Model-based optimal control of the production of polyvinyl acetate

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1. Abstract

In the environment of increasing demand and competitiveness the polymer manufacturers need to augment the production at the lowest cost. This work considers the polymerisation plant in Mitol factory in Sežana, Slovenia, which is nowadays working over ninety per cent of its capacity. The preliminary analysis of the production process of polyvinyl acetate in Mitol revealed that there are not bottlenecks in the process. To increase the production without further investments a reduction of the duration of the reaction should be achieved. The main variables affecting the duration of the reaction and thus the productivity are: the temperature in the reactor, the initial amount of monomer, the subsequent feed rate of monomer and the addition of initiator. Strong influence of the initial conditions on the final quality variables prevents us from modifying them. The optimisation of the profile of addition of monomer, based on a model-based estimation of its concentration in the reactor highly reduces the duration of the reaction. This optimal control of the addition of monomer will also require the automation of the addition of initiator, in order to keep the temperature in the required range, even under variable flow rates of monomer addition.

Keywords: Polyvinyl acetate, model-based optimisation, semi-batch.

2. Introduction

The main variables affecting the duration of the reaction and thus the productivity are: the temperature in the reactor, the initial amount of monomer, the subsequent feed rate of monomer and the addition of initiator.

Currently the temperature is controlled by manually adding the initiator. This can lead to the offset and oscillations of the reactor temperature, affecting both the final quality and the duration of the reaction. The ratio between the amount of monomer charged in the beginning and the amount charged during the reaction is feasible, but it has never been optimised. However the initial recipe has such a great influence on the final characteristics of the product that it is not advisable to modify them. We will focus on the second part of the reaction, during which 90% of the monomer is added, to try and shorten the duration of the reaction. Currently the feed rate of the monomer is kept constant, and the reaction is

controlled mainly by the addition of the initiator. The initiator is introduced manually at the operators' discretion. During the polymerisation reaction the operators take the responsibility to decide when to add more initiator, and when to start the addition of monomer. Their decisions are based on the observation of the temperature profile (figure 1), so we will make a few remarks on it.
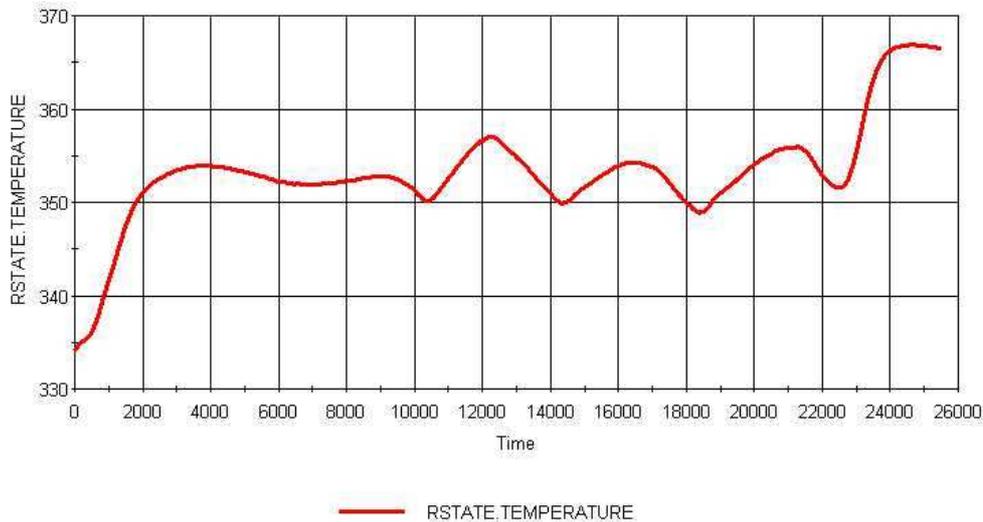

Figure 1: Temperature profile

At the beginning the mixture inside the reactor has to be heated by the external heating coat in order to reach the temperature at which the thermal initiator starts to decompose. At approximately 333 K we can observe a change in the curvature of the raising temperature. This is caused because the potassium persulfate (KPS) started its decomposition and so the exothermic reaction of polymerisation.

The reactor has to reach approximately 353 K, and it shouldn't surpass it, in order to keep the efficiency of the initiator. That is why, at around 343, the operators stop the flow of the heating water around the reactor. The exothermic reaction and the heat in the remaining water filling the heating coat, continue to raise the temperature over the set point.

Experimentally the operator knows when most of the initial amount of monomer in the reactor has been consumed. They observe the natural flow through the condenser, and visually determine when the reaction is prepared to receive more monomer. At this point they start the addition of the monomer at a constant rate, which has been proved to work well.

From this point, and until all of the monomer has been introduced in the reactor, the operator has to control the temperature around the set point at 353 K. When he observes that the temperature is starting to decrease, he has to add a fixed amount of initiator solution, in order to accelerate the reaction and increase the temperature again. This kind of control, so strongly human dependant has a poor performance as can be deduced from the shown profile. After all the monomer has been added a final amount of initiator is introduced in the reactor in order to finish the remaining monomer. It can be seen that at this point there is a sudden increment of the temperature in the reactor.

The purpose of this work is to find a strategy for the control of both the addition of the initiator and the flow rate of the monomer. It would be apparently optimal to introduce all the monomer in the beginning, with enough initiator, to achieve the minimum time for the reaction. However the system would be unable to evacuate the energy produced and the temperature would increase far beyond its limits.

The heat released by the reaction is evacuated somehow in three ways:

- Direct cooling in a condenser. The flow through the condenser happens by natural convection. The flow through the condenser cannot be measured with the available equipments.
- Losses to the surroundings.
- Heating the incoming flow rate of the monomer until the average temperature in the reactor.

Since the real plant cannot be used to perform experiments a model has been developed on which the optimisation algorithms can be applied. The details of this model can be found in a previous paper.

3. The model

The control strategy is depicted in figure 2. The controlled variables are the flow rate of initiator and the flow rate of monomer. The duration of the reaction depends mainly on the addition of monomer. The fastest we add the monomer, the sooner we finish the reaction. The control of the initiator is related to keeping appropriate conditions in the reactor, mainly the temperature.
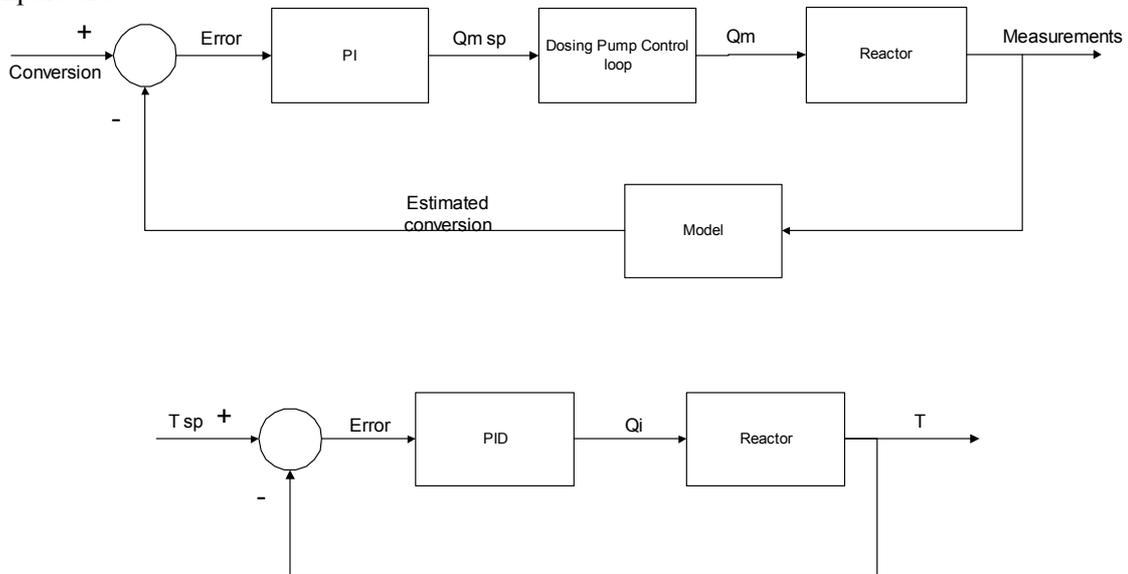
Figure 2: Control loops

The final objective is to reduce the total duration of the reaction. The maximum reduction would be achieved by adding all of the monomer at the start of the reaction. There are two reasons why we can not do so. The first reason is that the final quality of the product would very likely be affected. The reaction would become uncontrollable and the final characteristics of the product difficult to predict. The second reason is the limitation of temperature. The rate of production of heat would be much higher than the capacity of our installation to evacuate it. This is the main, hard constraint, which we will find when designing our control.

We have two loops of control which are deeply linked. The addition of the initiator will increase the consumption of the monomer and the temperature. On the other hand the addition of the monomer can cool the reactor too much leading to the addition of more initiator. We will manage this by giving priority to one of the loops and keeping the other as an accessory to the first.

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The main loop of control will handle the flow rate of the monomer. As can be seen in picture 1 the set point will be the maximum solubility of monomer in the water. The monomer is present in three forms in the emulsion. It can appear dissolved in the water, swelling the particles or forming its own separate phase, the droplets. Since the main locus of polymerisation is the particle phase, we want to keep the maximum concentration in this phase. To achieve that it would be necessary to keep the concentration in the water at its maximum, this is, close to the saturation point of the solution. However it is not advisable to surpass it, since the monomer will then form a separate phase. The presence of these droplets of monomer in the water will not increase the concentration of monomer in the aqueous phase nor in the particles, but can diminish the diffusion of the species which participate in the reaction. It is not possible nowadays to obtain an online measurement of the amount of monomer inside the reactor. A model, based on the energy balance, will be used to estimate the current amount of monomer and the partition between the aqueous phase and the particles. In the control of the addition of monomer we will also try to always maintain a conversion over 80%, what ensures the controllability of the reaction.

The addition of the initiator will be controlled similarly to what the operators are currently doing. The strategy will be the same, but the automation will have the reliability that is needed to keep the reaction under control with a variable flow rate of monomer. The set point will be a temperature of 353 K. The addition of initiator accelerates the reaction and consequently increases the temperature in the reactor. This effect is no immediate, so we have to wait some minutes to observe how the latest addition works. To avoid adding too much initiator together, the additions will be made in discrete fixed amounts, and the time between them will be at least 10 minutes.

We cannot test this strategy directly over the plant. First we will assess its performance on a model of the reaction. To provide the controller with some variables that are not directly measurable, we will use an additional model which will estimate the conditions inside the reactor. So in this simulation we will have three models working together: a model of the reaction, a model of the controller and the model that estimates the monomer present in the water. The whole set of equations for each of them is presented below. They have been developed with the mathematical modelling tool gPROMS.

3.1. The model of the reaction

This model is based on a more complex one presented in a previous paper [1]. That model had a great complexity, specially due to the equations involved in the calculation of the number of particles. As stated above the control strategy will be used only in the part of the reaction when we start to add monomer, keeping the initial conditions unchanged. As long as the number of particles becomes constant early in the reaction, we can start our reaction from a point where we consider that the number of particles is already stable. Apart from this simplification a few more changes have been introduced in order to improve the performance. The reduced model equations are shown below with a brief explanation where modifications have been introduced. For further details please refer to [1].

3.1.1. Aqueous phase
The initiator: The mass of initiator in the reactor (I) is increased by the flow rate of addition of initiator (I_e) and decreased by its thermal decomposition. To obtain the concentration of the initiator (I_w) in the aqueous phase we divide by the volume of water (V_w).

$$\frac{dI}{dt} = I_e - k_d [I_w] V_w$$

$$[I_w] = \frac{I}{V_w}$$

To account for the higher rate of decomposition of the KPS at higher temperatures we propose the use of the following quadratic function. Tstart is the temperature when an appreciable decomposition is observed.

$$K_d = 8.4 \cdot \left(\frac{T - Tstart}{40}\right)^2 + 1.6 \cdot \left(\frac{T - Tstart}{40}\right)$$

(3)

The polyvinyl alcohol: It gives stability to the emulsion. Its balance is expressed by the Langmuir model based on adsorption isotherms.

$$V_w[G_w] + K_{gd}[G_w]V_d + \frac{A_{c\infty}K_{ad}[G_w]A_{ps}}{1 + K_{ad}[G_w]} = \frac{G_0}{MW_{PVOH}}$$

where $[G_w]$ is the concentration of PVOH in the aqueous phase. Using the same method we obtain the number of molecules of alcohol per square centimetre (A_c):

$$A_c = \frac{A_{c\infty}K_{ad}[G_w]N_A}{1 + K_{ad}[G_w]}$$

The radicals: Once the number of particles becomes constant, the rate of polymerisation depends mainly on the number of radicals per particle and the concentration of monomer on the particles. In our case we start the simulation when the number of particles is nearly constant, so the modelling focus will be on the other two variables. $R_w\cdot$ is the total amount of radicals in the reactor, composed of those generated directly from the decomposition of the radical (and subsequent propagation), $RM\cdot$, and those yielded by transfer to monomer reactions, $M\cdot$. In all the equations related to the radicals the quasy steady state assumption has been considered.

$$R_w\cdot = RM\cdot + M\cdot$$

The $R\cdot$ radicals are obtained from the decomposition of the initiator. They are consumed in propagation (k_{iwm}) and transfer to monomer (k_{trm}) reactions. With the addition of new molecules of monomer the oligomers $RM_i\cdot$ are formed. The capture of the oligomers by the particles (k_c) is also taken into account.

$$R\cdot = \frac{2 f k_d [I_w]}{k_{iwm}[M_w] + k_{trm}[M_w]}$$

$$RM_1\cdot = \frac{k_{iwm}[M_w]}{(k_{pw} + k_{trm})[M_w] + k_{tw}R_w\cdot + k_C \dfrac{N_P}{V_w}} R\cdot$$

(8)

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$$RM_i := \frac{k_{pw}[M_w]}{(k_{pw} + k_{trm})[M_w] + k_{tw}R_w \cdot + k_C \dfrac{\sum\limits_{i=2}^{NoC} PSD_i}{V_w}} RM_{i-1} \cdot \quad \text{for i = 2, 3, ... , z-1}$$

$$RM := \sum_1^{z-1} RM_i \cdot$$

In a similar way we can obtain the oligomers formed by chain transfer to monomer reactions.

$$M_1 := \frac{K_{trm}[M_w](R_w \cdot - RM_1 \cdot) + R_{desorp}\dfrac{Np}{V_w}}{k'_{iwm}[M_w] + k_{tw}R_w \cdot + k_C \dfrac{Np}{V_w}}$$

$$M_2 := \frac{k'_{iwm}[M_w]}{(k_{pw} + k_{trm})[M_w] + k_{tw}R_w \cdot + k_C \dfrac{\sum\limits_{i=2}^{NoC} PSD_i}{V_w}} M_1 \cdot$$

$$M_i := \frac{k_{pw}[M_w]}{(k_{pw} + k_{trm})[M_w] + k_{tw}R_w \cdot + k_C \dfrac{Np}{V_w}} M_{i-1} \cdot \quad \text{for i = 3, 4, ..., z-1}$$

$$M := \sum_1^{z-1} M_i \cdot$$

Particle balance: Since we start the simulation at a point where the coagulation effects are much reduced. the balance of particles has been omitted. The final number of particles is used during the whole simulation.

Monomer balance: The total amount of monomer in the water, in moles, is expressed by the following equation.

$$\frac{dM}{dt} = Q_m - M_w\left(k_{iwm}R \cdot + k_{pw}(R_w \cdot - M_1 \cdot) + k'_{iwm}M_1 \cdot + k_{trm}(R_w \cdot - M_1 \cdot)\right) + k_{pp}[M_p]\bar{n}N_pV_w$$

This monomer can be found in three different phases: the aqueous phase, the particles phase and the droplets. When the concentration of monomer in the water is higher than its solubility, the part of the monomer that cannot be dissolved forms a separate phase. The following equations lead to the calculation of the partition of the monomer between the different phases.
First we determine the partition of the monomer between the aqueous phase and the particles by the following equations:

$$M_{wt} = M - M_p$$

$$M_{pf} = \frac{13.7 \cdot (M_{wf} \cdot 100)^2}{100}$$

$$M_{pol} = \frac{M_{pf} \cdot V_{ps} \cdot \rho_m \cdot \rho_p}{MW_m \cdot (\rho_p \cdot M_{pf} + (1 - M_{pf}) \cdot \rho_m)}$$

$$M_{wf} = \frac{[M_w] \cdot MW_m}{\rho_w}$$

$$[M_p] = \frac{M_p}{V_{ps}}$$

$$[M_w] = \frac{M_w}{V_w}$$

$$\phi = \frac{M_p \cdot MW_m}{\rho_m \cdot V_{ps}}$$

Once we found these values we can predict whether the monomer in the water form a different phase or not. If the amount of monomer in the water (Mwt) is higher than the solubility limit (Mws) the difference will form droplets.

$$M_w = M_{ws}$$

$$M_d = M_{wt} - M_w$$

Otherwise all of the monomer will be dissolved in the water.

$$M_w = M_{wt}$$

$$M_d = 0$$

The total amount of monomer that can be dissolved in the water is given by the following expression:

$$M_{ws} = \frac{0.025 V_w \varphi_w}{MW_m}$$

Polymeric phase: During the part of the reaction we are considering, the polymerisation takes place mainly in the particles. The polymerisation rate in the particles is expressed by the following term: $K_{pp}[M_p]\tilde{n}N_pV_w$. As we already mentioned the number of particles the number of particles is considered constant, $[M_p]$ has been already calculated and the only term we still have to estimate is $\tilde{n}$, the average number of radicals per particle. Three are the processes involved in the determination of this variable: the entry of the radicals into the particles, the desorption from the particles and the termination inside the particles.

$$\frac{dn}{dt} = R_{entry} - R_{desorp} - 2 \cdot R_{term}$$

The calculation of the terms representing the entry, desorption and termination is shown below.

$$R_{entry} = K_c \cdot \sum_{i=8}^{z-1} [RM_i \cdot + M_i \cdot]$$

$$K_c = 0,002 \cdot \pi \cdot D_{ps} \cdot N_A \cdot D_{wr}$$

$$\frac{dM'_1}{dt} \cdot = K_{trm} \cdot n_2 \cdot [M_p]$$

$$R_{desorp} = \frac{K_{desorp} \cdot K_{trm} \cdot n_2 \cdot [M_p]}{K_{desorp} + K_{pp} \cdot [M_p]}$$

$$K_{desorp} = \frac{12 \cdot D_{wr}}{D_{ps}^2}$$

$$D_{wr} = \frac{K_B \cdot T}{6 \cdot \pi \cdot \upsilon \cdot R_{oli}}$$

$$R_{term} = \frac{K_{tp} \cdot n_2^2}{v_s \cdot N_A}$$

Volume balance: The volume of water is constant, since we only add water in the beginning of the reaction. The volume of the species present in the aqueous phase is considered separately.

$$V_R = V_w + V_{ps} + V_{mw} + V_G$$

The volumes of the monomer and polymer are calculated from their molar masses and densities.

$$V_{mw} = \frac{(M_w + M_d) \cdot MW_m}{\varphi_m}$$

The volume of the particles is the sum of the volume of the polymer and the monomer surrounding it.

$$V_{ps} = v_u N_P + V_{mp}$$

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$$V_{mp} = \frac{M_p \cdot MW_m}{\varphi_m}$$

The average diameter of the particles is calculated from the monomer converted (MC). The rest of the geometrical characteristics of the particle are straightforward.

$$D_{pu} = \left(\frac{6000 M_c MW_m}{\pi \rho_p N_P} \right)^{1/3}$$

$$v_u = \frac{\pi \cdot D_{pu}^3}{6000}$$

$$v_s = \frac{V_{ps}}{N_p}$$

$$D_{ps} = \left(\frac{6000 \cdot v_s}{\pi} \right)^{1/3}$$

$$A_s = \pi \cdot D_{ps}^2 \cdot N_p$$

Model outputs: The solids content is expressed as the sum of the weights of the polymer and the PVOH divided by the total weight of the latex.

$$SC = \frac{M_c MW_m + G_0}{M_t MW_m + G_0 + V_w}$$

The viscosity is estimated from the following expression, given by Immanuel and Doyle [25].

$$\upsilon = \frac{\upsilon_0}{\left(1 - \dfrac{sc}{sc_{ref}} \right)^2}$$

Finally, the conversion is, by definition, the amount of monomer converted, divided by the total amount of monomer added to the reactor.

$$x = 100 \cdot \frac{M_c}{M_t}$$

Where M_t is the total amount of monomer added into the reactor and M_c is the monomer converted into polymer, expressed as the difference between the monomer added and the remaining monomer.

$$\frac{dM_t}{dt} = Q_m$$

$$M_c = M_t - M$$

3.2. Estimator of the conditions in the reactor

The following model is based on an energy balance of the reactor. The net production of heat in the reactor is calculated and the rate of polymerisation is calculated from it. With the rate of consumption of monomer and the flow rate of addition, the total amount of monomer in the reactor can be calculated. The partition of the monomer between the different phases can be obtained in the same way as in the previous model.

A list of the variables and their units is shown below:

Name	Description	Units
X	Global conversion	%
C_{pm}	Specific heat of vinyl acetate	J g^{-1} K^{-1}
C_{pp}	Specific heat of polyvinyl acetate	J g^{-1} K^{-1}
C_{pG}	Specific heat of polyvinyl alcohol	J g^{-1} K^{-1}
C_{pm}	Specific heat of water	J g^{-1} K^{-1}
G_o	Initial mass of polyvinyl alcohol	g
K_r	Heat capacity of the reactor	J/K
Q_m	Inlet flow of monomers	mol s^{-1}
Q_{HW}	Water mass flow through the heating jacket	g/s
Q_{HC}	Water mass flow through the condenser	g/s
M_{wt}	Total moles of monomer in the aqueous phase	mol
M_c	Total moles of monomer charged in the reactor	mol
M_{conv}	Moles of monomer converted into polymer	mol
M	Moles of monomer in the reactor	mol
M_{wf}	Weight fraction of monomer in the aqueous phase	Adim.
M_{pf}	Weight fraction of monomer in the particle phase	Adim.
M_w	Moles of monomer dissolved in the aqueous phase	mol
$[M_w]$	Concentration of monomer dissolved in the aqueous phase	mol l-aq^{-1}
M_{ws}	Moles of monomer dissolved in the aqueous phase at saturation	mol
M_d	Moles of monomer in droplets	mol
M_p	Moles of monomer in the polymer phase	mol
$[M_p]$	Concentration of monomer in the polymer phase	mol l^{-1}
T	Temperature	K
V_{mp}	Volume of the monomer in the particle phase	l
V_u	Total volume of the particles (unswollen)	l
V_w	Volume of the water	l
W_o	Initial mass of water	g
ΔH_R	Rate of change in the reactor enthalpy	J/s
ΔH_H	Rate of change in the reactor enthalpy due to the heating jacket	J/s
ΔH_C	Rate of change in the reactor enthalpy due to the condenser	J/s
ΔH_L	Rate of change in the reactor enthalpy due to losses to the surroundings	J/s

The parameters are shown below:

Name	Description	Units	Reference	Value
K_{pp}	Rate constant for propagation in the polymer phase	$l\ mol^{-1}\ s^{-1}$	17	3700
ρ_m	Monomer density	$g\ l^{-1}$	Product sp.	800
MW_I	Molecular weight of initiator	$g\ gmol^{-1}$	Product sp.	270.3
MW_m	Molecular weight of monomer	$g\ gmol^{-1}$	Product sp.	86.09
MW_E	Molecular weight of emulsifier	$g\ gmol^{-1}$	Product sp.	205000
MW_w	Molecular weight of water	$g\ gmol^{-1}$	-	18
N_A	Avogadro's number	-	-	6.02E+23
ρ_p	Polymer density	$g\ l^{-1}$	Polymer Handbook	1170
ρ_w	Water density	$g\ l^{-1}$	-	1000

Energy balance: We want to calculate the amount of heat released by the polymerisation reaction. The rest of the terms involved in the energy balance are known or estimated.

The total enthalpy in the reactor is the total heat capacity multiplied by the change in the temperature.

$$\Delta H_R = K_t \Delta T$$

The heat capacity of the reactor is calculated from the specific heat capacities and the masses of each of the components present in the reactor.

$$K_r = M \cdot MW_m \cdot C_{pm} + M_c \cdot MW_m \cdot C_{pp} + G_o \cdot C_{pG} + W_o \cdot C_{pw}$$

The enthalpy exchanged with the heating jacket is negligible since we are not heating during the period of the reaction considered. The heat exchanged in the condenser can be the difference between the inlet and outlet temperatures (both of them measured), multiplied by the mass flow of the water and the its specific heat.

$$\Delta H_C = (T_{CI} - T_{CO}) \cdot Q_{CW} \cdot C_{pw}$$

Another source of enthalpy for the reactor is the addition of monomer. The monomer comes from underground tanks at an almost constant temperature that contains the amount of heat given by the following equation.

$$\Delta H_M = Q_M \cdot MW_m \cdot C_{pm} \cdot T_m$$

The losses to the surroundings have been estimated. They are directly proportional to the difference between the temperatures in the reactor and outside. A simple equation has been proposed where K1 is an adjustable parameter that includes all the variables affecting the heat exchange, such as the area of the reactor, the materials from which it is built, the width of the heating jacket surrounding it… K1 has been estimated from the profile of the temperature at the end of the reaction, where polymerisation is not taking place, the addition of monomer is zero and the condenser is not cooling anymore. In this situation we can consider that the decrease in the temperature is entirely due to the losses to the surroundings, and easily estimate K1.

$$\Delta H_L = K1 \cdot (T_R - T_{EXT})$$

3.3. Monomer balance.

The monomer converted is calculated from the net heat produced in the reactor in moles per second divided by the heat of polymerisation in calories per mole.

$$\frac{dM_c}{dt} = \frac{\Delta H_R - \Delta H_C - \Delta H_J - \Delta H_L}{\Delta H_p}$$

The total mass, in moles, added in the reactor can be described by the following differential equation.

$$\frac{dM_t}{dt} = Q_m$$

The moles of monomer in the water (M_{wt}) are calculated as the difference between the total mass of monomer in the reactor and the monomer swelling the particles (M_p).

$$M_{wt} = M - M_p$$

The amount of monomer in the water (M_{wt}) is compared to the maximum amount that can be dissolved in the water that is present in the reactor (M_{ws}). In the case when M_{wt} is greater than M_{ws} the monomer dissolved in the water (M_w) will be equal to the saturation level (M_{ws}), and the remainder will form droplets.

$$M_w = M_{ws}$$

$$M_d = M_{wt} - M_w$$

When monomer droplets disappear the following equations apply.

$$M_w = M_{wt}$$

$$M_d = 0$$

The maximum number of moles that can be dissolved in a volume V_w of water is expressed as:

$$M_{ws} = \frac{0.025 V_w \varphi_w}{MW_m}$$

The monomer concentration in the aqueous phase, excluding the monomer in droplets, is:

$$[M_w] = \frac{M_w}{V_w}$$

The difference between the monomer added and the monomer converted into polymer is the monomer remaining in the reactor

$$M = M_t - M_c$$

The monomer in the particles maintains an equilibrium with that dissolved in the aqueous phase.

$$M_{pf} = \frac{13.7 \cdot (M_{wf} \cdot 100)^2}{100}$$

Where M_{pf} is the weight fraction of monomer in the polymer phase and M_{wf} is the weight fraction of monomer in the aqueous phase.

The moles of monomer in the particles (M_p) can be easily derived from the weight fraction, as follows:

$$M_{pol} = \frac{M_{pf} \cdot V_{ps} \cdot \rho_m \cdot \rho_p}{MW_m \cdot (\rho_p \cdot M_{pf} + (1 - M_{pf}) \cdot \rho_m)}$$

The expressions for the calculation of M_{wf}, the concentration of monomer in the particles are straightforward.

$$M_{wf} = \frac{[M_w] \cdot MW_m}{\rho_w}$$

$$[M_p] = \frac{M_p}{V_{ps}}$$

$$V_{mp} = \frac{M_p \cdot MW_m}{\varphi_m}$$

We also need to obtain some geometrical features of the particles which are involved in the calculation of the amount of monomer swelling the particles. The total volume of the unswollen particles can be obtained from the amount of monomer converted.

$$V_u = \frac{M_c MW_m}{\rho_p}$$

The total volume of the particles is that of the unswollen particles plus the monomer surrounding them.

$$V_{ps} = V_u + V_{mp}$$

The conversion is the percentage of the total monomer (Mt) that has been converted (Mc).

$$x = 100 \cdot \frac{M_c}{M_t}$$

3.4. The Controllers

The two controllers, the one for the addition of the monomer and the control of the addition of initiator, are deeply interrelated. The addition of monomer tends to decrease the temperature since the temperature of the incoming monomer is below the reactor temperature. The temperature directly affects the rate of decomposition of the initiator. On the other hand the addition of the initiator increases both the temperature and the consumption of monomer.

There are different ways to deal with this situation. In this case we will decouple the loops of control by giving one of them the highest priority. The controller of the addition of monomer will be a relatively fast PI, so that we keep the monomer concentration within the limits imposed by the set point and the other constraints at any moment.

The addition of the initiator will be done in discrete intervals. We will add a certain amount of initiator whenever the temperature is below the set point and a certain "security" time has passed since the last addition. Doing so we know the effects of the last addition of initiator before proceeding to add more. It also allows making the addition of the monomer more reliable and effective.

3.4.1. Monomer control

In the reactor we can find the monomer in three different phases. It can be dissolved in the water up to its limit of solubility (aqueous phase). From the water it diffuses to the particles, swelling them (particle phase). If the amount of monomer in the water is higher than its solubility, then the monomer forms a different phase, the so called droplets. To have the highest polymerisation rate possible we need to have the maximum concentration of monomer in the particles, which are the main locus of polymerisation. We can achieve this by keeping the concentration of the monomer dissolved in the water at its maximum, but without forming droplets that interfere in the diffusion of the reactants. So the set point would be the maximum solubility of monomer, and the input to the controller the concentration of the monomer in the aqueous phase. Since the concentration of the monomer cannot be measured online, this variable has to be estimated from the other measurements available. For this purpose the model described in point 3.2 will be used. However other restrictions apply that prevent us from ever reaching the set point in the real control. Since in this part of our work we are simulating the effects of the controller on a model of the reaction, there are uncertainties that we cannot obviate. The reactor has been modelled based on experimental data in a narrow range of temperatures and conversions. If the simulation takes the conditions in the reactor far from the modelling point, the results may be unrealistic. We also have limitations given by the installation, specifically in the maximum flow rate of monomer we can provide. This results in the following constraints that will apply during the whole reaction:

- The conversion in the reactor will not be allowed to go below 80%.
- The maximum flow rate of the monomer will be limited to 1000 Kg/h.

Some simulations were performed with this kind of control. It could be seen that keeping the conversion over 80% was a harder constraint than keeping the concentration of the monomer in the water closed to the saturation point. As can be seen in figure 3 the saturated solution of monomer was never reached, and the constraints acted much before. The flow rate of the monomer was at its maximum trying to reach the set point. Before we could reach the it the conversion would go below 80% and the output of the controller was set to zero. This strategy of control resulted in a on-off control of the pump that could result harmful for it.
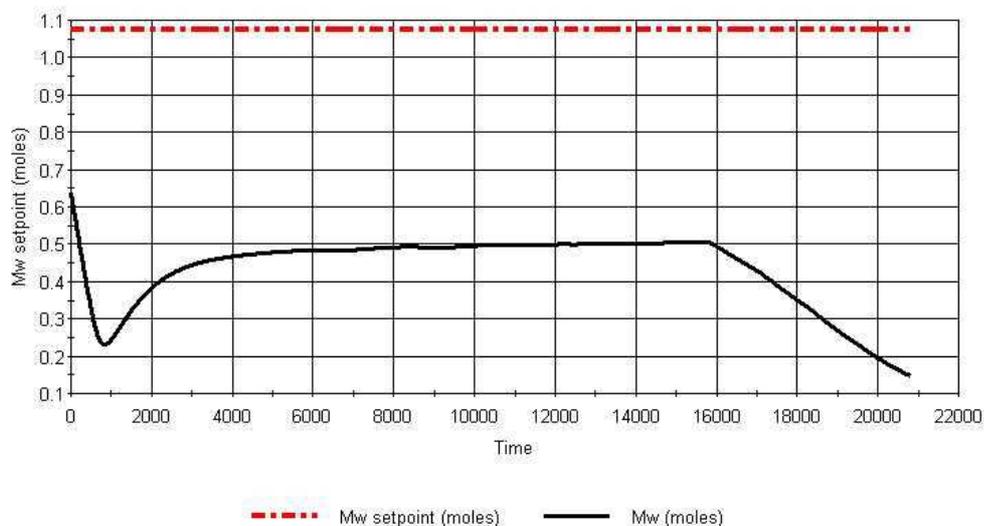
Figure 3: Moles of monomer in the water and set point

Since the real limitation has been proved to be the conversion, a second strategy has been designed (figure 2). The objective is to have a smoother control of the pump. A PI controller has been experimentally adjusted.

3.4.2. Initiator control

The addition of the initiator is used to control the temperature in the reactor. The control will replicate the control currently manually performed by the operator, but the reliability and repeatability will be higher, what allows us to have a variable flow rate of monomer addition.

When the temperature decreases we will add initiator whenever the security time has passed since the latest addition. The addition of the initiator will be done in fixed amounts of 1 litre of solution 2.4% in weight. The security time has been set experimentally to 10 minutes, what has proved to be enough to observe the effects of the previous addition.

Several attempts have been made to estimate the temperature in the reactor without good results. A complete energy balance such as the one presented in the model used for the estimation of the conditions of the reaction. To obtain an estimation of the temperature inside the reactor it is absolutely necessary to know the flow rate through the condenser. It is a natural convection driven flow, no measured in the plant. Even if its total value could be measured or estimated, the composition also plays a key role.

Given this limitation a new approach has been considered to run the simulations. In figure 4 the rate of polymerisation is plotted against a binary signal that shows 1 when the temperature is rising and 0 when it is decreasing. It can be deduced from this plot, that there is a certain value of the rate of polymerisation (and so the heat production in the reactor) that saturates the capacity of the condenser, and makes the temperature rise. The idea is to control the addition of the initiator in order to keep the average of the rate of polymerisation below this value. This value has been chosen in a very conservative manner, because it is necessary to ensure that the temperature can be kept within its limits at any moment. Quite probably the rate of polymerisation could be higher during the reaction and its duration will be shorter once implemented, but never longer.
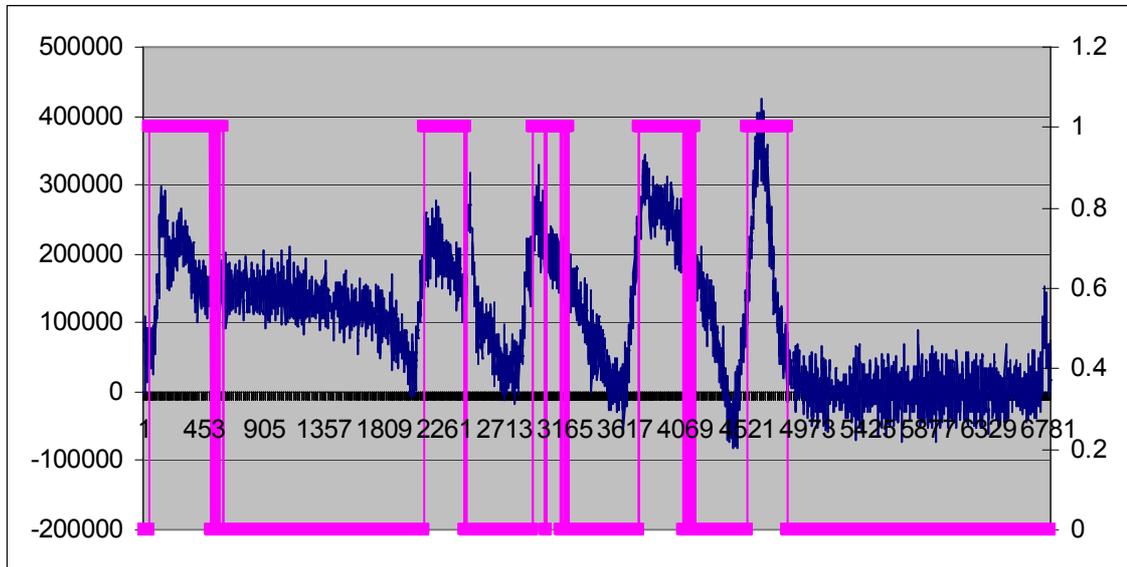
Figure 4: Rate of polymerisation and sign of the derivative of the temperature

4. Simulation results

Simulations have been run in order to adjust the controller and test its performance. The very conservative values chosen for the rate of polymerisation will ensure the feasibility of the results achieved. It is possible that the implementation in the real life reactor, with a control of the addition of the initiator directly based on the temperature, will yield even better results.

In figure 5 the flow rate of the monomer is depicted. In figure 6 we can observe the discrete additions of the initiator.
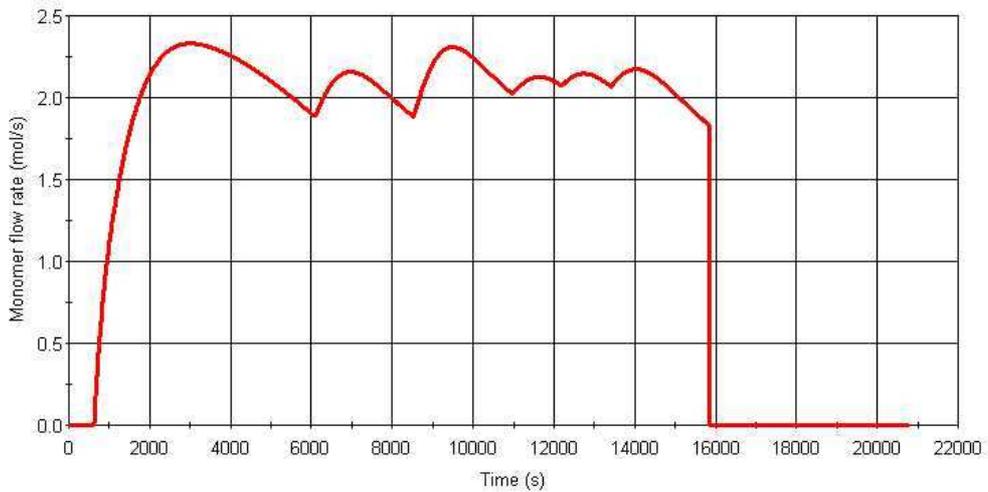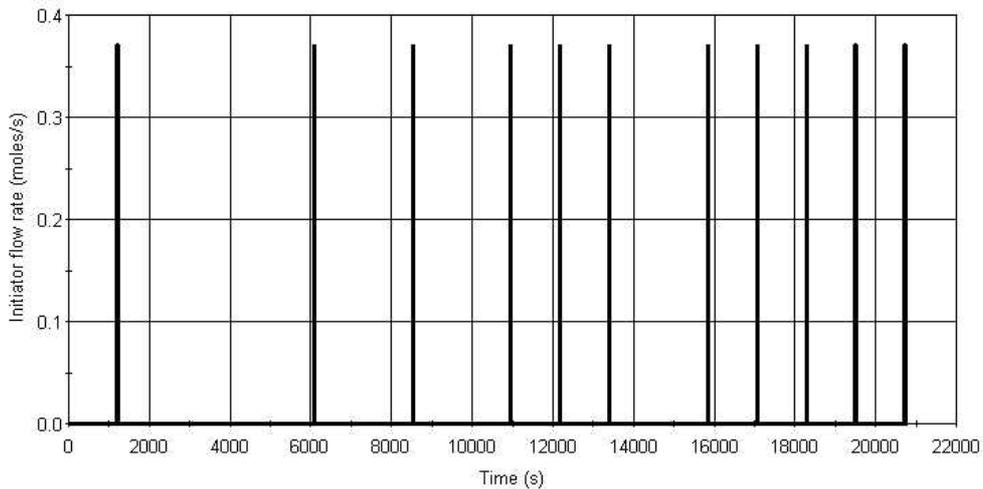

Figure 5: Flow rate of monomer

Figure 6: Flow rate of initiator

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The conversion is shown in figure 7. After an initial time during which the conversion reaches higher values the addition of the initiator adjust the conversion to the set point, at 80%. When all of the monomer has been added the conversion rapidly reaches the objective value of 98%, when we consider the reaction finished. The time of the reaction is counted from the start of the addition of monomer and until the conversion reaches 98%.
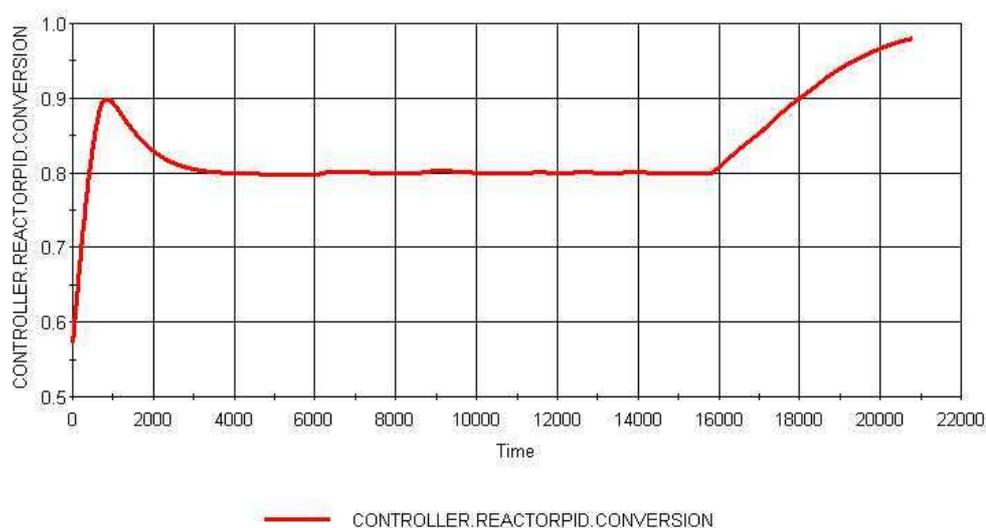

Figure 7: Conversion

The time of the reaction has been reduced from 23250 s. (approximately 6 hours and a half) to 20820 (around 5h 45 m). The reduction achieved is greater than 10%. It is important to mention as well that the control of the initiator addition is very conservative, in order to ensure that the temperature and conversion constraints are kept. Further adjustment of the controller once implemented in the real plant would surely improve the productivity even more.

5. Conclusions

A control strategy for the polymerisation process has been designed. The outputs of the controller will be the flow rate of initiator and the flow rate of the monomer. The inputs are the conversion and the polymerisation rate. These variables cannot be measured on-line, so a simple model based on the energy balance has been developed to estimate it.

Two different control strategies for the addition of the monomer have been developed. A PI controller with a conversion of 80% as the set point has been finally chosen. The initiator is added in discrete additions replicating the logic currently followed by the operators. Several temperatures are collected: the inlet and outlet temperatures to the condenser, both in the product and the water sides, the temperature inside the reactor.

The resulting control strategy has been validated off-line, based on the previously developed model, and will be implemented in the factory. The strategy has proved that the reaction time can be reduced by ten per cent. However a very conservative strategy has been used, and further adjustment of the controller, once installed in the plant, may yield a greater reduction.

6. References

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