

Reaction heat flow control by dynamically calibrated thermometers

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Abstract

A large number of industrial processes, such as the production of fine chemicals, pharmaceuticals, bioproducts, as well as other products for which continuous production is very often not feasible, are operated batchwise. In many cases this mode of operation is used to manufacture a variety of products that involves significantly different characteristics such as conversion time, heat of reaction etc. Good control in such reactors is quite often difficult to achieve due to their flexible and multipurpose character and is essentially requires solving the problem of temperature control. Therefore the design of accurate controllers for frequent set-point changes, operation at constant and driven set-points are very important. Also the disturbances need to be handled, measuring or predicting disturbances can improve controller performance. For controller design well detailed test equipments like software based simulators or pilot plants are needed to simulate industrial environment. In this paper we would like to present an economic solution for reaction simulation in a pilot plant batch reactor without chemicals. With this kind of solution the disturbing affect of reaction heat can be investigated in a more economic way than using chemicals. With the extension of the batch reactor including a circulating pump, an electric heater, a thermometer and a flow meter after the heater, the exotherm heat of the reactions can be promoted into the reaction mixture.

In our case we chose a feed forward heat flow controller using the heat exchanger's characteristics. For reliable characteristic several preliminary measurements are needed. For heat flow calculations two temperature signals, the reactor and the heater outlet temperature were used. In the course of the measurements steady state and dynamic difference between the two thermometers were discovered. However for heat flow calculation two similarly acting thermometers are needed to avoid inaccuracy, so calibration is very important.

In this paper we also present a test showing the effect of the reaction heat on the behaviour of the reactor's temperature controller. Our aim was to lead the reactor on a temperature profile while simulating the reaction heat with our feed forward controller using dynamically calibrated thermometers. With this test we would like to show the disturbing effect of the reaction heat.

Keywords: batch reactor, reaction simulation, heat flow control

I. Introduction

A large number of industrial processes, such as the production of fine chemicals, pharmaceuticals, bioproducts, as well as other products for which continuous production is very often not feasible, are operated batchwise. In many cases this mode of operation is used to manufacture a variety of products that involves significantly different characteristics such as conversion time, heat of reaction etc. Good control in such reactors is quite often difficult to achieve due to their flexible and multipurpose character and is essentially requires solving the problem of temperature control. [1]

Moreover, the control performance mainly depends on the heating-cooling system associated with the reactor. Two main types of heating-cooling systems are commonly used in industry: the alternate

system or multifluid system and the monofluid system. The well-known alternate system applies utility fluids flow alternatively in the jacket. The monofluid system uses a single-fluid, the temperature of which can be modified to achieve the desired reactor temperature by an intermediate thermal loop which may include heat exchangers, power heaters, etc. An example for this kind of system can be seen on Figure 1. [2],[3]

II. The monofluid heating-cooling system

In our laboratory a 50 litre stirred batch reactor with a monofluid heating-cooling including three temperature levels can be found. The main part of this system can be seen on Figure 1. The monofluid system contains three 100 litre tanks, one for each temperature loop (in our case $-15\text{ }^{\circ}\text{C}$ / $20\text{ }^{\circ}\text{C}$ / $110\text{ }^{\circ}\text{C}$). The three loops providing the three different temperature levels have the same glycol-water fluid inside and have independent control. The coolest fluid is produced with a refrigerator, the middle with a heat exchanger cooled with tap water and the hottest with electric heaters. The appropriate temperature can be chosen with the ball valves at the boundary of the reactor jacket's loop and the flow rate can be adjusted with a control valve on the returning stream.

III. Temperature control

As mentioned above accurate temperature control is one of the most important control functions in batch processes. Therefore the design of accurate controllers for frequent set-point changes, operating at constant and driven set-points is very important. Also the disturbances need to be handled: measuring or predicting disturbances can improve controller performance. For controller design well detailed test equipments like software based simulators or pilot plants are needed. In this paper we would like to present an economic solution for reaction simulation in a pilot plant batch reactor without chemicals. With this kind of solution the disturbing effect of reaction heat can be investigated in a more economic way than using chemicals. [4]

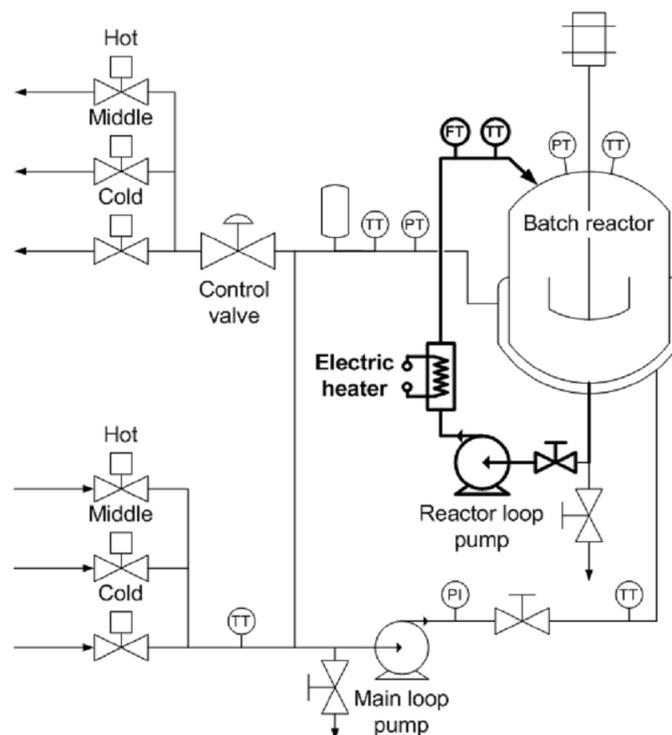


Figure 1: The 50 litre batch reactor and the reaction simulation loop

IV. Reaction simulation

With the extension of the batch reactor (Fig. 1) including a circulating pump, an electric heater, a thermometer and a flow meter after the heater, the exotherm heat of the reactions can be promoted into the reaction mixture (usually water). A quick acting controller is needed for heat flow control to follow the changes of the reaction heat.

In our case we chose a feed forward controller using the heat exchanger's characteristics (heat flow vs. manipulated variable). For reliable characteristic several preliminary measurements were needed. For heat flow calculations two temperature signals, the reactor and the heater outlet temperature were used. The flow rate in this loop is a constant value, and it can be monitored with the built in flow meter on the outlet of the heater. Besides these data the specific heat capacity and the density of the reaction mixture (water) was used.

In the course of measurements significant steady state and dynamic difference between the two thermometers were discovered. However for heat flow calculation two similarly acting thermometers are needed to avoid inaccuracy, therefore appropriate calibration is very important. The thermometer with faster dynamic response (the thermometer after the heater) can be modulated with a first order filter and a temperature dependent steady state offset to match the signal of the slower one. The measurement for determining the filter parameters can be seen on (Fig. 2) and the recording of the heater's characteristics on (Fig. 3) and (Fig. 4). (The manipulated variable is the on/off rate of the heater in a certain time period, so 50 % means half period on and half period off) This confirms that no accurate controller can be designed without the use of suitable thermometer calibration.

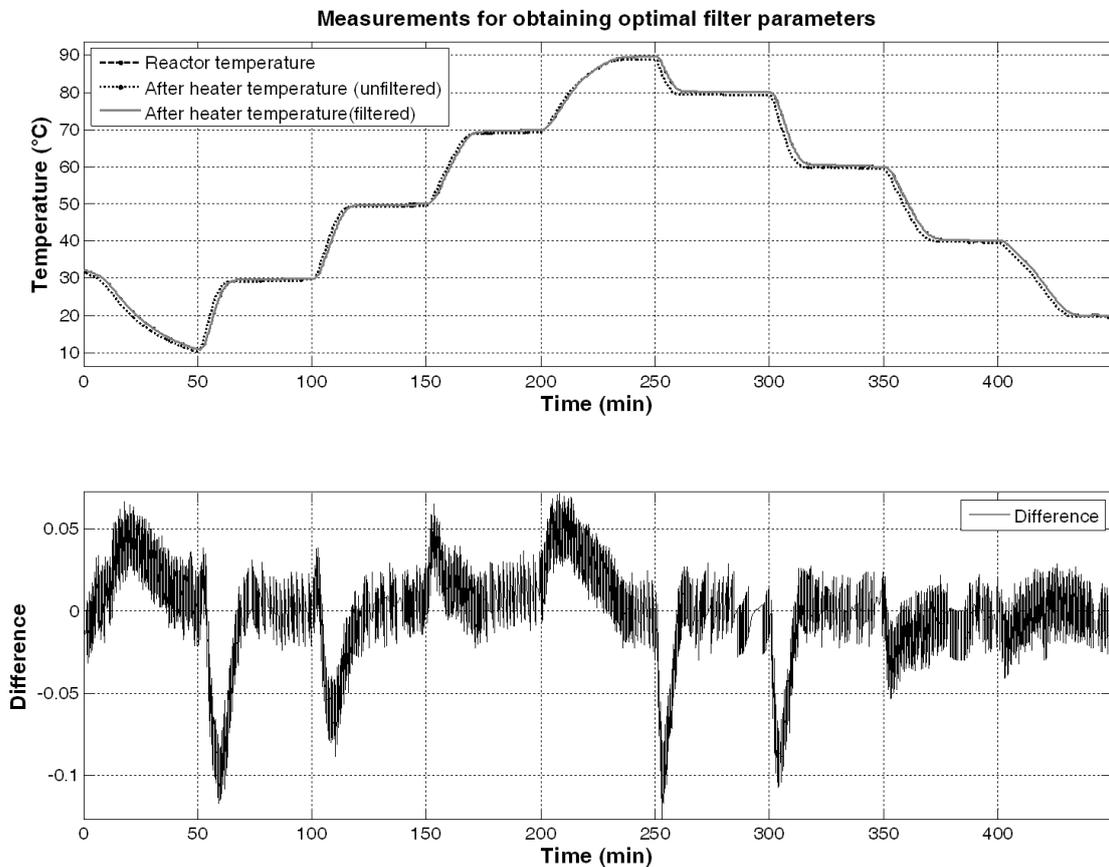


Figure 2: Measurements for obtaining optimal filter parameters

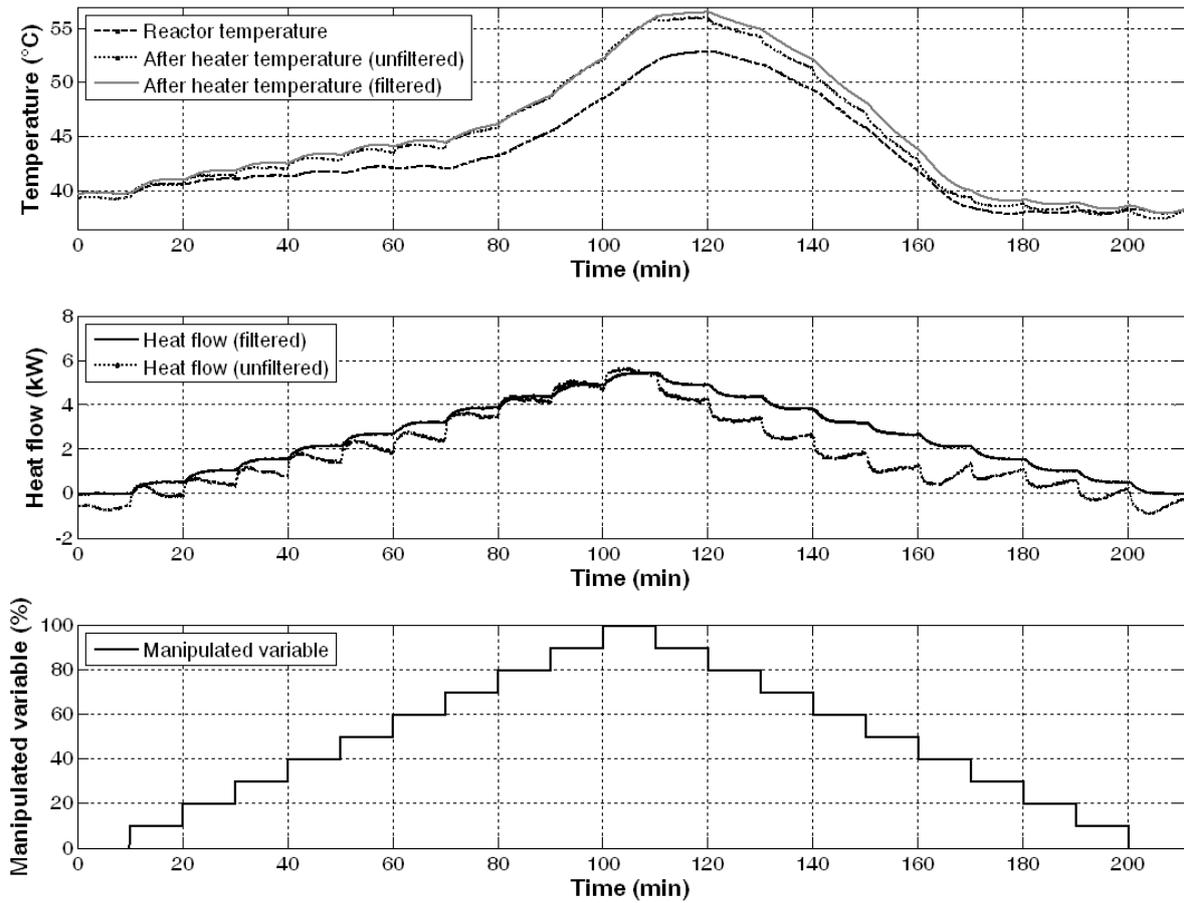


Figure 3: Measurement for recording the heater's characteristics

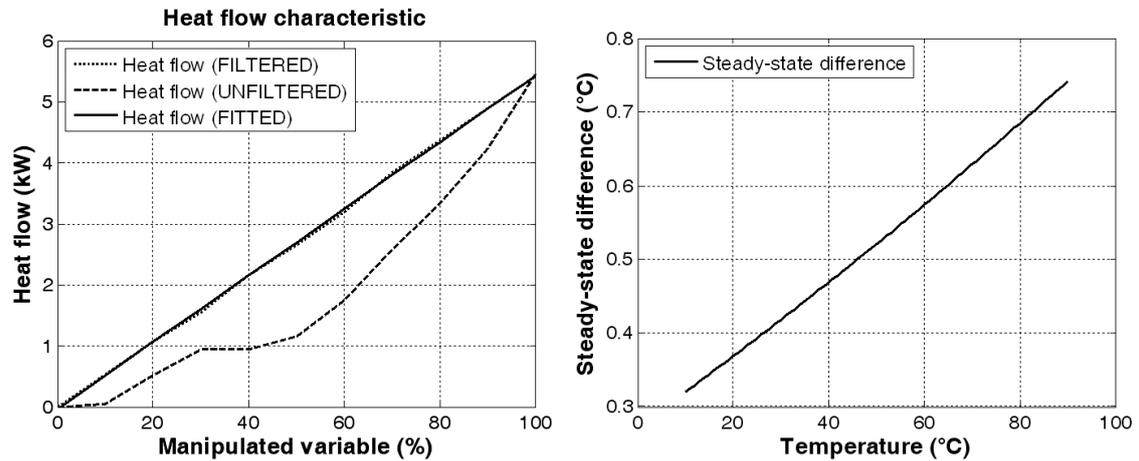
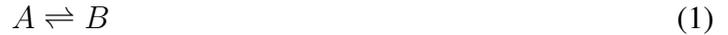


Figure 4: The characteristics of the heater and the temperature dependent steady-state difference between the thermometers thermometer

V. Simulation of chemical reaction heat

Our aim was to show the ability to simulate heat of reaction and to lead the reactor on an optimal temperature profile. First the reaction had to be chosen for investigation. Normally a reactor is designed after gathering information about the reaction kinetics. In our case we already had a batch reactor, thus

we had to design a model reaction that fits the abilities of the reactor. We selected a simple first order equilibrium reaction:



A is the raw material, B is the product. The reaction takes place in homogenous liquid phase and no catalyst is needed. The equations describing the kinetics of the reaction are:

$$r_1 = k_{0,1} \exp\left(-\frac{E_{a,1}}{RT}\right) \quad (2)$$

$$r_2 = k_{0,2} \exp\left(-\frac{E_{a,2}}{RT}\right) \quad (3)$$

Where index 1 marks the forward, 2 the backward reaction. k_0 is the preexponential factor of the Arrhenius-equation ($1/s$), E_a is the activation energy (J/mol), R is the universal gas constant ($\frac{J}{molK}$), T is the absolute temperature (K), c_A and c_B are the concentrations of component A and B (mol/m^3). In a single phase batch reactor the source of change in the concentration of a component is solely the chemical reaction. The time derivatives can be expressed in the following way:

$$\frac{dc_A}{dt} = -r_1 + r_2 \quad (4)$$

$$\frac{dc_B}{dt} = r_1 - r_2 = -\frac{dc_A}{dt} \quad (5)$$

The initial concentration is $c_{A,0}$ for component A and 0 for component B. The concentration of B can be expressed as:

$$c_B = c_{A,0} - c_A \quad (6)$$

These equations describe the concentration changes, and if the temperature history is available, then the concentrations and reaction rates can be calculated. This is needed to calculate the actual rate of heat generated by the reaction:

$$Q_r = V_r \Delta H_r (r_1 - r_2) \quad (7)$$

Where Q_r is the heat generated by the reaction (J/s), V_r is the volume of the liquid inside the tank (m^3), ΔH_r is the absolute value of enthalpy change of reaction (J/mol). Using the above equations an expression for the reactor temperature can be derived. This is the temperature where the change of concentrations is the fastest, thus resulting the highest possible conversion in a given time.

$$T_{reactor} = -\frac{E_{a,2} - E_{a,1}}{R \ln\left(\frac{k_{0,1} E_{a,1} c_A}{k_{0,2} E_{a,2} (c_{A,0} - c_A)}\right)} \quad (8)$$

There are several limitations in choosing the kinetic parameters. One of them is the applicable temperature range. The reaction should take place between 20 °C and 80 °C, to ensure the temperature gradient between the jacket and the tank is satisfactory. The composition of the reaction mixture should depend highly on the temperature in this range. This way we are able to show that at the start of the reaction high temperature is needed but for product-rich final composition low temperature is preferred.

The reaction needs to be definitely exothermic since the heat of reaction is introduced to the system by an electric heater. The temperature changes are limited by the heat capacity of the body of the equipment. The maximum heat of reaction should not exceed the maximum power of the heater. This can be manipulated easily since the maximum heat of reaction is proportional to the starting concentration of component A.

We obtained a temperature profile close to the optimal one, but with regard to the above limitations (it is not optimal in the aspect of the reaction). This way we expected that the controller would be able

to follow the temperature profile with small error. The reactor's temperature controller is not detailed here. It is a model based master-slave controller. The slave level controls the temperature of the jacket using the flow rate of the appropriate heating/cooling fluid as manipulated variable. The master level controls the temperature of the reactor and supplies the set point to the slave level as its manipulated variable.

Our aim was to show the disturbing effect of the reaction on the performance of the reactor's temperature controller. Figure 5 shows two tests to present the difference between the control with and without reaction heat disturbance. The reactor's temperature controller was unchanged during the tests.

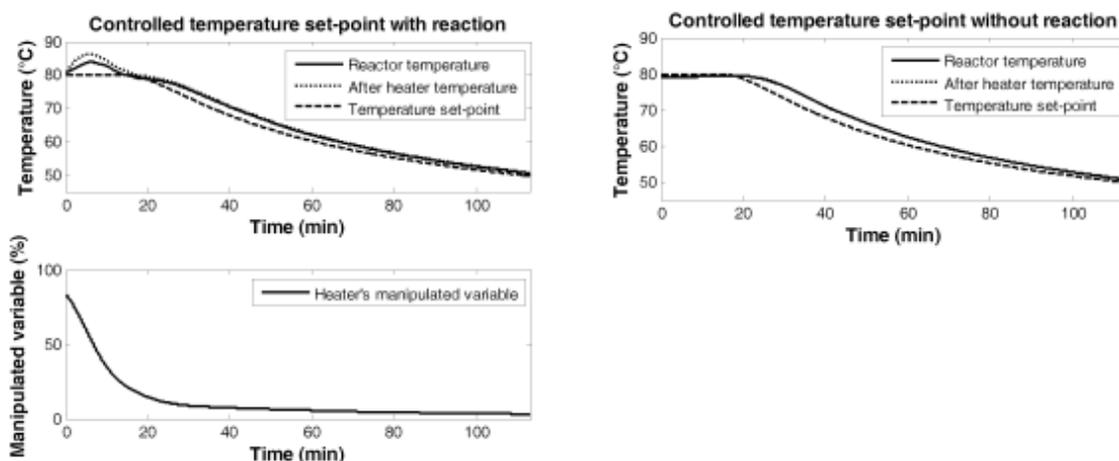


Figure 5: Temperature control with and without reaction

VI. Conclusions

The batch reactor extended with an electric heater enables the consideration of the exotherm heat of reactions without using any chemical compounds, which helps designing controllers with better performance. In this paper we revealed the importance of thermometer calibration in aim of reliable heat flow controller design. With this heat flow controller and extension of the reactor the performance of the reactor's temperature controller algorithm can be developed.

Acknowledgement

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