

# CONTROLLABILITY ANALYSIS OF A COMBINED BATCH REACTOR/BATCH DISTILLATION PROCESS

Eva Sørensen and Sigurd Skogestad\*

Chemical Engineering Department  
University of Trondheim-NTH  
N-7034 Trondheim, Norway

January 15, 1992

## Abstract

The paper addresses control of a batch chemical reactor with simultaneous distillation. A non-linear model of the process is developed. A linearised version of this model is used to analyze the controllability for different reactor conditions and times during a batch. Different alternatives for manipulated inputs and controlled variables are discussed, and various alternatives are compared based on controllability. In some cases the process becomes strongly interactive (large RGA-values) and feedback control is difficult. The batch reactor/column behaviour is compared with that of a conventional column.

## 1 Introduction

Batch distillation is one of the oldest separation processes known. It is used in the chemical industry for the production of small amounts of products with high added value and for processes where flexibility is needed, for example, when there are large variations in the feed composition. Batch reactors with distillation is used to improve the product yield.

Often the control objective when considering batch processes is either to i) minimise the

batch time or ii) maximise the product yield or quality. However, sometimes the control objective is simply to obtain the same conditions in each batch. This was the case for the specific industrial application which was the starting point for our interest in this problem and which is to be presented later.

Mathematical models of batch distillation processes without chemical reaction were published as early as in the sixties. Huckaba and Danly [1] presented the first significant simulation of a batch distillation column. The main assumptions made included binary system, constant weight holdups, linear enthalpy relationship, and adiabatic operation. Meadows [2] presented the first multicomponent batch distillation model. The solution of such models, using various numerical methods, was presented by Distefano [3]. Other contributions to improved model formulation and numerical solution methods have been presented by Domenech *et al.* [4] and [5], Gallun and Holland [6], Sadotomo and Miyahara [7] and Mujtaba and Macchietto [8].

Few authors have considered batch distillation with chemical reaction. Egly *et al.* [9] included the possibility of chemical reactions in the column. They presented a method for the optimisation of batch distillation based upon models which included the non-ideal behaviour of multicomponent mixtures and the kinetics

---

\*Author to whom correspondence should be addressed. E-mail: SKOGE@KJEMI.UNIT.NO, phone: 47-7-594154, fax: 47-7-594080.

of chemical reactions. The column operation was optimised by using the reflux ratio as a control function. A feed of one of the reactants during the reaction was also considered. In a later paper, Egly *et al.* [10] consider control of the column based on temperature measurements. Cuille and Reklaitis [11] formulated a model and solution strategies for the simulation of a staged batch distillation unit with chemical reaction in the liquid phase. Reuter *et al.* [12] incorporated the simulation of PI-controllers in their model of a batch column with reaction in the still. They mention that the model can be used for the investigation of control structure with the aid of Relative Gain Array analysis (RGA) but without giving any examples of this. Albet *et al.* [13] presented a method for the development of operational policies based on simulation strategies for multicomponent batch distillation applied to reactive and non-reactive systems.

In this work we have developed a model of an industrial process consisting of a batch reactor with a rectifying column on top. Based on a linearised version of this model we want to compare different operating points to show how the controllability differs; that is whether the process is easier to control at the beginning or at the end of a batch, if the same controller settings can be used for different reactor conditions or reactor temperatures etc. In the various operating points we have looked at the stability of the system and the response to step changes in flows. We have considered "one point control", when one part of the column/reactor is controlled, and "two-point control", when both the top and the bottom part are controlled. We have used the Relative Gain Array (RGA) analysis for the investigation of control structures in "two point control". We have also looked at the similarities and differences between our process and a conventional continuous distillation column.

## 2 Relative Gain Array analysis

The Relative Gain Array (RGA) has found widespread use as a measure of interactions and as a tool for control structure selection for single-loop controllers. It was originally defined at steady-state (Bristol [14]), but it may easily be extended to higher frequencies (Bristol [15]). Important advantages with the RGA is that it depends on the plant model only and that it is scaling independent. Most authors have confined themselves to use the RGA at steady state, and a thorough review of the use and interpretation of the steady state RGA is given by Grosdidier *et al.* [16]. Use of frequency dependent RGA for control structure selection is presented by Skogestad and Hovd [17].

## 3 Mathematical model

In this section we consider the mathematical description for the batch distillation column shown in Fig. 1.

The equations for the individual stages consist of the total mass balance, the mass balance for each component, tray hydraulics and phase equilibrium and are valid under the following assumptions :

- A1** A stage model is used for the distillation column.
- A2** Perfect mixing and equilibrium between vapour and liquid is assumed on all stages.
- A3** The vapour phase holdup is assumed to be negligible compared to the liquid phase holdup.
- A4** The stage pressures and the plate efficiencies are constant.
- A5** Constant molar flows.
- A6** Linear tray hydraulics is assumed.
- A7** The chemical reaction is limited to the reactor.
- A8** Raoult's law for the VLE.

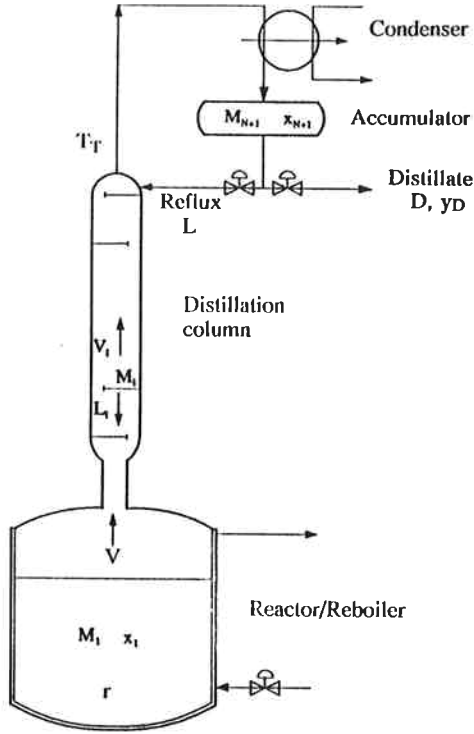


Figure 1: Batch distillation column/reactor.

The following differential and algebraic equations result.

reactor/reboiler,  $i=1$  :

$$dM_1/dt = L_2 - V + \sum \xi_j r \quad (1)$$

$$d(M_1 x_1)/dt = L_2 x_2 - V y_1 + \xi_1 r \quad (2)$$

column tray,  $i=2, N$  :

$$dM_i/dt = L_{i+1} - L_i \quad (3)$$

$$d(M_i x_i)/dt = L_{i+1} x_{i+1} + V y_{i-1} - L_i x_i - V y_i \quad (4)$$

accumulator,  $i=N+1$  :

$$dM_{n+1}/dt = V - L_{N+1} - D \quad (5)$$

$$d(M_{N+1} x_{N+1})/dt = V y_N - L_{N+1} y_D - D y_D \quad (6)$$

tray hydraulics :

$$L_i = L_{oi} + \frac{M_i - M_{oi}}{\tau} \quad (7)$$

liquid-vapour equilibrium :

$$y_i = \frac{\alpha_i x_i}{1 + (\alpha_i - 1) x_i} \quad (8)$$

relative volatility :

$$\alpha_i = f(T_i) = \frac{P_1^{sat}(T_i)}{P_2^{sat}(T_i)} \quad (9)$$

temperatures :

$$T_i = f(x_j, P) \quad (10)$$

On vector form the differential equation system to be solved can be written

$$dx/dt = f[x(t), u(t)] \quad (11)$$

In addition there is a set of algebraic equations, equations (7)-(10)

$$0 = g[x(t), u(t)] \quad (12)$$

### 3.1 Linear model

In order to investigate the controllability of a process using available tools a linear model is needed. Based on the non-linear model described by eq. (11) and (12) a linear model can be developed by linearising the equation system at a given operating point. For continuous processes there normally is only one operating point considered; that of the steady state conditions. The linear model is then found by linearising around this operating point. This model will be valid for small deviations from the steady state. When considering batch processes there is no steady state; the conditions in the reactor or column are changing with time. A linear model of a batch system will only be valid in the close vicinity of the operating point chosen.

A linearised model of the process can be described by the following equations

$$dx/dt = Ax + Bu$$

$$y = Cx$$

$$y = G(s)u \quad (13)$$

Where

$$\begin{aligned} \mathbf{x} &= [\Delta x_1, \Delta M_{1..}]^T \\ \mathbf{y} &= [\Delta M_D, \Delta y_D, \Delta T_B]^T \\ \mathbf{u} &= [\Delta D, \Delta L, \Delta V]^T \end{aligned} \quad (14)$$

The linear model represents deviations from "natural drift" with  $D$ ,  $L$  and  $V$  constant. The control problem will thus have the following controlled and manipulated variables :

Controlled variables ( $\mathbf{y}$ ) :

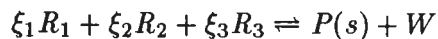
- condenser holdup  $M_D$
- distillate composition  $y_D$
- reactor temperature  $T_B$

Manipulated variables ( $\mathbf{u}$ ):

- distillate flow  $D$
- reflux flow  $L$
- vapour flow  $V$

## 4 Simulation results

The simulation results are presented with reference to the example of an equilibrium esterification reaction of the type



The reaction takes place in a reactor heated by a heating jacket with heat oil. The equilibrium is pushed towards the product side by distilling off the low boiling by-product ( $W$ ) from the reactor. Separation of the reactant  $R_2$  and the by-product  $W$  takes place in the column. The reaction rate is reported to be of zero order; independent of compositions. Due to lack of data we also assume it to be independent of temperature.

The industrial application which forms the basis for this paper can be represented by Fig. 2. The by-product is drawn off at the top of the column and the reactant is fed back to the reactor. The startup conditions are total reflux

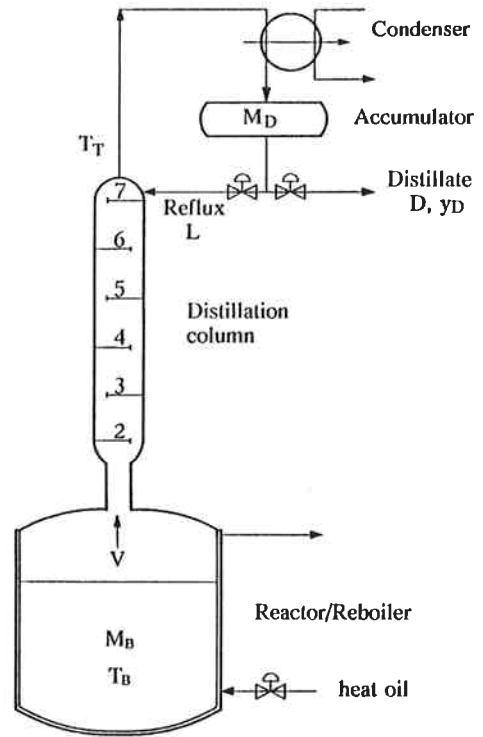


Figure 2: Batch distillation example.

Column:	6 trays + accumulator
Reaction:	$0.5 R_1 + 0.36 R_2 + 0.14 R_3 \rightarrow P(s) + W$
Volatile components:	$W$ and $R_2$
Relative volatility, $\alpha=f(T)$ :	8-32
Startup time:	30 min
Total reaction time:	15 h
Pressure in column and reactor:	1 atm/1.2 atm
Constant reaction rate, $r$ :	1.25 kmol/h
Initial vapour flow, $V$ :	16.8 kmol/h
Hydraulic time constant, $\tau$ :	0.0018 h
Initial holdups:	reactor: 24 kmol accumulator: 1.6 kmol trays: 0.09 kmol

Table 1: Process data for simulation.

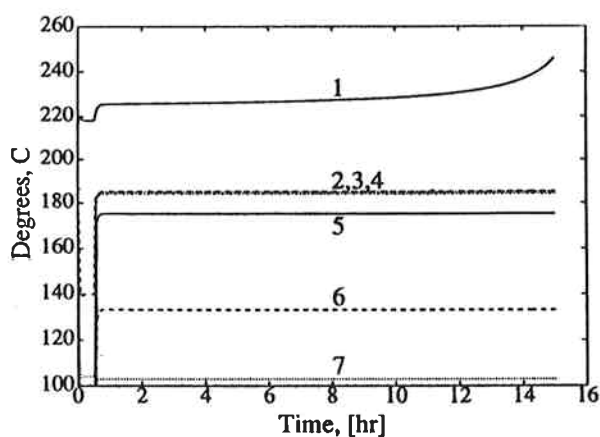


Figure 3: Today's temperature profile.

and no reaction. A summary of the process data is given in table 1.

Today's operating practice is to use "one-point control", that is the temperature at the top of the column,  $T_T$ , is kept constant at about  $103^\circ\text{C}$  which gives a distillate composition of 0.996 of the light component  $W$ . The vapour flow is kept constant by using maximum heating of the reactor and the accumulator level is controlled by the distillate flow  $D$ . The temperature profile in the column/reactor is given in Fig. 3. The reactor temperature is almost constant at the beginning but increases as reaction proceeds. The conditions on tray 2, 3 and 4 are practically equal because the column has more stages than needed for the desired separation.

Today's control scheme gives a varying loss of the heavy component, reactant  $R_2$ , and more severely, there is no direct control of the reactor temperature,  $T_B$ . This leads to a varying quality of the product  $P$  between batches.

To illustrate how the process behaviour changes during the batch we linearise the equation system (eq. 11 and 12) at different operating points; that is at different reactor conditions or times during a batch. These models will only be valid in the close vicinity of the operating points. However, their initial responses to changes in the process will be the same as for the non-linear models.

In this work, these linear models are found

by first running a non-linear simulation of the process with given control loops implemented. The simulations are then stopped at the specified time, the controller loops are opened and the model is linearised numerically. The resulting model will thus be an open loop description of the process at the given time and conditions.

We will consider the following operating procedures

- I Today's operating practice,  $T_T = 103^\circ\text{C}$  (one point top,  $V$  constant)
- II  $T_B = 200^\circ\text{C}$  (one-point bottom,  $V$  constant)
- III  $T_B = 228^\circ\text{C}$  (one-point bottom,  $V$  constant)

An *operating point* is then specified as *procedure-time*, eg. I-8 is the conditions after 8 hr reaction time with operating procedure I.

#### 4.1 Differences between reactor conditions

To illustrate how the process behaviour changes with conditions in the reactor we consider step changes in the linearised open loop models midway through the batch.

The effect on the distillate composition  $y_D$  by a step in the reflux flow  $L$  for two different reactor conditions is given in Figure 4. Operating point I-8 is the conditions after 8 h by today's operating practice; that is when the top temperature  $T_T$  is  $103^\circ\text{C}$  ( $T_B \approx 227^\circ\text{C}$ ). Operating point II-8 is the conditions at the same time but now the reactor temperature  $T_B$  is  $200^\circ\text{C}$ .

From Fig. 4 we see that the response is similar for the two operating points but that they differ in magnitude. This is because in operating point II-8, where we have a low reactor temperature, we have a very pure distillate product. The increase in reflux will only increase the purity marginally. Whereas in operating point I-8, we have a distillate which is less pure so the increase here will be larger.

If we look at the effect on reactor temperature  $T_B$  by the same step, Fig. 5, we now

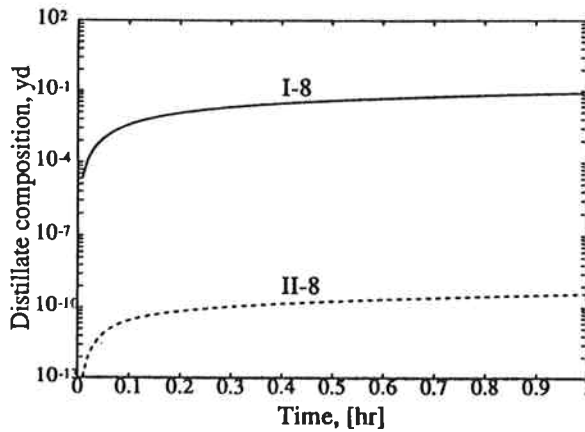


Figure 4: Different reactor conditions. Step in  $L$  : effect on  $y_D$ .

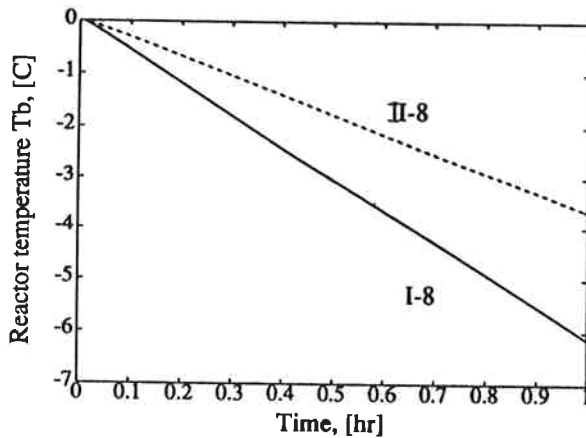


Figure 5: Different reactor conditions. Step in  $L$  : effect on  $T_B$ .

see that the effect is somewhat larger when we have a low reactor temperature. This is because the light component  $W$  is pushed downwards in the column to keep the low reactor temperature. An increase in the down coming liquid flow will push the light component front further down and thereby decreasing the reactor temperature. The more light component there is in the lower part of the column, the larger the decrease in the temperature will be.

#### 4.2 Differences in the same batch

What is perhaps more interesting to look at is the differences between various operating points at different times during the same batch, III-2 to III-15. Fig. 6 shows the effect on the

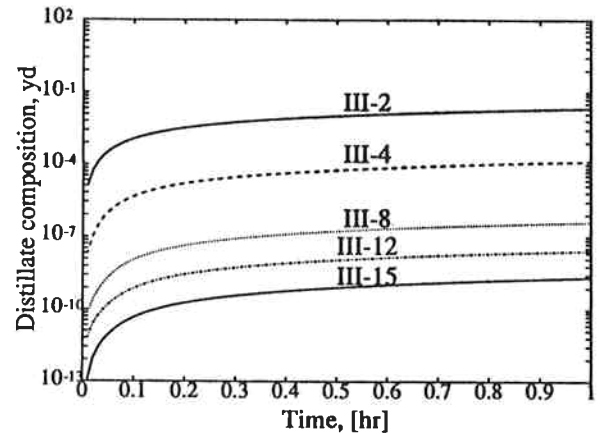


Figure 6: Different times during batch. Step in  $L$  : effect on  $y_D$ .

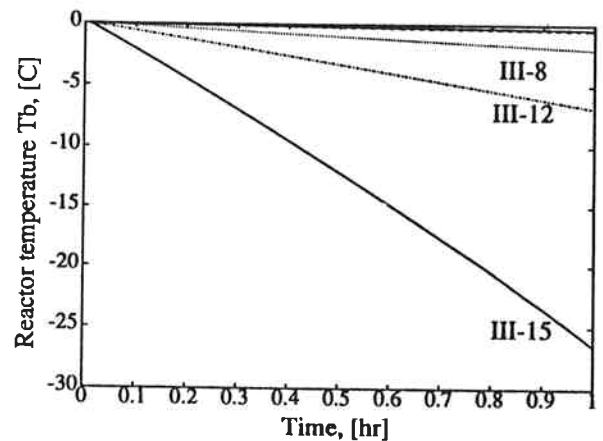


Figure 7: Different times during batch. Step in  $L$  : effect on  $T_B$ .

distillate composition  $y_D$  of a step in the reflux flow  $L$  for five different times during the same batch when the reactor temperature is  $228^\circ\text{C}$ . With such a high temperature at the bottom of the column, the light component front will be pushed out of the column initially; there will be a substantial loss of reactant  $R_2$ . The front will however, decrease during the batch as the reaction proceeds. From Fig. 6 we see that the effect by the increase in the liquid flow differs considerably during the batch. At the beginning the effect is large, but decreases as time goes by.

If we look at the effect on the reactor temperature  $T_B$  during the batch, Fig. 7, we see that it increases during the batch as the light

component front is moving downwards in the column.

### 4.3 Reducing non-linearity

Another interesting feature in Fig. 4 and 6 is that the responses have the same shape on a log-scale. By using a log transformation on the distillate composition  $y_D$  the inherent non-linearities in this variable can therefore be reduced. From Fig. 5 and 7 there is no obvious transformation that can be suggested to deal with the non-linear effect for the reactor temperature.

### 4.4 Control configuration for two-point control

After looking at responses of the process at different conditions, we now want to focus on how to control the process using a decentralised PI-controller; two control loops with a PI-controller in each ("two-point control"). We use the linearised model for operating point I-8 since operating procedure I is how the process is run today. The results will only differ in magnitude for the different operating points; the conclusions will be the same.

We consider two control configurations for the column; *LV*-configuration and *DV*-configuration. In the first we control the accumulator level using the distillate flow  $D$ . This leaves the reflux flow  $L$  and the vapour flow  $V$  to control the distillate composition  $\ln(y_D)$  and the reactor temperature  $T_B$ . In the latter we use the reflux flow  $L$  to control the accumulator level, and leaving  $D$  and  $V$  to control  $\ln(y_D)$  and  $T_B$ .

The Relative Gain Array (RGA) is a measure of the interactions in the process. Systems with no interactions will have an RGA-value of 1 and a phase of 0. The larger the deviation from 1, the larger the interaction. RGA-values less than 0 should be avoided. RGA(1,1) for both the *LV*- and *DV*-configuration are given in Fig. 8. From the figure we see that for the *LV*-configuration the RGA is high and over 1000 at low frequencies; when the system is going

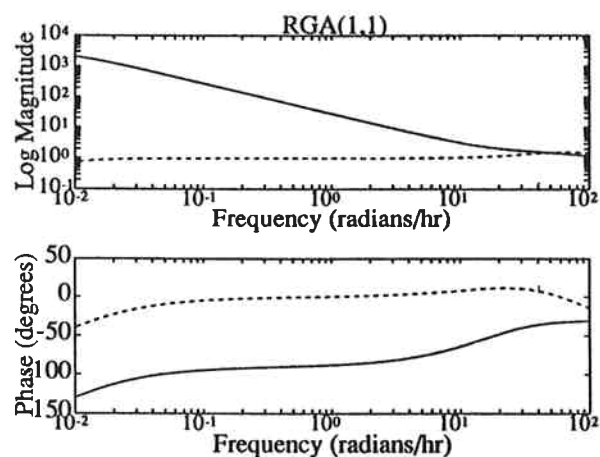


Figure 8: RGA for *LV*- and *DV*-configuration. Solid line: *LV*, Dotted line: *DV*.

to a steady state. RGA for *DV* is low at all frequencies. This difference is the same as for a continuous distillation column.

However, the control characteristics for the *LV*-configuration are not as bad as it seems since the systems bandwidth, the time for response to changes, is in the frequency range around 10 *rad/h* and RGA is closer to 1 here.

From the figure we also see that the loop pairing always should be to use the vapour flow  $V$  to control the reactor temperature  $T_B$  and either the reflux flow  $L$  or the distillate flow  $D$  to control the distillate composition or the loss of reactant  $R_2$ ,  $\ln(y_D)$ .

The decentralised controller is implemented in the linear model of the process. Fig. 9 shows the effect on  $y_D$  and  $T_B$  from set point changes. Both the configurations follow the set point changes, but *LV* is slower than *DV*. There are large interactions using the *LV*-configuration since a step change in  $T_B$  gives a large change in distillate composition  $y_D$ . Again these are the same effect as one would find in a conventional column.

### 4.5 Comparison with conventional distillation columns

The difference between the investigated distillation column and a conventional one is that the feed is now replaced by a reaction and there

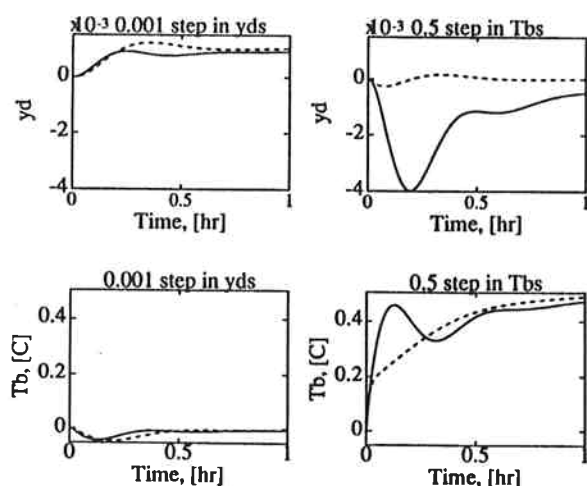


Figure 9: Set point changes for LV- and DV-configuration. Solid line: LV, Dotted line: DV.

is no stripping section. By comparing this column with a conventional we find that most conclusions from conventional columns carry over. The only difference we have found is that  $RGA \approx 1$  for the DV-configuration also for pure top product columns which means that the vapour flow should be used to control the reactor temperature always. For a conventional column the pairing is opposite for a high purity column (see Shinsky [18]).

## 5 Discussion

In this paper we have developed a dynamic model of a combined batch reactor/distillation process. Based on a linearised version of this model we have analyzed the controllability of the process depending on different reactor conditions and different times during a batch. We have found that the controllability of our industrial example changes considerably with operating point. Today's operating practice, controlling the temperature at the top of the column using the reflux flow, is poor and leads to a varying loss of reactant  $R_2$  and a varying product quality.

Controlling the reactor temperature directly using "one-point bottom control", will give a more stable product quality. However, since the controllability changes with time a non-

linear controller is needed.

"Two-point control" allows also the distillate composition to be controlled using reflux or distillate flow. This is desired if e.g. the distillate is to be used in another process or if it is to be dumped as waste. By using "two-point control" energy will be saved compared with "one-point control" as the vapour flow can be reduced. A major problem however, are the strong interactions in the system which makes feedback control difficult.

Implementation of non-linear controllers in the non-linear model of the process is in progress.

## NOTATION

$A$	system matrix
$B$	system matrix
$C$	system matrix
$D$	distillate flow, $kmol/h$
$G(s)$	transfer function
$L$	reflux flow, $kmol/h$
$L_i$	internal liquid flow, $kmol/h$
$L_{oi}$	initial liquid flow, $kmol/hr$
$M_i$	liquid holdup, $kmol$
$M_B$	liquid holdup in reactor, $kmol$
$M_D$	liquid holdup in accumulator, $kmol$
$M_{oi}$	initial liquid holdup, $kmol$
$P_j^{sat}$	saturated vapour pressure, $atm$
$r$	reaction rate, $kmol/h$
$T_i$	temperature, $K$
$T_B$	reactor temperature, $K$
$T_T$	temperature at top of column, $K$
$u$	control vector
$V$	vapour flow, $kmol/h$
$x$	state vector
$x_i$	mole fraction of light component in liquid
$y_D$	mole fraction of light component in distillate
$y$	measurement vector
$y_i$	mole fraction of light component in vapour

### Greek letters

$\alpha_i$	relative volatility
$\Delta$	deviation from operating point
$\tau$	hydraulic time constant, $h^{-1}$
$\xi_j$	stoichiometric coefficient

### subscript

$i$	tray number
$j$	component number



## References

- [1] Huckaba, C.E. and Danly, D.E. (1960). "Calculation Procedures for Binary Batch Rectification", *AIChE J.*, **5**(2), 335-342.
- [2] Meadows, E.L. (1963). "Multicomponent Batch-Distillation Calculations on a Digital Computer", *Chem. Engng. Progr. Symp. Ser.*, **59**(46), 48-55.
- [3] Distefano, G.P. (1968). "Mathematical Modeling and Numerical Integration of Multicomponent Batch Distillation Equations", *AIChE J.*, **14**(1), 190-199.
- [4] Domenech, S. and Enjalbert, M. (1974). "Modèle mathématique d'une Colonne de Rectification Discontinue - I. Établissement du Modèle", *Chem. Engng. Sci.*, **29**, 1519-1528.
- [5] Domenech, S., Ginglion, C. and Enjalbert, M. (1974). "Modèle mathématique d'une Colonne Rectification Discontinue - II. Exploitation Numérique", *Chem. Engng. Sci.*, **29**, 1529-1535.
- [6] Gallun, S.E. and Holland, C.D. (1982). "Gear's Procedure for the Simultaneous Solution of Differential and Algebraic Equations with Application to Unsteady State Distillation Systems", *Comp. Chem. Engng.*, **6**(3), 231-244.
- [7] Sadotomo, H. and Miyahara, K. (1983). "Calculation Procedure for Multicomponent Batch Distillation", *Int. Chem. Engng.*, **23**(1), 56-64 (first published in 1981, Japan).
- [8] Mujtaba, I.M. and Macchietto, S. (1988). "Optimal Control of Batch Distillation", *12th IMACS World Conf., Paris, France, 1988*, **2**, CERFIDN, Villeneuve d'Osc, 635-638.
- [9] Egly, H., Ruby, V. and Seid, B. (1979). "Optimum design and operation of batch rectification accompanied by chemical reaction", *Comp. Chem. Engng.*, **3**, 169-174.
- [10] Egly, H., Ruby, V. and Seid, B. (1982). "Optimale Betriebsweisen diskontinuierlicher Rectificationen mit überlagerten Reaktionen und deren Regelung", *Chem. Ing. Tech.*, **54**(9), 842-843.
- [11] Cuille, P.E. and Reklaitis, G.V. (1986). "Dynamic Simulation of Multicomponent Batch Rectification with Chemical Reactions", *Comp. Chem. Engng.*, **10**(4), 389-398.
- [12] Reuter, E., Wozny, G. and Jeromin, L. (1988). "Modeling of Multicomponent Batch Distillation Processes with Chemical Reaction and their Control Systems", *Proc. CHEM-DATA '88, Gothenburg*, 322-329.
- [13] Albet, J., Le Lann, J.M., Joulia, X. and Koehret, B. (1991). "Rigorous Simulation of Multicomponent Multisequence Batch Reactive Distillation", *Proc. COPE'91, Barcelona, Spain*, 75-80.
- [14] Bristol, E.H. (1966). "On a New Measure of Interactions for Multivariable Process Control", *IEEE Trans. Automat. Control*, **AC-11**, 133-134.
- [15] Bristol, E.H. (1978). "Recent Results on Interactions for Multivariable Process Control", Paper at *AIChE Annual Meeting, Chicago, IL*.
- [16] Grosdidier, P., Morari, M. and Holt, B.R. (1985). "Closed-Loop Properties from Steady-State Gain Information", *Ind. Eng. Chem. Fundam.*, **24**, 221-235.
- [17] Skogestad, S. and Hovd, M. (1990). "Use of Frequency-Dependent RGA for Control Structure Selection", *Proc. ACC'90, San Diego*, 2133-2139.
- [18] Shinsky, F.G. (1984). "Distillation Control", 2. ed., *McGraw-Hill Inc.*