CHEMICAL ENGINEERING TRANSACTIONS
VOL. 39, 2014
A publication of AIDIC
The Italian Association of Chemical Engineering
www.aidic.it/cet
Guest Editors: Petar Sabev Varbanov, Jiří Jaromír Klemeš, Peng Yen Liew, Jun Yow Yong

**ISBN** 978-88-95608-30-3; **ISSN** 2283-9216
DOI: 10.3303/CET1439072

# Design of Model Predictive Control for Butyl Acetate Production in Reactive Distillation

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An efficient operation of the reactive distillation is challenging due to its complex dynamics resulting from the coupling of reaction and separation tasks into a single unit. Conventional control schemes cannot always handle with highly nonlinear and multivariable systems. Therefore, this study considers the implementation of a model predictive control (MPC) to the reactive distillation column in which the esterification of butanol and acetic acid to produce butyl acetate is chosen as a case study. The control objective is to maintain the butyl acetate product at a desired purity without the accumulation of unreacted reactants in the reactive zone of the reactive distillation. Two alternative control structures of MPC controller are proposed. The first one is based on the two-point temperature control (CS1), whereas the second one involves the one-point temperature control and the direct control of the acetic acid composition (CS2) within the reactive distillation. It is found that the MPC with CS2 structure shows a better performance under disturbance rejection and set-point tracking case studies; faster and smoother responses with less oscillation are observed.

## 1. Introduction

Butyl acetate is widely used in chemical industries as an intermediate in organic synthesis and a solvent for paint and coatings. Generally, butyl acetate is produced directly from acetic acid and butanol via an esterification reaction (Arpornwichanop et al., 2008). This reversible reaction needs to be catalyzed by acidic agents. Conventional processes for the production of butyl acetate consist of a packed-bed reactor, where the esterification of acetic acid and butanol is occurred, connected with a train of distillation columns for product separation. In general, the conversion of acetic acid in the reactor is limited by chemical equilibrium. In addition, separation of butyl acetate as a desired product from the solution mixture is a difficult task, requiring the column with many stages and high energy consumption.
There are a number of research efforts concerning the use of a reactive distillation (RD) for equilibrium-limited reactions (Vlad and Bildea, 2012). RD combines both the chemical reaction and separation tasks into single unit operation and offers several advantages over the traditional approach of reaction processes followed by separations (Simasatitkul et al., 2011). The direct removal of products during reactions proceeding increases both the reactant conversion and selectivity. Furthermore, a higher energy efficiency could be achieved in case of exothermic reaction systems because the heat generated by reactions reduces the reboiler heat input to the column (Modla and Lang, 2013).
Operation and control of the RD has known to be much more difficult than conventional processes in which the reactant conversion and the product purity can be controlled separately. The interaction of reaction and separation makes the RD column exhibit complex behavior, such as, multi-steady state condition, high nonlinearity and strong interactions of process variables (Yamaki and Matsuda, 2012). Kaymak and Luyben (2005) proposed investigated two-temperature control structures for reactive distillation column and found that election of the manipulated fresh feed stream has an important role in the stability of the system. Presently, a conventional control is still widely used because of its simplicity and robustness;

Please cite this article as: Komkrajang T., Kheawhom S., Paengjuntuek W., Arpornwichanop A., 2014, Design of model predictive control for butyl acetate production in reactive distillation, Chemical Engineering Transactions, 39, 427-432 DOI:10.3303/CET1439072

however, the performance of PID control is degraded when facing highly nonlinear processes (Modla and Lang, 2013).

This study is focused on the implementation of a model predictive control to a reactive distillation for the production of butyl acetate from esterification of acetic acid and butanol, as a case study Two alternative control structures are proposed to maintain the reactant conversion and product purity at desired conditions through two-point temperature control strategy and one-point temperature control coupled with the direct control of reactant composition. Performance of the MPC with two control structures is investigated via closed-loop dynamic simulations and compared with a PI control strategy under set point change and disturbance rejection cases.

## 2. Modelling of reactive distillation for butyl acetate production

### 2.1 Reactive distillation

Figure 1 shows the schematic diagram of the reactive distillation for butyl acetate production used in this work. The rectification and stripping zones operate exactly as a nonreactive distillation column to purify top and bottom products. Butyl acetate is formed in the reaction zone. The overhead vapour with a composition close to the heterogeneous ternary azeotrope of water, butanol and butyl acetate, is condensed and then separated into the aqueous and organic phases in a decanter. The aqueous phase is completely withdrawn, whereas the organic phase is refluxed to the column. Pure butyl acetate is removed at the bottom. The mathematical model of the reactive distillation is developed based material balance, phase equilibrium, mole fraction summations and heat balance equations, as summarized in Table 1.

### 2.2 Reaction kinetics

Butyl acetate is produced from the esterification reaction of acetic acid and butanol. Kinetic data for butyl acetate synthesis catalyzed by ion-exchange resins can be explained by the pseudohomogeneous and adsorption-based model - Eq(1) (Steinigeweg and Gmehling, 2002).

$$r = \frac{1}{m_{\text{cat}}} \frac{1}{\nu_i} \frac{dn_i}{dt} = k_1 a_{\text{acetic acid}} a_{\text{butanol}} - k_{-1} a_{\text{butyl acetate}} a_{\text{water}} \tag{1}$$

where $k_1$ and $k_{-1}$ are the forward and backward reaction rate constants following Arrhenius' law and $a_i$ is the liquid phase activity calculated by UNIQUAC model

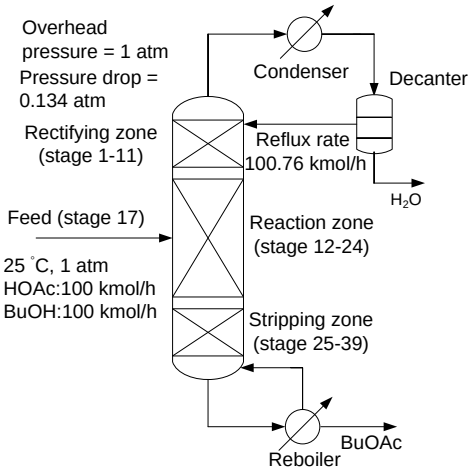


*Figure 1: Reactive distillation column for butyl acetate synthesis*

### 2.3 Steady-state analysis

The primary control objective of the RD is to produce n-butyl acetate with purity at its desired value. In order to reduce the expensive cost of an online composition measurement and the problem of its large measurement lag, an inferential control strategy based on stage temperature measurement is applied. The location of the temperature control within the column is determined through an open-loop sensitivity analysis; the temperature at the column stage showing nearly linear dynamic behaviour and high sensitivity to input changes is selected. Figure 2(a) shows the relations of stage temperatures and reboiler duty. It can be seen that the temperature at the column stage 37 can be good a candidate for the

controlled variable. Figure 2(b) shows the linear relationship of the butyl acetate purity at the bottom purity and the 37$^{th}$ stage temperature.

In order to prevent the accumulation of unreacted reactants in the reactive stages, the column feed have to be kept in a correct stoichiometric ratio. The simplest way to maintain this balance is to use a feed ratio control via a feed forward control scheme. However, when there is a measurement error or impurity in feed stream, the feed ratio control will not be able to maintain the stoichiometric balance. To overcome this difficulty, Al-Arfaj and Luyben (2000) suggested that the composition of reactants in some column stages should be controlled by the feed ratio. Due to an expensive on-line composition measurement, the reactive stage temperature is measured to infer the reactant composition in the reactive zone. From the sensitivity analysis of the RD column, it is found that the acetic acid composition and the temperature at stage 17 have a linear relation under the studied operating conditions. Therefore, the column temperature at the 17$^{th}$ stage is chosen as another controlled variable.

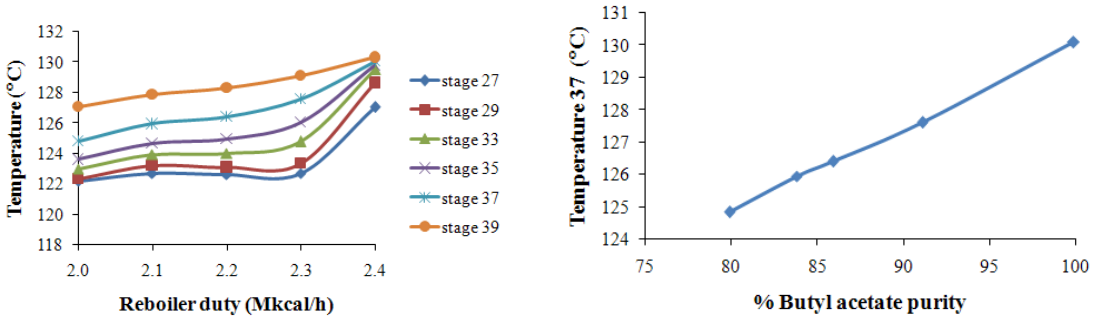


*Figure 2: (a) Effect of reboiler duty on stage temperatures and (b) Relation of butyl acetate purity and temperature at the 37$^{th}$ stage of the RD column*

## 3. Model predictive control

### 3.1 Model identification

The full nonlinear dynamic model of RD as mentioned above is complicated for direct use in a control system design. In this study, a simplified input-output process model is developed and employed in the formulation of a model predictive control (MPC). The RD model is solved and a step test is applied to generate input-output dynamic data from changes in reboiler duty and feed ratio. These data are then used to build the input-output dynamic model using the system identification toolbox in MATLAB. Table 2 summarizes the obtained transfer functions explaining the relationships of two inputs (i.e. reboiler duty and feed ratio) and three outputs (i.e., acetic acid composition at the 17$^{th}$ stage and temperatures at 17$^{th}$ and 37$^{th}$ stages).

### 3.2 Control configuration

Two alternative control structures of the RD for butyl acetate production are considered here. The first one is based on the two-point temperature control, whereas the second one involves the one-point temperature control and the direct control of the acetic acid composition within the reactive distillation. The control objective is to maintain the bottom product purity of butyl acetate at desired value. The disturbances include butanol feed flow rate and acetic acid feed composition. The first control structure CS1 is employed to control tray temperatures at the 17$^{th}$ and 37$^{th}$ stages by manipulating the feed ratio of acetic acid to butanol and the reboiler duty. The second control structure CS2 is applied to control the acetic acid composition at the 17$^{th}$ stage and the tray temperature at the 37$^{th}$ stage by manipulating the same inputs as in the CS1 control structure.

### 3.3 Controller setup

In this study, the formulation of a MPC problem for controlling the RD is performed using the model predictive control toolbox in Matlab. The simplified process model developed from the system identification mentioned above is converted to a linear time-invariant system and used in the MPC formulation based on the control interval of one min. The prediction and control horizons are set to be 100 and 5 steps ahead. Two manipulated variables are the reboiler duty and feed ratio, whereas two outputs include the acetic acid composition or temperature at stage 17 and temperature at stage 37. The reboiler duty can be changed from 1.8 to 2.6 Mkcal/h and feed ratio is limited by a range of 0.9 and 1.1.

*Table 1: Model of the reactive distillation column*

Stage *i*

$$\frac{dU_j}{dt} = V_{j+1} + L_{j-1} + F_j - V_j - L_j + \left(\sum_i v_i\right) r_j \quad (1)$$

$$\frac{dU_j x_{i,j}}{dt} = y_{i,j+1} V_{j+1} + x_{i,j-1} L_{j-1} + z_{i,j} F_j - y_{i,j} V_j - x_{i,j} L_j + v_i r_j \quad (2)$$

$$\frac{dU_j H_j}{dt} = V_{j+1} H_{j+1}^V + L_{j-1} H_{j-1}^L + F_j H_j^F - V_j H_j^V - L_j H_j^L - Q_j \quad (3)$$

Condenser

$$\frac{dU_1}{dt} = V_2 - L_1 \quad (4)$$

$$\frac{d(U_1 x_{i,1})}{dt} = y_{i,2} V_2 - x_{i,1} L_1 \quad (5)$$

$$\frac{d(U_1 H)}{dt} = V_2 H_2^V - L_1 H_1^L - Q_c \quad (6)$$

Reboiler

$$\frac{dU_N}{dt} = L_{N-1} - L_N - V_N \quad (7)$$

$$\frac{d(U_N x_{i,N})}{dt} = x_{i,N-1} L_{N-1} - x_{i,N} L_N - y_{i,N} V_N \quad (8)$$

$$\frac{d(U_N H)}{dt} = L_{N-1} H_{N-1}^L - L_N H_N^L - V_N H_N^V + Q_r \quad (9)$$

Decanter

$$L_1 = R + D \quad (10)$$

$$x_{i,1} L_1 = x_{i,1}^O R + x_{i,1}^A D \quad (11)$$

Vapor-liquid equilibrium relations

$$y_{i,j} P = \gamma_{i,j} x_{i,j} P_j^0 \quad (12)$$

Liquid-liquid equilibrium relations

$$\gamma_{i,j}^a x_{i,j}^a = \gamma_{i,j}^o x_{i,j}^o \quad (13)$$

Mole fraction summations

$$\sum x_{i,j} = 1 \qquad \sum y_{i,j} = 1 \quad (14)$$

*Table 2: Input-output model of the reactive distillation column*

| | Acetic acid composition at the 17th stage | Temperature at the 17th stage | Temperature at the 37th stage |
|---|---|---|---|
| Reboiler duty | $\frac{-0.335}{34.548s+1}$ | $\frac{33.413}{21.682s+1}$ | $\frac{16.737}{102.878s+1}$ |
| Feed ratio | $\frac{0.940}{57.42s+1}$ | $\frac{-71.491}{57.642s+1}$ | $\frac{18.228}{98.0267s+1}$ |

## 4. Results and discussion

Performances of the MPC controller with the control structures CS1 and CS2 are tested under disturbance rejection and set-point tracking cases. Feed composition and feed rate changes are the most unpredicted disturbances that affect the reactive distillation column. In addition, the set-point change in the 37th stage temperature is applied to the reactive distillation column.

### 4.1 Control structure CS1

Figure 3 shows the responses of the temperatures at the 17th and 37th stages under a step change in the butanol feed flow rate (±20 %). It is found that the MPC with CS1 structure can control the temperatures at their desired set points and the purity of butyl acetate product is maintained close to its steady state value. An increase in the butanol feed flow rate initially decreases tray temperatures within the RD. As a result of the increased butanol feed rate, high reboiler duty and low feed ratio are required to maintain the temperatures. In case of decreasing the flow rate of butanol feed (-20 %), the performance of the MPC

with CS1 structure is not good and an oscillatory response around the set points is found. However, the purity of butyl acetate product is kept within one percentage of the desired value.

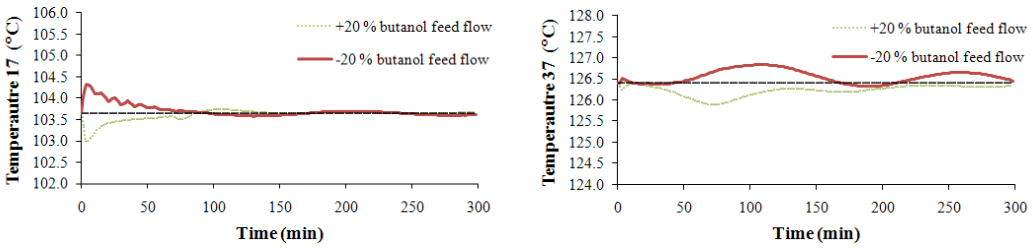


*Figure 3: Responses of the MPC controller with CS1 structure: changes in butanol feed flow*

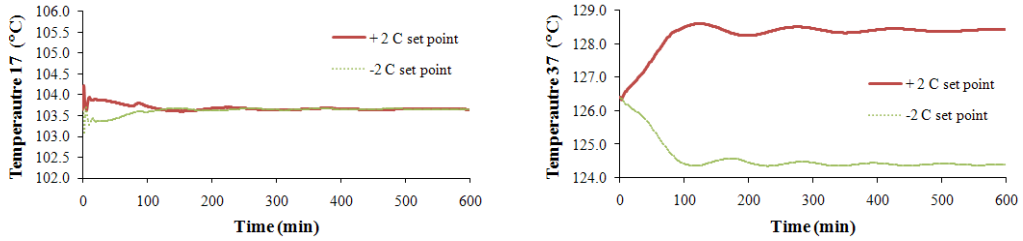


*Figure 4: Responses of the MPC controller with CS1 structure: changes in temperature set point*

For the set point change in the 37$^{th}$ stage temperature (±2 $^{o}$C), the MPC controller can track the new temperature set point quite well (Figure 4), although a small oscillation in the controlled variable is observed. It is noted that when the stage temperature increases, the purity of the bottom product, butyl acetate, increases. This implies that the temperature at the 37$^{th}$ stage can be used as a adjusting process variable for controlling the product purity.

**4.2 Control structure CS2**

The close-loop response of the MPC controller in CS2 structure is tested when the butanol feed flow rate (±20 %) is set as a process disturbance. The results show that the MPC controller can control the acetic acid composition at the 17$^{th}$ stage and temperature at the 37$^{th}$ stage close to their set-points without any oscillation. Because there is no direct control of the bottom product composition, the purity of butyl acetate product is slightly deviated from its original steady-state value. To reduce such the deviation in product purity, the temperature set-point at the 37$^{th}$ stage can be manually adjusted to get the product at desired purity. When comparing with CS1 structure, CS2 control structure gives smoother and faster responses to the disturbance in butanol feed flow. The direct control of acetic acid composition at the 17$^{th}$ stage (CS2 structure) instead of the inferential control of the 17$^{th}$ stage temperature (CS1 structure) can give better control performance. Because a change in butanol feed directly affect the acetic acid composition within RD column, the controller acknowledges this disturbance and adjust the feed ratio to maintain it accurately. For CS1 structure, the temperature at the stage does not depend only on a change in acetic acid composition, but relies on all the components.

**4.3 Comparison of MPC and PI controllers**

The previous control results show that MPC with the control structure CS2 provides good control performance for set point tracking and disturbance rejection cases. Based on the control structure CS2, the performance of MPC controller is compared to that of a conventional PI controller. Two PI controllers are employed to control the 37$^{th}$ stage temperature by manipulating the reboiler duty and the acetic acid composition at the 17$^{th}$ stage by manipulating the feed ratio. Tuning of the PI control parameters is based on IAE performance criteria and the optimal values of these parameters are: $K_{P1}$= 1.162 and $\tau_{I1}$= 0.12 min and $K_{P2}$ = 0.046 and $\tau_{I2}$=0.51 min. Constraints on the reboiler duty (*Q*) and feed ratio (*Fr*) are as follows: 1.8 ≤ Q (Mkcal/h) ≤ 2.6 and 0.9 ≤ Fr ≤ 1.1

Figure 5 shows the dynamic responses in case of the set-point change in the temperature at the 37$^{th}$ stage (±2 $^{o}$C). It is obvious that the MPC controller ensures fast and smooth response, while the PI controller shows overshoot and oscillation responses. When the flow rate of butanol is changed by ±20 % from its nominal value, it is found that performance of the MPC is superior to the PI controller; the temperature at

the 37th stage is less overshoot with faster settling time. However, for the control of acetic acid composition at stage 17, the PI controller can give much faster settling time than the MPC controller without offset in the controlled variable.

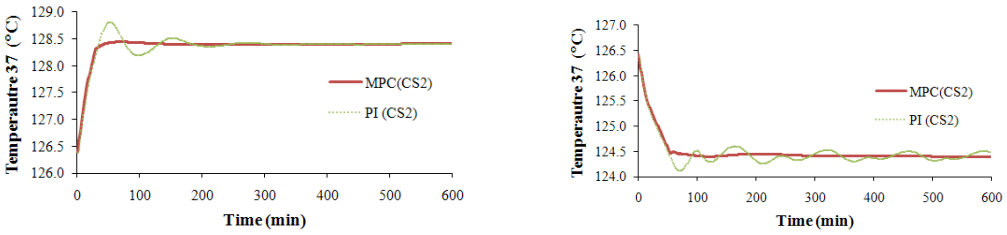


*Figure 5: Comparison of MPC and PI control of the reactive distillation*

## 5. Conclusions

In this work, the control of a reactive distillation (RD) for butyl acetate production was studied. Two alternative control structures using a model predictive control (MPC) were proposed. The control objectives are to maintain the bottom product composition at desired purity and to prevent the accumulation of unreacted reactants in the reactive zone of the RD. The first control structure CS1 uses the two-point temperature control to infer butyl acetate bottom product purity and acetic acid composition by adjusting reboiler heat duty and feed ratio, respectively. The second control structure CS2 uses the one-point temperature control to infer butyl acetate bottom product purity by manipulating reboiler heat duty and the direct control of acetic acid composition by manipulating feed ratio. The simulation results indicated that the MPC with CS2 structure shows a better performance under disturbance rejection and set-point tracking case studies; it gives faster and smoother responses with less oscillation. Based on the CS2 control structure, use of a conventional 2x2 PI control strategy for the RD was also studied. The PI control performs a good control of the acetic acid composition, but less attractive in the temperature control, which is the primary control objective.

### Acknowledgements

Support from the Graduate School and Ratchadaphiseksomphot Endowment Fund of Chulalongkorn University is gratefully acknowledged.

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