools that are not suitable for modeling the wider operating flexibility of controlled industrial systems.

The reaction calorimeters available on laboratory scale are generally also not quite appropriate for complex (chemical and control) development of technologies and for the transfer of the controller program from laboratory to industrial level because their software is equipment specific and not flexible enough for elaborating new model based algorithms.

The aim of this work was the development of a new system combining the benefits of reaction calorimeters and industrial controlled reactors using reactors of higher flexibility than the conventional systems or reaction calorimeters. It needed the consideration of the following principles:

- The equipment must be able to make as many different products as possible, and the process control systems must accommodate changes quickly and easily.
- The user must be able to configure and modify the system, develop the necessary product-specific programs without relying on a computer specialist.
- A hierarchy of software (and hardware structure) is the basis of such a system.
- The development of a 'library' of pre-programmed basic operations, called 'Grund Operationen' (GOs) is recommended. For each GO, there may be several versions. No matter what product a GO is being used to manufacture, it generally performs similar steps in a comparable sequence. These include: dosing, mixing, heating, cooling, distilling, neutralizing, etc. Parameters and equipment specific values are not assigned, so the standardized GOs are not bound to any particular product or process, as it has been applied by MASSEY (1990).

Such a system could serve educational and research purposes as well.

This paper summarizes the hardware and software developments of the system and testing of its operation, performing simple processes.

2. Experimental

The communication between the computer and controller units is ensured by an UAM-512 I/O type card. The card contains an ADC (analogue-digital converter), of 12 bit, a 8255 type PIO chip, latched input port of 8 bit, simple 4 bit input and 8 bit output ports. The 16-channel of ADC receives the signals in range of $-5\ldots+5$ V and converts them to 8 or 12 bit numbers. A HANDLER program belongs to the card, adjusting the A/D measurement to the software. At running this program checks the hardware, produces the display of the current settings, it is resident in the memory if no failure is estimated. The HANDLER is available for the other programs through a software-interrupt (in normal cases it is: $60).

Pump feeder: Peripump D 5187 type (Kutesz Ltd., Hungary) peristaltic pumps have been used. They are controlled by a 6 bit digital port with a D/A interface containing CMOS units.

Valve controlled feeder: As two position feeders 4933001000 type magnetic valves, produced by Radelkis (Hungary), have been used, the working voltage of them: 24 V.

A Radelkis (Hungary) equipment has been used with a Radelkis OP-0808P type glass electrode for pH measurements. The communication with the computer is solved through an output containing 15 digital line. The data channels of pH and level meters get to the computer through a multiplexer.

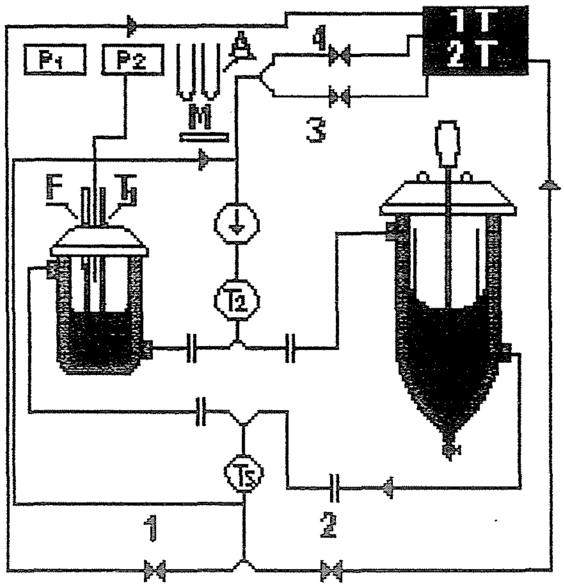

Fig. 1. Reactors and auxiliary tools

$1T$	Cooling thermo-stat (min. −15°C)	p_1	Peristaltic pump 1
$2T$	Warming thermo-stat (max. 120°C)	p_2	Peristaltic pump 2
T_1	Reactor thermometer	1, 2, 3, 4	Magnetic valves
T_2	Input thermometer	A	Feeders
T_5	Output thermometer	F	Electrical heater
M	Sartorius balance	−‖−	Heat-exchanger circuit valve

A Sartorius digital balance is working independently from the A/D card. The communication with the computer is realized through the standard line adapter (COM2). The asynchronous line driver used for data transfer is working with double (input and output) buffer. The baud rate: 2400 baud, which is low, but safe data exchange can be ensured in this way. The characters representing the weighted mass arrive to the computer with 1 s frequency. This sampling rate meets the adjustment rate of the balance.

The system is shown in *Fig. 1.*

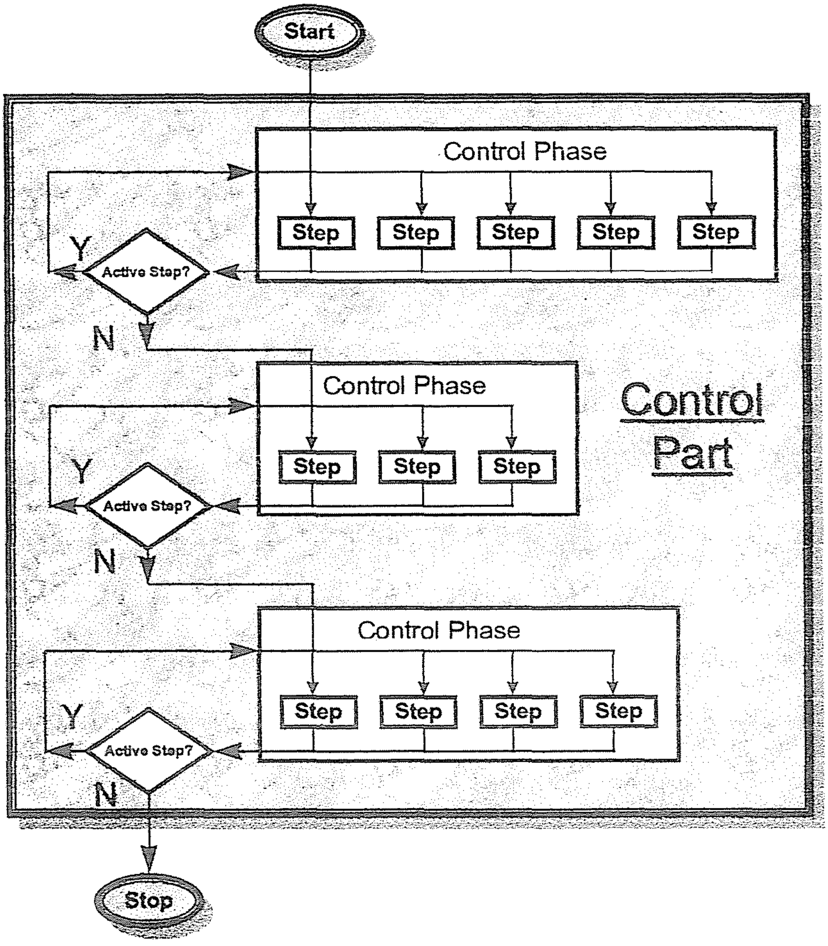

Fig. 2. The structure of recipes

Glass reactors are used, that allow visual process checking. Two reactors can be operated simultaneously or separately. Their volumes are 1500 and 300 cm^3 and maximal surfaces of heat exchange are 0.075 and 0.024 m^2 respectively. The common heat-exchanger circuit of the system can be applied for controlling reactions at separate or integrated operation of the reactors depending on setting of the heat-exchanger circuit valve. The electrical heater is generally used for the determination of the coefficients of specific heat. Controlled, independent feeding of four components can be accomplished.

3. Results and Discussion

An interactive, menu-driven program has been developed written in Pascal language using the object oriented technique of Turbo Vision program package. The structure of recipes is shown in *Fig. 2.*

The structure of a recipe is divided into three levels:

- Control Step
- Control Phase
- Control Part

Control Instructions can be given at *Control Step* level. *Control Steps* are operating in parallel, while the *Control Phases* follow each other in sequential order. The *Control Phases* (including the GOs) can be assorted to *Control Part.* The recipe consists of a series of *Control Parts.*

During running the working parameters (reference signals, settings, etc.) can be modified at any time. Once all the functions for a particular phase are performed, the batch moves to the next phase.

Heat balance is used for calculating the enthalpies of reactions or in course of simulation of the process. The heat balance of the system ($\sum Q[W]$) comprises the following terms:

$$Q_{flow} + Q_{accu} + Q_{dos} + Q_r + Q_{refl} + Q_{loss} + Q_{add} = 0,$$

where

Q_{flow}	heat flow through the reactor wall,
Q_{accu}	heat accumulation,
Q_{dos}	heat flow due to dosing of reactants,
Q_r	heat production of chemical reactions,
Q_{refl}	heat flow of reflux condenser,
Q_{loss}	heat losses due to dissipation,
Q_{add}	any other, additional heat flow.

For special cases the reactor can be isolated, in that case the Q_{loss} can be neglected, but in general cases considerable heat loss occurs. At lower temperature the heat loss can be calculated using a linear approximation. For heat loss measurement the temperature is kept at the required temperature at equilibrium state and the jacket and reactor temperature is registered.

The heat transfer from the jacket to the reactor is given by the equation:

$$Q_{flow} = kA\left(T_j - T_r\right),$$

where

k heat transfer coefficient [W/m^2 °C],
A heat exchange area [m^2],
T_j jacket temperature [°C],
T_r temperature of the reaction mass [°C].

The heat loss:

$$Q_{loss} = \alpha_v (T_r - T_{amb}),$$

where

Q_{loss} heat loss [W],
α_v heat loss coefficient [W/K],
T_{amb} ambient temperature [°C].

The resulting function:

$$\alpha_v = kA \frac{(T_j - T_r)}{(T_r - T_{amb})} = kA \tan\gamma,$$

where $\tan\gamma$ = the slope of the curve, in present case the $\tan\gamma = 0.0255$ (*Fig. 3*).

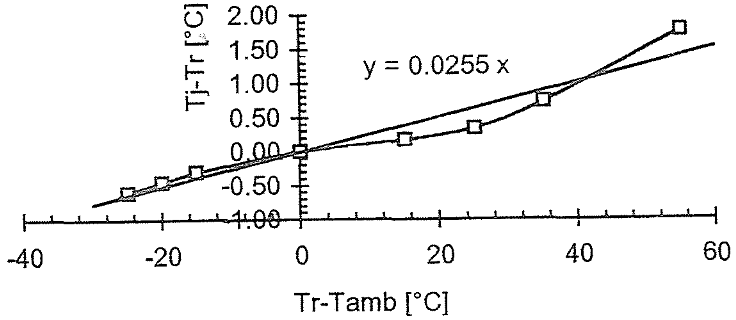

Fig. 3. Linear approximation of heat loss

Near to the boiling point of the solvent of the reaction, the heat loss is much higher due to the reflux on the wall, so it must be determined experimentally for each case. The heat and material balance of a reaction are calculated simultaneously with the control parameters, which can be utilized in two ways:

- the enthalpy of the process can be calculated and
- simulation can be run parallel with the process.

As a simple model process for the first function, the heat effect of melting of ice has been measured. The results of this model experiment are given in *Fig. 4*. The reactor was filled with 500 cm^3 water and an equilibrium temperature of 30°C was set by the computer. The amount of 100 g ice was put two times into the reactor and the melting process determined.

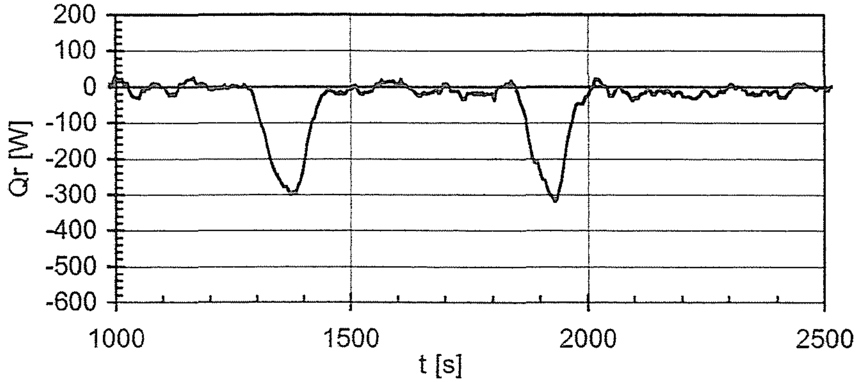

Fig. 4. Enthalpy curves calculated simultaneously with the control process

The determined value of enthalpy showed good accuracy.

A model process for testing the simulation possibilities of an exothermic reaction could be performed using the electrical heater tool. The results of this model experiment are given in *Figs 5* and *6.* The reactor was filled with 200 cm^3 water and an equilibrium temperature of 25°C was set by the computer. The heater was switched on and an amount of 25.63 W heat was transferred into the reactor under continuous temperature control.

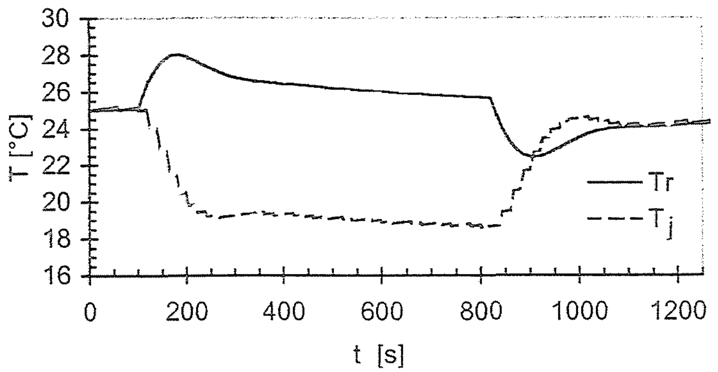

Fig. 5. The calculated changes of controlled temperature of the reactor at starting and finishing of an exothermic process

The slight difference between the measured and simulated results of the process is caused by the isolating effect of the glass cover of the electrical heater.

After the model experiments the exothermic reaction of NaOH solution with HCl solution (heat of reaction is 57750 J/mol) was carried out.

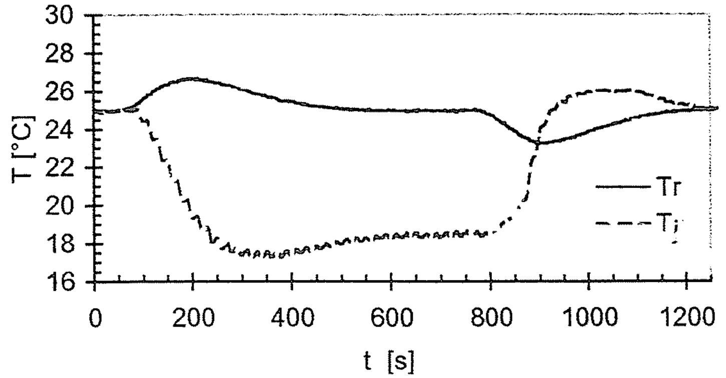

Fig. 6. The measured changes of controlled temperature of the reactor at starting and finishing of an exothermic process

The procedure was carried out by filling 250 cm^3 NaOH solution of 6 mol/dm^3 concentration into the reactor and 250 cm^3 HCl solution of 6 mol/dm^3 was added at controlled speed.

The recorded data and simulation can be compared in *Figs 7* and *8*.

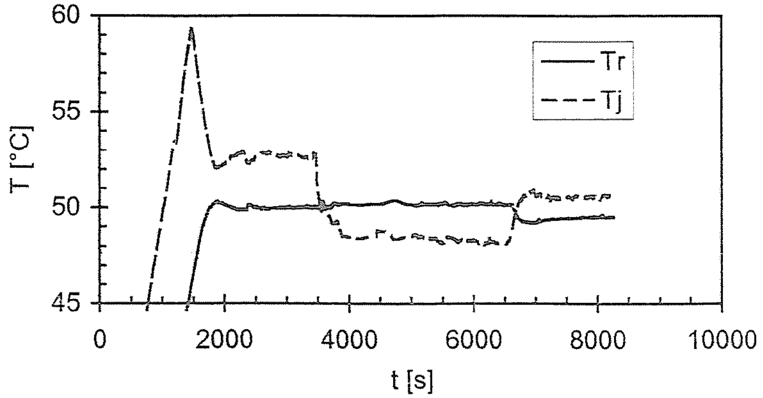

Fig. 7. Measured curves of acid-base reaction

4. Conclusions

The application of microcomputer-controlled reactors on laboratory level being appropriate for modeling the industrial processes could support the development of batch chemical technologies. A controlled reactor system

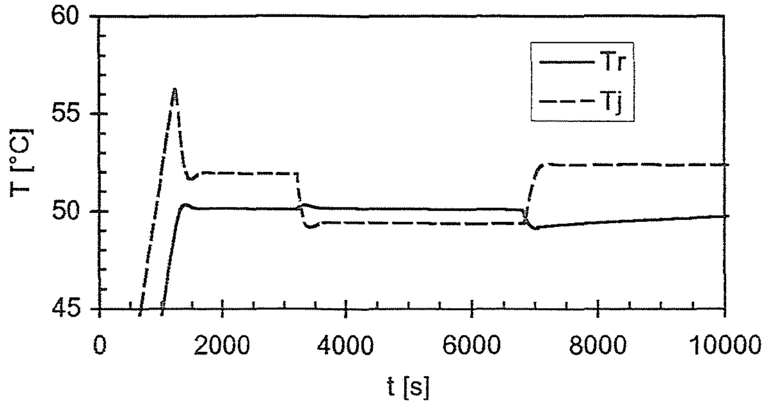

Fig. 8. Simulated curves of acid-base reaction

consisting of two reactors, common heat- exchanger circuit, reflux, distillation and stirring torque measurement facilities was constructed. A hierarchic and interactive software structure including the physical-chemical knowledge on heat and material balance support the wide applicability of the system. The control and simultaneous calculation of enthalpy or simulation was tested with model experiments, using ice and electrical heater, as well as in acid-base reactions. The experiments suggest that the hardware and software system applied accomplish appropriate control and can be used for development of chemical technologies with parallel measurement of the heat effects.

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