The effect of operational conditions on the performance of batch polymerization reactor control

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Abstract

In this work, the effect of operational conditions on the performance of a controlled batch polymerization reactor was investigated experimentally. The effect of agitation speed on conversion and heat transfer coefficient in free radical chain growth polymerization in this controlled, stirred, jacketed batch reactor was also investigated. The transient response of the polymerization reactor following sequential step changes in agitation speed has been obtained experimentally. The experiments were conducted under optimal loading conditions calculated by using Lagrange's multiplier method. The reactor temperature was controlled by manipulating the heat input to the reactor. Some correlations are also provided relating the viscosity and the overall heat transfer coefficient to the monomer conversion. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Controlled batch polymerization reactor; Heat transfer coefficient; Reactor control

1. Introduction

Agitated polymerization reactors play a key role in polymer engineering. Due to the complex nonlinear nature of batch process dynamics, an accurate model cannot be generated generally. These reactors require a heat-removal capability and sufficient mixing in order to be controlled precisely. In polymer processes, the viscosity increases with conversion and this makes perfect mixing impossible. Viscosity has a significant effect on the heat transfer characteristics as well. Non-ideal mixing conditions in stirred tank reactors are often suspected to be the cause of unexpected broad chain length distribution in free radical chain polymerization [1]. In the literature, there have been relatively few studies on the effect of mixing on polymerization reactions in stirred tank reactors. Tosun [1] studied theoretically the effect of mixing on number and weight average degree of polymerization in free radical homopolymerization in solution in a semibatch-stirred tank reactor. Lee and Lee [2] studied the effect of mixing on conversion in a fast growth polymerization in a stirred adiabatic tank reactor. Baade et al. [3]

studied the kinetics of high conversion polymerization of vinyl acetate, effect of mixing, and reactor type on polymer properties. They found that there is no significant influence of stirring rate. Contrary to this study, Gunesch and Schneider [4] have stated that doubling of the stirring rate almost cuts in half the polymerization rate for the vinyl acetate–dibenzyl peroxide system. Hicks and Gates [5] gave some information about fluid agitation in polymer reactors. Kalfas et al. [6] investigated the effect of stirring speed, temperature, initiator concentration, and some other parameters on the conversion profiles and on the molecular weight, droplet, and particle size distributions in batch suspension polymerization. They found that increasing stirring speed causes the PSD to become narrower. Mankar et al. [7] have reported viscosities of the reaction mixture measured by a Haake viscometer continuously during bulk polymerization of methyl methacrylate for the four combinations of temperature and initial initiator concentration. They developed appropriate correlations for viscosity, η, of the reacting mass. Srınıvas et al. [8] studied the effect of temperature changes on bulk polymerization of MMA in a semibatch reactor. They used a PI algorithm to control the reactor temperature and they conducted a series of experiments on bulk polymerization of MMA under isothermal conditions by applying step changes to temperatures on the semibatch reactor. Monomer conversion and average molecular weight were taken as the output variables.

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Nomenclature

A	heat transfer area (m²)
A_d, A_p, A_t	frequency factor for initiator decomposition, propagation, and termination, respectively (1/s, 1/(s mol), and 1/(s mol), respectively)
C_p, C_{pc}	specific heats for reactor content and coolant, respectively (kJ/(kg K))
D_e	equivalent diameter of jacket (m)
D_i	impeller agitator diameter (m)
D_T	inside tank diameter (m)
E_d, E_p, E_t	activation energy for initiator decomposition, propagation, and termination, respectively (kJ/(mol K))
f	initiator efficiency
h_i	inside heat transfer film coefficient (W/(m² K))
h_o	outside heat transfer film coefficient (W/(m² K))
H_i	impeller height from the vessel bottom (m)
ΔH_p	heat of polymerization reaction
I, I_0	initiator concentration and initial initiator concentration, respectively (mol/l)
k, k_c	thermal conductivity of reactor content and coolant, respectively (W/(m K))
k_d, k_p, k_t	kinetic constants for initiator decomposition, propagation, and termination, respectively (1/s, 1/(s mol), and 1/(s mol), respectively)
K_c	controller gain
m_c	coolant flow rate (kg/s)
M, M_0	monomer concentration and initial monomer concentration, respectively (mol/l)
Q	heat given from the electrical heater (W)
R_{wall}	resistance of wall ((m² K)/W)
t, t_f	time and polymerization end time, respectively (min)
T	reactor temperature (°C)
T_c, T_{ci}, T_{co}	average, inlet, and outlet coolant temperature, respectively (°C)
U	overall heat transfer coefficient (W/(m² K))
V	volume of the reactor (m³)
V_c	volume of jacket (m³)
w	reactor wall thickness (m)
x, x^*	monomer conversion and desired monomer conversion, respectively
X, X_n^*	number of average chain length and desired number of average chain length, respectively

Greek letters

μ	viscosity of the reacting mixture (cp)
μ_I	ith moment of dead polymer distribution
μ_w	wall viscosity (cp)
ρ, ρ_c	density of reactor content and coolant density, respectively
τ_d, τ_I	derivative time constant and integral time constant, respectively

The experimental results show fairly good agreement with predictions from the theoretical model of Ray et al. [9]. The validity of these models under semibatch reactor conditions has been established by Dua et al. [10]. Step changes in the initiator concentration were applied during the course of polymerization. Experiments were carried out at constant temperatures. Also they established the applicability of these models for more general semibatch reactor operations and model-based optimal control of industrial reactors.

Furthermore, heat transfer in agitated polymerization reactors is very important, because the temperature in the reactor is one of the most significant factors determining the outcome of the polymerization processes. The intensity of heat transfer during mixing of fluids depends on the type of agitator and conditions of the process.

Agitated polymerization reactors are traditionally divided into four categories: bulk, suspension, solution, and emulsion polymerization equipment. In the present work, the effect of mixing on conversion and heat transfer was studied experimentally for free radical chain solution polymerization of styrene. For a good temperature control and desired product quality, the batch must be vigorously agitated to assure overall uniformity. The optimal loading conditions were computed as $T = 103\,°C$ and $I_0 = 1.26 \times 10^{-2}$ mol/l for 50% desired conversion and 500 desired number of average chain length by applying Lagrange's multiplier method to the mathematical modeling of this reactor system. A number of experiments were conducted under these optimal conditions by changing the agitator speed from 190 to 1300 rpm. The polymerization reactor temperature was controlled by manipulating the power to the heater and the change of heating rate during polymerization was monitored to see the control performance. The transient response of the reactor temperature and the change of the manipulated variable with time were investigated by giving step changes to the agitator speed at certain time intervals. There appears to be little information on heat transfer and heat transfer coefficient of polymerization reactors in the literature. In this study, we also investigated the change of overall heat transfer coefficient in a jacketed batch polymerization reactor during unsteady-state operation under temperature control. Some correlations for overall heat transfer coefficient and viscosity of reactor mixture were obtained using experimental data and by correlating this data with regression analysis.

2. Mathematical model and optimization

To obtain optimum conditions and theoretical conversion values, the following mathematical model is developed based on mass and energy balances concerning the reactor mixture and the jacket. The standard, free radical polymerization kinetic mechanism [11] shown in Table 1 was taken and the following assumptions were made:

1. Quasi-steady-state approximation (QSSA) for live radicals and long-chain hypothesis (LCH) are valid.
2. All the reaction steps are irreversible.
3. The rate of chain transfer to solvent reactions are negligible.
4. There is no change in volume.
5. Perfect mixing and constant-reacting heat capacity and density exist.
6. The jacket temperature is uniform and the heat losses with the ambient surroundings are negligible.
7. There is no chain transfer.

The modeling equations consist of six nonlinear-state equations:

$$\frac{dI}{dt} = -k_d I \tag{1}$$

$$\frac{dM}{dt} = -k_d \left(\frac{2fk_d I}{k_t}\right)^{0.5} M \tag{2}$$

$$\frac{d\mu_0}{dt} = 2f\left(1 - \frac{\nu}{2}\right)k_d I \tag{3}$$

$$\frac{d\mu_2}{dt} = (2 + \nu)\frac{k_p^2}{k_t} M^2 \tag{4}$$

$$\frac{dT}{dt} = \frac{Q}{mC_p} + \frac{R_m V}{mC_p} - \frac{UA(T - T_c)}{mC_p} \tag{5}$$

Table 1
Parameters and given conditions for styrene polymerization reactor

A_d (1/s)	2.6×10^{16}
A_p 1/(s mol)	1.051×10^7
A_t 1/(s mol)	1.255×10^9
E_d (kJ/(mol K))	143.161
E_p (kJ/(mol K))	29.553
E_t (kJ/(mol K))	7.0325
Initiator efficiency, f	0.5
Gel effect, g	1
$\nu = \dfrac{k_{tc}}{k_t}$	1
Styrene molecular weight, M_w (g/mol)	104.14
Solvent fraction (volume)	0.3
Density of the mixture, ρ (kg/m^3)	983.73
Heat of reaction, ΔH (kJ/(kg K))	57766.8
Reactor volume, V (l)	2
Jacket volume, V_c (l)	2
Tank diameter, D_T (m)	0.15
Propeller diameter, D_i (m)	0.04
S	5.4534
C	0.012

$$\frac{dT_c}{dt} = \frac{m_c(T_{ci} - T_{co})}{V_c \ell_c} + \frac{UA(T - T_c)}{V_c \ell_c C_{pc}} \tag{6}$$

where R_m is the overall rate of heat production by the reactions which is given by

$$R_m = (-\Delta H_p)k_p \left(\frac{2fk_d I}{k_t}\right)^{0.5} M \tag{7}$$

The initial conditions are: $I(0) = I_0$, $M(0) = M_0$, $\mu_0(0) = \mu_2(0) = 0$, $T(0) = T_0$, and $T_c(0) = T_{co}$.

The control variables in the isothermal batch-jacketed reactor are reaction temperature and initial initiator concentration. In order to obtain optimal operating conditions to achieve a predetermined conversion and average number of molecular weight in a minimum time, the method of Lagrange's multiplier [12] is used. According to this method, necessary equations to obtain T and I_0 can be obtained by optimizing the function:

$$W = t_f(T, I_0) + \lambda g(T, I_0) \tag{8}$$

where λ is the Lagrange's multiplier, and t_f and g are the functions obtained from the solution of mathematical model equations [13] given as

$$t_f(T, I_0) = -\frac{1}{A_d \exp(-E_d/RT)} \ln\left(1 - \frac{M_0 x^*}{f I_0 X_n^*}\right) \tag{9}$$

$$g(T, I_0) = -\ln(1 - x^*) + 2A_p \left(\frac{2f}{A_d A_t}\right)^{0.5}$$
$$\times \exp\left(-\frac{1}{2RT}(2E_p - E_d E_t)\right)$$
$$\times \left[\left(1 - \frac{M_0 x^*}{f I_0 X_n^*}\right)^{0.5} - 1\right] \tag{10}$$

The mathematical model of the polymerization process was solved with the fourth-order Runge–Kutta integration method under the optimal conditions computed from the related equations obtained by optimizing Eqs. (8)–(10). Then the predicted conversion values were obtained as a function of time.

3. Heat transfer coefficient

As the polymerization reactions proceed, the viscosity of the reacting mixture increases significantly and the heat transfer coefficient decreases sharply with conversion. In the literature, the decrease of the overall heat transfer coefficient has been modeled through the use of empirical correlation. Takamatsu et al. [14] used an empirical correlation of the form

$$U = U_0 \left[1 - \alpha \exp\left(1 - \frac{1}{x_m}\right)\right] \tag{11}$$

where α is a constant parameter, x_m the conversion, and U_0 the value of the overall heat transfer coefficient at the

beginning ($x_m = 0$). Chylla and Haase [15] proposed an empirical exponential correlation to relate U to the viscosity of the reacting mixture as

$$U = \frac{1}{(1/143.4 \exp(-5.13 \times 10^{-3} \mu_{\text{wall}})) + 1/h_f} \quad (12)$$

where h_f is the fouling factor.

Soroush and Kravaris [16] used the empirical correlation below:

$$U = U_0[\alpha + (1 - \alpha) \exp(-\beta x_m^{\gamma})] \quad (13)$$

where α, β, and γ are the parameters determined experimentally.

Generalized equations for heat transfer in jacketed, agitated vessels have been presented in various literatures. The following correlation for the inside film heat transfer coefficient of vessels having nonstandard impeller sizes [17] was utilized for the solution polymerization of styrene at low conversions

$$\frac{h_i D_T}{k} = 1.01 (N_{Re})^{0.66} (N_{Pr})^{0.33} \left(\frac{\mu}{\mu_w}\right)^{0.14}$$
$$\times \left(\frac{H_i}{D_T}\right)^{0.12} \left(\frac{D_i}{D_T}\right)^{0.13} \quad (14)$$

If this equation is arranged and solved for h_i, the equation below is obtained:

$$h_i = S N^{0.66} \mu^{-0.33} \Phi_{\text{vis}} \quad (15)$$

where

$$S = \frac{1.01 k^{0.67} \rho^{0.56} D_i^{1.45} C_p^{0.33} H_i^{0.12}}{D_T^{1.25}} \quad (16)$$

$$\Phi_{\text{vis}} = \left(\frac{\mu}{\mu_w}\right)^{0.14} \quad (17)$$

The outside film heat transfer coefficient may be found from the equation for laminar flow of fluids inside the pipes:

$$h_o = 2.016 \frac{k_c^{0.67}}{D_e} \left(\frac{m_c C_p}{D_T}\right)^{0.33} \left(\frac{D_e}{D_T}\right)^{0.33} \left(\frac{\mu_c}{\mu_w}\right)^{0.14} \quad (18)$$

So the overall heat transfer coefficient can be expressed as a function of viscosity and agitator speed for a constant flow rate of coolant as

$$U = \frac{1}{S^{-1} N^{-0.66} \mu^{0.33} \Phi_{\text{vis}}^{-1} + A} \quad (19)$$

where

$$A = R_{\text{wall}} + \frac{D_T}{(D_T + 2w) h_o} \quad (20)$$

4. Reactor control

Temperature control of polymerization reactors is very important in the production of polymers. The reaction

temperature determines the chemical composition and a precise control of the temperature is required to produce an acceptable product. In this work, the manipulated variable was taken as the heat input to the reactor. A PID control algorithm was implemented on the experimental reactor system to control the temperature under optimal conditions. The optimum values of the PID control parameters were computed using Rosenbrock's optimization techniques [18,19] as $K_c = 7.2$, $\tau_I = 1.3$, $\tau_d = 0.3$. These parameters were taken the same for all experiments.

5. Experimental system

A schematic diagram of the experimental apparatus is provided in Fig. 1. The reactor was a 2 l stainless steel cylindrical vessel. A condenser is attached to the head port to provide an exit for the nitrogen and to condense toluene and monomer, because solvent evaporation takes place in the temperature range under consideration. Nitrogen is bubbled through the reaction mixture to keep oxygen out of the system. The temperatures of the reactor, inlet, and outlet cooling water were measured by Fe–constantin thermocouples and sent to the computer. The reactor mixture was agitated by a propeller of 14 cm diameter. A variable speed driver permitted the impeller speed to be varied from 190 to 1300 rpm. An immersed heater in a quartz sheath was provided inside the reactor for supplying the appropriate heat. A thyristor unit regulated the voltage to the immersed heater depending upon the reactor temperature. The flow rate of the coolant was measured by a flowmeter. As coolant, the city water was used. A computer with A/D and D/A converters was employed to control the temperature of the reactor.

In the experimental work, commercial styrene was firstly vacuum-distilled to remove the inhibitor. Benzoyl peroxide which was dissolved in chloroform and then recrystallized in methanol was used as initiator. Toluene was chosen as solvent. At the start of the experiment, the reactor content (1400 ml of styrene + 600 ml of toluene) was brought to the desired optimal temperature (103 °C). Then the optimal amount of benzoyl peroxide was added to the reactor and a PID control method was applied to this system to keep the reactor temperature at the desired temperature. Polymer samples were withdrawn from the reactor at certain intervals for conversion and viscosity measurements during the polymerization. The temperatures of the reactor system were monitored and observed on the computer during the experiments.

Transient experiments were carried out by giving sequential step changes to the agitator speed at certain intervals. At intervals of 15 min, 5 ml samples were taken from the reactor for the off-line analysis. The monomer conversion was determined by the precipitation method. The viscosity of the reacting mixture was measured immediately after being taken out of the reactor, using a falling-ball viscometer.
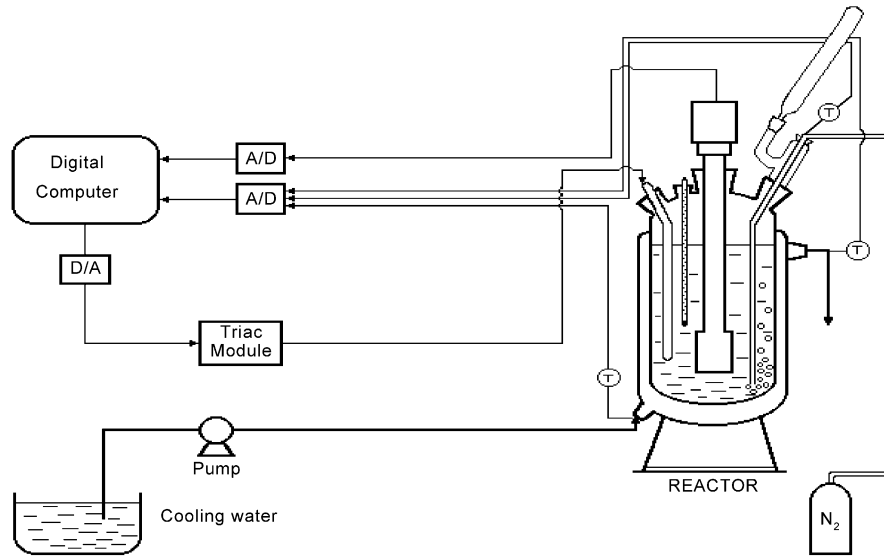
Fig. 1. Schematic diagram of the polymerization reactor system.

Table 2
Operating conditions

I_0 (mol/l)	M_0 (mol/l)	T (°C)	m_c (kg/s)	T_{ci} (°C)	x^* (%)	X_n^*	t_f (min)	N (rpm)
0.0126	6.699	103	0.00041	21	50	500	151	190–1300

6. Results and discussion

Using Lagrange's multiplier method, optimal temperature, and initial initiator concentration were computed for different desired conversion and average number of molecular weight values. For this study, desired conversion and number of average chain length were chosen as 50% and 500, respectively. Although 100% conversion is required

to obtain the polymer economically, it is very difficult to achieve this conversion and styrene does not exhibit an effective gel effect up to 50% conversion. To reach these desired values, optimal values for T and I_0 were computed as 103 °C and 1.26×10^{-2} mol/l, respectively. The optimal conditions used in the experimental studies are given in Table 2.

Effective agitation of the polymerization reactors requires a detailed analysis of viscosity, heat transfer, and dynamic
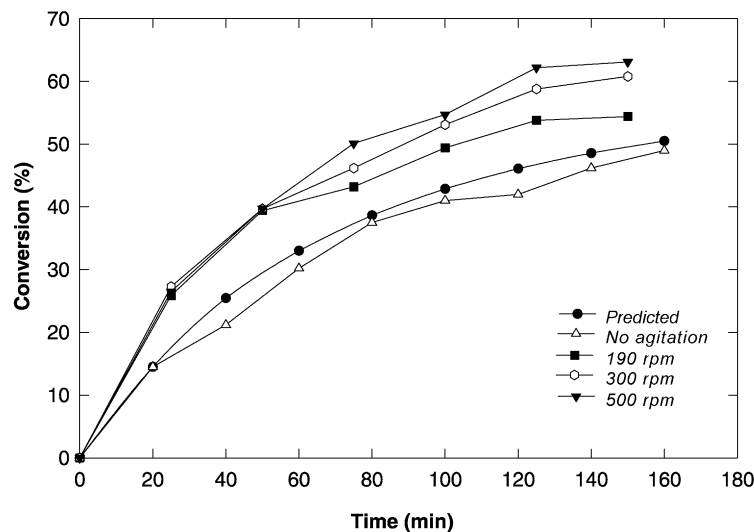

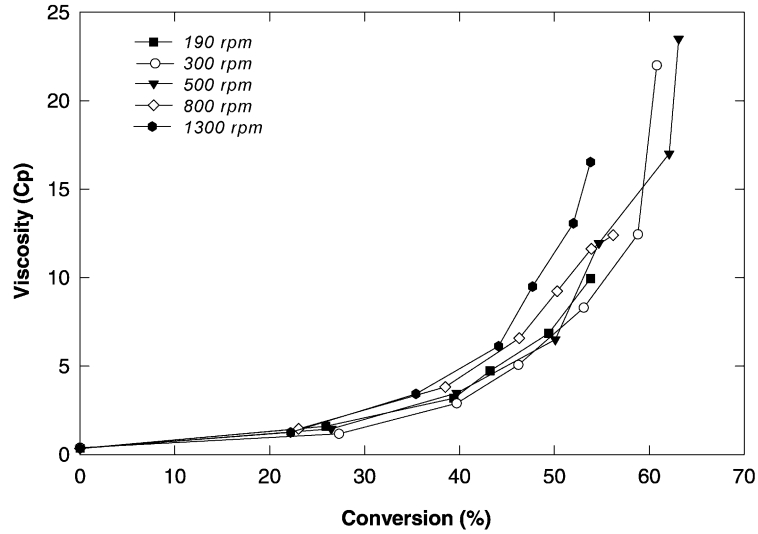Fig. 2. Time–activity curves of different stirring rates.

Fig. 3. The change of viscosity with conversion.

Table 3
Experimental conversion values at different stirring rates

t (min)	x				
	190 rpm	300 rpm	500 rpm	800 rpm	1300 rpm
0	0	0	0	0	0
25	25.90	27.30	26.50	23.05	22.20
50	39.40	39.70	39.70	38.50	35.41
75	43.20	46.20	50.10	46.30	44.12
100	49.40	53.10	54.70	50.30	47.72
125	53.80	58.80	62.20	53.90	52.01
150	54.40	60.80	63.10	56.20	53.81

Table 4
Experimental polymerization time for 50% conversion

N (rpm)	Predicted	0	190	300	500	800	1300
t (min)	151	180	101	91	75	100	120

the same conditions to test experimental reproducibility. Fig. 2 shows the time–activity curves of different stirring rates from 190 to 500 rpm. The curves for no stirring, and the predicted curve are also illustrated in this figure. It is remarkable that while the conversion for no stirring is lower than predicted conversion, increasing stirring speed causes the conversion to increase. But, there is a decrease in conversion after 800 rpm (Table 3). So it was concluded that a satisfactory fluid motion in this solution polymerization

response for the polymerization system. In the first part of this study, a number of experiments was carried out to see the effect of agitator speed on the conversion. The measurements were repeated five times at every stirring rate under
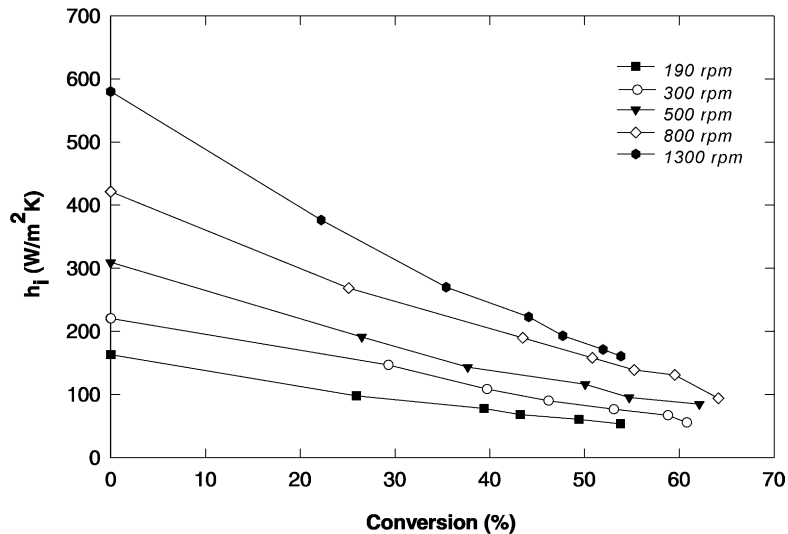

Fig. 4. The change of inside film heat transfer coefficient with conversion.
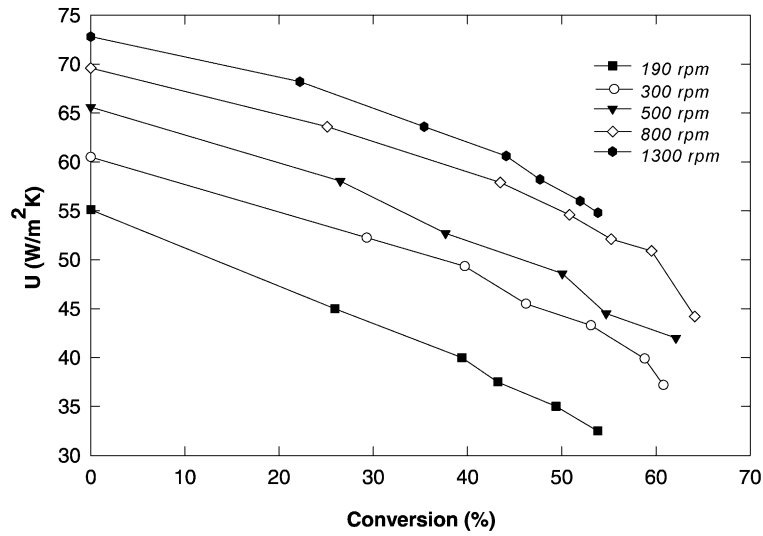
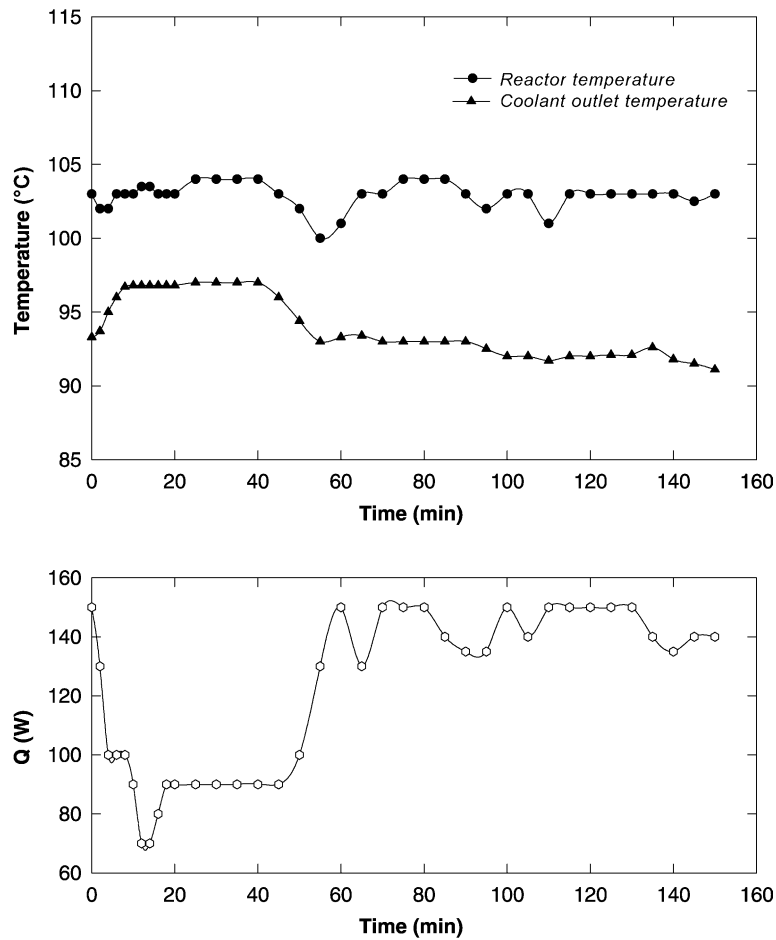Fig. 5. The change of overall heat transfer coefficient with conversion.

Fig. 6. The change of reactor temperature, coolant outlet temperature, and heat input with time ($N = 500$ rpm).

reactor was achieved at a stirring speed of 500 rpm. However, the conversion values are about the same at low conversions and the effect of the stirring speed can be best seen at high conversion. No significant influence of the stirring rate has been observed for conversions up to 30% where viscosity of the mixture is rather low. These results can be attributed to the fact that conversion rates depend on the initiator efficiency *f* which is generally related to mixing in the reactor and it was taken as 0.5 in the modeling. When *f* falls as it does for the no agitation case, conversion rates are lower than the predicted ones. With agitation, efficiency increases up to a certain value. Other possible causes for the discrepancy between the predicted and experimental conversion values are oversimplified kinetics for the polymerization and the assumption of perfect mixing in the reactor. Solvent evaporation may also play an important role in causing discrepancies between the model predictions and experimental results.

The change in the conversion was also investigated by giving sequential step changes to the agitator speed. Table 4 shows the time required for 50% conversion at different agitator speeds. The polymerization time decreases as *N* (agitator speed) increases up to 500 rpm, after which there

Table 5
Values of constants of the overall heat transfer coefficient

N (rpm)	U_0 (W/(m^2 K))	α
190	55.1	0.4
300	60.5	0.33
500	65.6	0.33
800	69.5	0.33
1300	72.8	0.33

is an increase in time with stirring speed. As a result, it can easily be seen that the reaction time has a minimum value depending on the stirring speed.

As the reaction proceeds, the viscosity of the reacting mixture was measured continuously by using a falling-ball viscometer at the reaction temperature of 103 °C. Fig. 3 presents the viscosities found during the polymerization reactions as a function of conversion. Eq. (21) is viscosity correlation which has been determined empirically from this experimental data using regression analysis:

$$\mu = 0.16\,e^{8.17x} \tag{21}$$

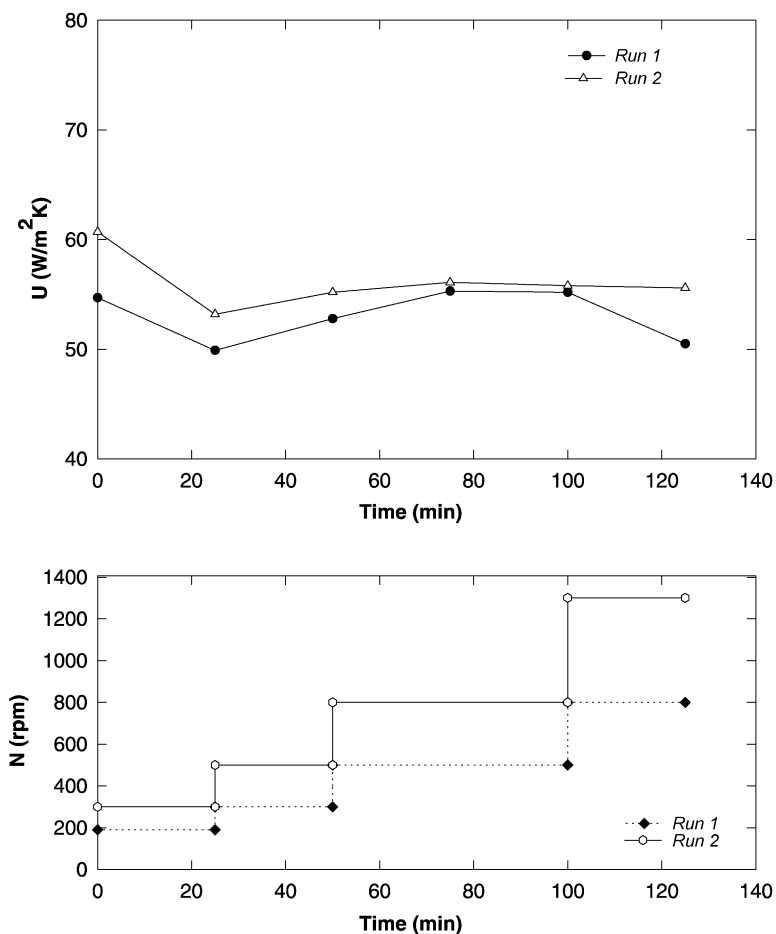where μ is the reactant viscosity (cp) and x the monomer conversion.

Fig. 7. Overall heat transfer coefficient change with sequential step changes.

Mankar et al. [7] developed correlations for the viscosity of the polymerization mass and established the feasibility of using these as a software sensor for estimating the state of the system and for implementing on-line optimizing control. In this work, we have also obtained a relation between conversion and viscosity (Eq. (22)). By using this equation, conversion and concentration of the polymer can be automatically calculated. In this way, the control of conversion can be made easier:

$$x = 0.12 \ln(6.25\mu) \tag{22}$$

The film heat transfer coefficients for the reactor system were calculated from Eq. (15) using the data collected during the polymerization experiments. The film heat transfer coefficient falls sharply during the batch, as the viscosity increases. The change of film heat transfer coefficient with conversion is shown in Fig. 4. The data for overall heat transfer coefficient which was obtained from Eq. (19) is plotted against the conversion. These data can be correlated by a straight line adequately up to 50% conversion. Increasing the polymer concentration with monomer conversion produces an almost linear decrease in overall heat transfer coefficient. The lines obtained by a least-square fitting of the coefficients

Table 6
Experimental conversion values obtained under sequential step changes

t (min)	Run 1		Run 2	
	N (rpm)	x (%)	N (rpm)	x (%)
0	190	0	300	0
25	300	25.26	500	28.20
50	500	36.67	800	37.98
75	800	43.78	800	44.25
100	800	52.53	1300	49.76
125	800	55.89	1300	52.42

for different agitator speeds are very nearly parallel with slopes varying between 0.33 and 0.4 for various operating conditions. Hence, the overall heat transfer coefficient up to 50% conversion can be expressed by the equation:

$$U = U_0 - \alpha x_P \tag{23}$$

where U_0 is the coefficient at $x_P = 0$ and x_P the percent conversion. The values of U_0 and α are given in Table 5.

Changes of the reactor temperature, coolant output temperature, and the manipulated variable (Q) with time during the polymerization reaction are shown in Fig. 5 for agitator speed of 500 rpm as an example. The measured temperature
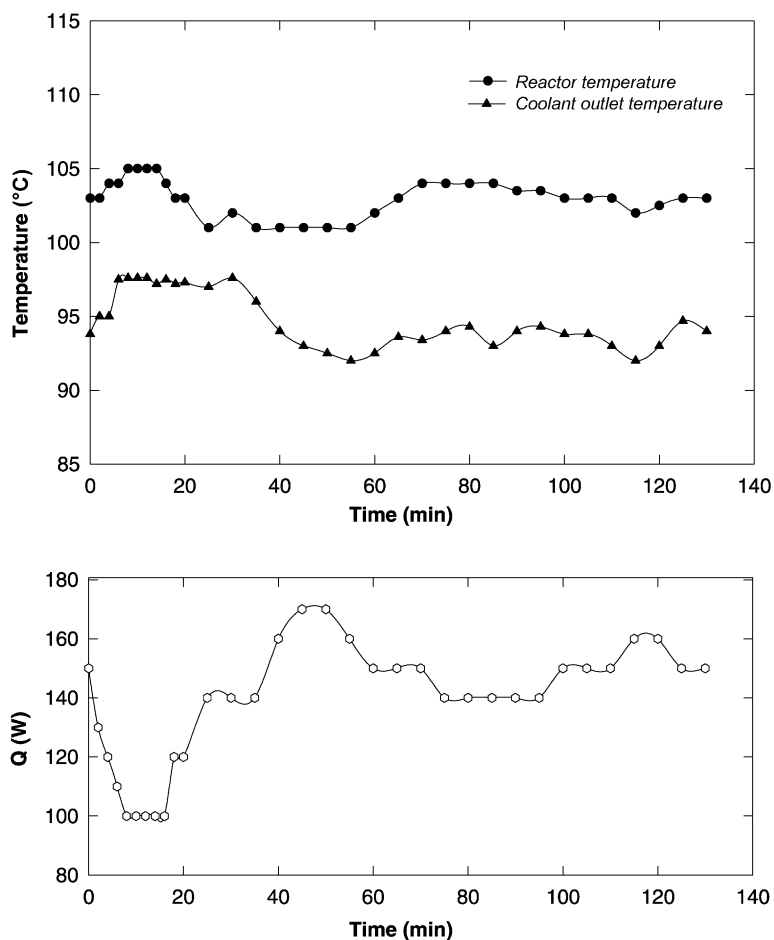

Fig. 8. The change of reactor temperature, coolant outlet temperature, and heat input with time (Run 2).

T and manipulated variable *Q* obtained show many small disturbances under PID control.

In a final experiment to examine the control efficiency with stirring rate, the reactor temperature, coolant output temperature, and the amount of heat supplied (*Q*) were investigated by giving step changes to the agitator speed as shown in Fig. 6. The change of *U* with time is given separately in the same figure. As can be seen from this figure, the heat transfer coefficient was almost constant whilst changing stirring speed. Fig. 7 presents the measured behavior of the reactor variables under changing agitator speed conditions. From the comparison of Figs. 5 and 7, it can be seen that the variation in the manipulated variable (heat input *Q*) is reduced and the control variable (reactor temperature) is maintained closer to its set point for the case of sequential step changes to agitator speed. This means a better controller performance which was achieved by changing the agitator speed and by improving the heat transfer between reactor and jacket. As depicted in this figure, although the variation of the heat input (*Q*) at the beginning of the reaction is significant, the change of agitator speed eliminates this oscillation later and manipulated variable shows much smoother performance. The change of agitator speed leads to smaller temperature differences with the set point and to fewer changes in electric power supply and more changes in the cooling water temperature. While the change in overall heat transfer coefficient is 36% for $N = 500$, the change in this coefficient following the sequential step changes in the agitator speed is only 0.084%. So the change in agitator speed affects the overall heat transfer coefficient, the heat transfer characteristics of a polymerization reactor and this makes the heat exchange more easy. The conversion values obtained during the polymerization reaction under sequential step change conditions for two different cases are shown in Table 6. It is observed that these values are in agreement with the average conversion values corresponding to different agitator speeds (Fig. 8).

As we have seen, a good agitation improves the process control and efficiency of the control performance by improving heat transfer between the jacket and the reactor. As a result, much more satisfactory control was obtained. Agitation speed has been shown to be quite promising in improving the heat transfer and temperature control. Obviously, more work is desirable to improve the overall heat transfer coefficient especially at high conversion polymerization processes.

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