Decoupling PID Control of a Reactive Packed Distillation Column

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Abstract

This work has been carried out to demonstrate the application of decouplers in the temperature control of a reactive packed distillation column using PID controllers and the production of ethyl acetate as the case study. Ziegler-Nichols and Cohen-Coon techniques were used to calculate the tuning parameters of the controllers for simulation with the aid of MATLAB/Simulink. In the set-point tracking study, top segment, reaction segment and bottom segment temperatures were the controlled variables while reflux ratio, feed ratio and reboiler duty were chosen as the manipulated variables. For the disturbance rejection study, the reboiler duty was chosen as the disturbance variable. After the simulation, the results obtained from the simulations showed that decoupling PID control has been successfully applied to the reactive packed distillation column. In addition, the lower values of IAEs and ISEs of Cohen-Coon tuning technique revealed that it was better than Ziegler-Nichols tuning technique for this process.

Key words: Reactive distillation, System Identification, MATLAB/Simulink, Decoupling PID, Ethyl acetate

1. Introduction

Reactive distillation combines both chemical reaction and multicomponent separation into a single unit [5]. It is sometimes an excellent alternative to conventional flowsheets with separate reaction and separation sections [3]. It has been used in a small number of industrial applications for many years, but the last decade has shown an increase in both its research and applications [2]. In applying reactive distillation, the reactant and product volatilities must be such that products can be removed and reactants retained inside the column. The temperature levels for both reaction and vapour–liquid equilibrium must overlap [3]. By carrying out the chemical reaction and the separation in one process step, the operating and investment costs can be minimized. Some additional benefits offered by reactive distillation technology include [12]: (i) increased yield, because of overcoming chemical and thermodynamic equilibrium limitations, (ii) improved selectivity via suppression of side reactions, (iii) reduced energy consumption, due to the effective utilization of reaction heat, in the case of exothermic reactions, (iv) avoidance of hot spots by simultaneous liquid evaporation, and (v) ability to separate close boiling components. Due to these advantages and with growing process understanding, the chemical process industry has developed an increasing number of processes based on reactive distillation [4]. However, reactive distillation is not extensively used in industry since it is perceived that its operation will always be more difficult and will pose higher requirements on the quality of the design and control system than the conventional flowsheet in which a reactor is typically followed by a train of distillation columns. This behaviour can be mainly attributed to the complex interactions between the underlying physical phenomena taking place in reactive columns, having a significant influence on the robust operation under variations. The control of reactive distillation has received some attention only recently. Sneesby et al. (1997) worked on the dynamic simulation and control aspects of reactive distillation for the synthesis of ethyl tert-butyl ether and presented general recommendations for the control of the reactive column of that type including the need for addressing the control issues early in the design process. Bock et al. (1997) developed a control structure for a reactive column with a recovery column by analysing the reaction column's steady state and dynamic sensitivity of possible disturbances and manipulated variables. Sneesby et al. (1999) used an ethyl tert-butyl ether reactive distillation column as a case study to show how a two-point control configuration, which recognized the importance of both composition and conversion, can be developed and implemented for a reactive distillation process. Kumar and Daoutidis (1999) studied the dynamic behaviour and control of an ethylene glycol reactive distillation column by deriving a detailed tray-by-tray model that

explicitly included the vapor-phase balances. They developed a nonlinear controller that yielded good performance with stability in the high-purity region with the aid of a physical insight into the nonminimum phase behaviour, and the superior performance of the developed controller over linear PI controllers was demonstrated through simulations. Monroy-Loperena et al. (2000) also studied the control problem of an ethylene glycol reactive distillation column with the control objective of regulating the ethylene glycol composition in the product by manipulating the reboiler boil-up ratio. They proposed a new idea for robust stabilization based on the analysis of the underlying input/output bifurcation diagram and on modelling error compensation techniques. Al-Arfaj and Luyben (2000) explored the closed-loop control of a reactive distillation column with two products and discovered that single-end temperature control could keep both products at or above the specified purity values, even for large disturbances, if reactive-zone holdup was sufficiently large. Vora and Daoutidis (2001) studied the dynamics and control of an ethyl acetate reactive distillation system and designed model-based linear and nonlinear state feedback controllers, along with conventional SISO PI controllers. They demonstrated the superior performance of the nonlinear controller over both the linear controller and the conventional PI controller. Khaledi and Young (2005) investigated the nonlinearity of an ethyl tert-butyl ether reactive distillation column and developed a 2 x 2 unconstrained model predictive control scheme for product purity and reactant conversion control by using the process dynamics approximated by a first-order plus dead time model to estimate the process model for the model predictive controller. They found that the controller was very efficient for disturbance rejection and set-point tracking. Völker et al. (2007) designed a multivariable controller for a medium-scale semi-batch reactive distillation column and showed experimentally that the controller performed well for large set-point changes and in the face of process disturbances. Grüner et al. (2003) applied asymptotically exact input/output-linearization to an industrial reactive distillation column and found through simulation studies that, in comparison with a well-tuned linear controller, the controller showed a superior performance with respect to set-point changes and disturbances, even in the presence of unknown input delays.

Reactive distillation is a multi-input multi-output (MIMO) system and, based on that, the system is expected to be controlled as a MIMO type or using one of the early approaches to multivariable control which is “loop decoupling”. Loop decoupling can be realized by adding additional controllers called decouplers to a conventional multi-loop configuration. In principle, decoupling control schemes can provide two important benefits [13]:

(i) Elimination of control loop interactions which result in the stability of the closed-loop system being determined solely by the stability characteristics of the individual feedback control loops.
(ii) A set-point change for one controlled variable having no effect on the other controlled variables.

Considering the two points highlighted above, it is expected that decoupling control should provide good performance in the control of chemical systems especially in the control of MIMO systems like the reactive packed distillation process being considered in this work. Therefore, the aim of this work is to study the application of decoupling PID controllers for the control of some segment temperatures of reactive packed distillation column using the production of ethyl acetate and water (by-product) from the esterification reaction between acetic acid and ethanol as the case study.

2. Procedures

2.1. Experimental Procedure

The process involved in this work was an esterification reaction occurring simultaneously with distillation operation that were carried out in the experimental pilot plant of the reactive packed distillation column (RPDC) set up as in Figure 1a (pictorially) and Figure 1b (schematically) and as also shown in the work of Giwa and Karacan (2012). The column, excluding the condenser and the reboiler, had a height of 1.5 m and a diameter of 0.05 m. It consisted of a cylindrical condenser of a diameter and a height of 5 and 22.5 cm respectively. The main column section of the plant was divided into three subsections of 0.5 m each. The upper, middle and lower sections were the rectifying, the reaction and the stripping sections respectively. The rectifying and the stripping sections were packed with raschig rings while the reaction section was filled with Amberlyst 15 solid catalyst that had a surface area of 5300 m^2/kg, a total pore volume of 0.4 cc/g and a density of 610 kg/m^3. The reboiler was spherical in shape with a volume of 3 Litre. The column was fed with acetic acid at the top (between the rectifying and the reaction sections) while ethanol was fed at the bottom (between the reaction and the stripping sections) with the aid of peristaltic pumps which were operated with the aid of a computer via MATLAB/Simulink program. All the signal inputs (reflux ratio (R), feed ratio (F) and reboiler duty (Q)) to the column and the measured outputs (top

segment temperature (T_{top}), reaction segment temperature (T_{rxn}) and bottom segment temperature (T_{bot})) from the column were sent and recorded respectively on-line with the aid of MATLAB/Simulink computer program and electronic input-output (I/O) modules that were connected to the equipment and the computer system. The esterification reaction occurring in the column was an equilibrium type given as:

$$CH_3COOH + C_2H_5OH \xleftrightarrow{K_{eq}} CH_3COOC_2H_5 + H_2O \quad (1)$$

The experimental reactive distillation process together with the column described above was used to generate the data used for the development of the transfer function models for the process with the aid of System Identification Toolbox contained in MATLAB [10].

Two different types of experiments were carried out to generate two different sets of data. The first one was used for the development of MIMO transfer functions which were used as the models of the process while the second one was used for the development of SISO transfer functions that were used for the calculation of the tuning parameters of the controllers. The conditions used in carrying out the experiments of this study are as shown below in Table 1 for the first experiment.

Table 1. Input values of experiment 1

S/N	Parameter	Signal	Level	
			Initial	Final
1	R	Step	3	5
2	F	PRBS	0.5	2
3	Q (kJ/s)	PRBS	0.595	0.63

Also shown in Table 2 are the values of the input variables used to accomplish the second experiment of this study.

Table 2. Input values of experiment 2

S/N	Parameter	Signal	Level	
			Initial	Final
1	R	PRBS	1	5
2	F	PRBS	1	2
3	Q (kJ/s)	PRBS	0.49	0.63

(a)

(b)

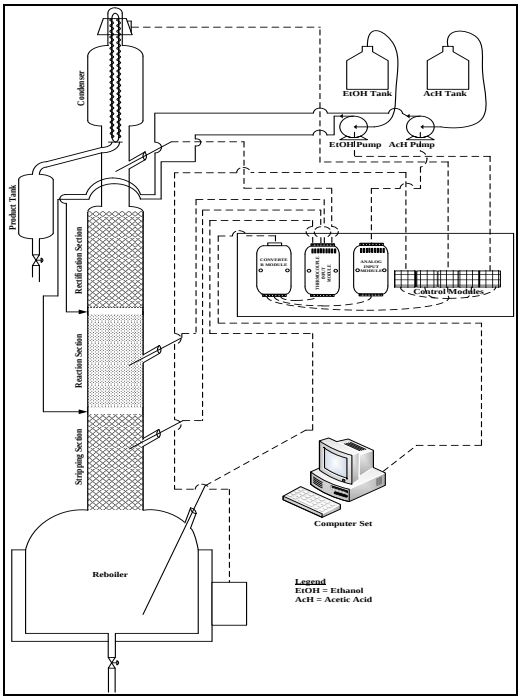

Figure 1. Reactive packed distillation column. (a) Pictorial view; (b) Sketch view

2.2. Control Configuration and Simulation

In the control aspect of this work, the temperatures of three segments of the reactive packed distillation column were considered and the process was tested for both set-point tracking and disturbance rejection. For the set-point tracking study, the system, being a MIMO type, comprised three controlled variables (top segment temperature, reaction segment temperature and

bottom segment temperature) and three manipulated variables (reflux ratio, feed ratio and reboiler duty) while for the disturbance rejection study, chosen the reboiler duty as the disturbance variable, in this case, there were two controlled variables (top segment temperature and reaction segment temperature) and two manipulated variables (reflux ratio and feed ratio). The MIMO system was decoupled and controlled like a SISO type by the application of decouplers that were estimated from the MIMO transfer function models of the system. After the decoupling, the reflux ratio, feed ratio and reboiler duty were used as the manipulated variables of top segment temperature, reaction segment temperature and bottom segment temperature respectively for the set-point tracking study. In the disturbance rejection study, the reflux ratio and feed ratio were used as manipulated variables for top segment temperature and reaction segment temperature respectively. The decoupling studies of the set-point tracking and the disturbance rejection were carried out using program coded with Script and Simulink environment of MATLAB [10].

2.3. Tuning of Controllers

The controllers designed for the reactive packed distillation column were tuned using two different techniques, namely, Ziegler-Nichols (Z-N) and Cohen-Coon (C-C) tuning methods using the SISO transfer function models developed. With the transfer function of the PID controller given as,

$$G_c(s) = K_c\left(1+\frac{1}{\tau_I s}+\tau_D s\right) \qquad (2)$$

the relationships used for the calculation of the tuning parameters of the two techniques are as given in Table 3 below.

Table 3. Tuning parameters equations [16]

Parameter	Z-N	C-C
K_c	$\frac{K_c}{1.7}$	$\frac{1}{K_p}\frac{\tau}{t_d}\left(\frac{4}{3}+\frac{t_d}{4\tau}\right)$
τ_I	$\frac{P_u}{2}$	$t_d\frac{32+6t_d/\tau}{13+8t_d/\tau}$
τ_D	$\frac{P_u}{8}$	$t_d\frac{4}{11+2t_d/\tau}$

3. Results and Discussions

3.1. Experimental Studies

In the experimental studies, after applying the inputs shown in Table 1, the response obtained from experiment 1 are as shown in Figures 2, 3 and 4 below for the top segment temperature, the reaction segment temperature and the bottom segment temperature respectively. As can be observed from the results, the application of the inputs resulted in changes in the dynamic responses of the segment temperatures. This is an indication that the segment temperatures were functions of the inputs. This is, of course, the reason for choosing the inputs as the manipulated variables of the control of this process.

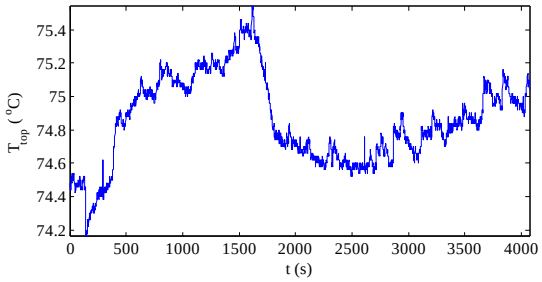

Figure 2. Experimental dynamic response of top segment temperature to the inputs of experiment 1

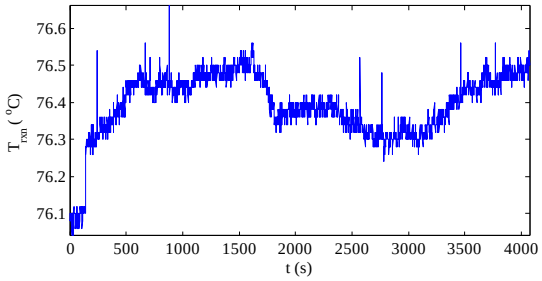

Figure 3. Experimental dynamic response of reaction segment temperature to the inputs of experiment 1

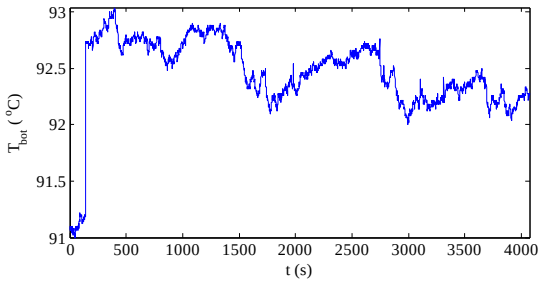

Figure 4. Experimental dynamic response of bottom segment temperature to the inputs of experiment 1

Furthermore, the application of the inputs of experiment 2 given in Table 2 resulted in the responses given in Figures 5, 6 and 7 respectively for the top segment temperature, the reaction segment temperature and the bottom segment temperature. As was observed in the case of experiment 1, there were significant dynamic responses towards the individual inputs for each of the segment temperatures.

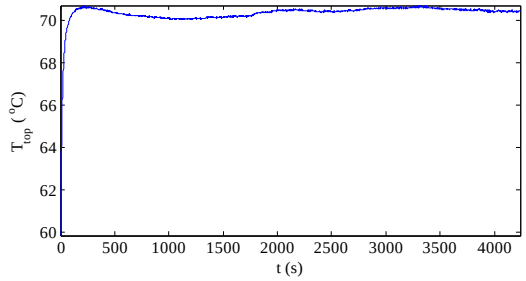

Figure 5. Experimental dynamic response of top segment temperature to the inputs of experiment 2

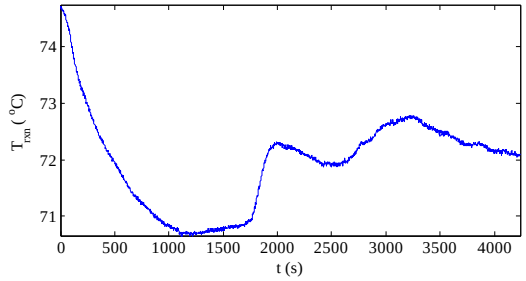

Figure 6: Experimental dynamic response of reaction segment temperature to the inputs of experiment 2

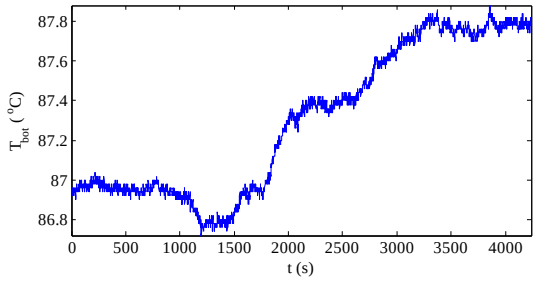

Figure 7: Experimental dynamic response of bottom segment temperature to the inputs of experiment 2

3.2. System Identification Studies

With the three (3) inputs (reflux ratio, feed ratio and reboiler duty) and three (3) outputs (top segment temperature, reaction segment temperature and bottom segment temperature) chosen as the variables of this process, the results (Figures 2 - 4) obtained from experiment 1 were used to develop the MIMO transfer function models of the process with the aid of System Identification Toolbox of MATLAB. The forms of the process models developed are as given in Equations (3 - 5) below.

$$T_{top}(s) = \frac{k_{p_{1,1}} e^{(\tau_{d_{1,1}} s)}}{\tau_{1,1} s + 1} R(s) + \frac{k_{p_{1,2}} e^{(\tau_{d_{1,2}} s)}}{\tau_{1,2} s + 1} F(s) + \frac{k_{p_{1,3}} e^{(\tau_{d_{1,3}} s)}}{\tau_{1,3} s + 1} Q(s) \quad (3)$$

$$T_{rxn}(s) = \frac{k_{p_{2,1}} e^{(\tau_{d_{2,1}} s)}}{\tau_{2,1} s + 1} R(s) + \frac{k_{p_{2,2}} e^{(\tau_{d_{2,2}} s)}}{\tau_{2,2} s + 1} F(s) + \frac{k_{p_{2,3}} e^{(\tau_{d_{2,3}} s)}}{\tau_{2,3} s + 1} Q(s) \quad (4)$$

$$T_{bot}(s) = \frac{k_{p_{3,1}} e^{(\tau_{d_{3,1}} s)}}{\tau_{3,1} s + 1} R(s) + \frac{k_{p_{3,2}} e^{(\tau_{d_{3,2}} s)}}{\tau_{3,2} s + 1} F(s) + \frac{k_{p_{3,3}} e^{(\tau_{d_{3,3}} s)}}{\tau_{3,3} s + 1} Q(s) \quad (5)$$

The values of the model parameters shown in Equations (3 - 5) above are as given in Table 4. It can be seen from the model parameters that while some static gains (K_p) were positive, others were negative. The static-gain sign change is one of the phenomena occurring as a result of the complex nature of the reactive packed distillation process. Also, it was observed that the maximum static gain occurred between input 2 and output 2 (that is, between the feed ratio and the reaction segment temperature).

Table 4. Process model parameters

G(s)	K_p	τ (min)	T_d (min)
$g_{11}(s)$	-79.321	5.503516	0.00092
$g_{12}(s)$	-1.3652	5.184786	0.495887
$g_{13}(s)$	-89.1061	5.169331	0.001784
$g_{21}(s)$	31.94718	8.465251	0.489046
$g_{22}(s)$	427.8996	5.266308	0.009036
$g_{23}(s)$	-18434.9	5.290776	0.009019
$g_{31}(s)$	-739.469	35.64333	8.59E-06
$g_{32}(s)$	6.791428	1.005618	0.499407
$g_{33}(s)$	-231.189	0.790481	0.5

Considering the time constant (τ) of the process, the transfer function of the relationship between the reflux ratio and the bottom segment temperature was found to possess the highest value. This is an indication of the fact that when the same input unit is applied to the process, this part of the process is likely to have the highest effect on the time required for the process to get to the steady state.

As can be seen from the values of the delay time (T_d) also shown in Table 4, the maximum delay time possessed by the system was found to be 0.5 min. This means that the output variables of the reactive packed distillation studied in this work were responding quickly to the changes in the input variables.

The results of experiment 2 were similarly used to develop the SISO transfer function models that were used to calculate the tuning parameters of the controllers. Also, the three SISO transfer function models between the individual input and the corresponding output are as outlined in Equations 6, 7 and 8 below respectively for the top segment temperature, the reaction segment temperature and the bottom segment temperature.

$$T_{top}(s) = \frac{k_{p_1} e^{(T_{d_1} s)}}{\tau_1 s + 1} R(s) \quad (6)$$

$$T_{rxn}(s) = \frac{k_{p_2} e^{(T_{d_2} s)}}{\tau_2 s + 1} F(s) \quad (7)$$

$$T_{bot}(s) = \frac{k_{p_3} e^{(T_{d_3} s)}}{\tau_3 s + 1} Q(s) \quad (8)$$

The parameters of the SISO transfer functions, given in Equations 6, 7 and 8, calculated with the aid of MATLAB, are as shown in Table 5.

Table 5. Control model parameters

G(s)	K_p	τ (min)	T_d (min)
$g_1(s)$	23.64362	165.1444	0.33942
$g_2(s)$	47.79774	18.93721	0.127159
$g_3(s)$	173.0116	579.5258	0.211721

According to the results shown in Table 5, the model with the highest static gain and time constant was discovered to be that of the bottom segment temperature. This is revealing that, especially when this system is simulated as a SISO type, the reboiler must be heated for a long time before the bottom segment temperature can response. That is, the reboiler heat must be applied to the system for a long time before the liquid mixture will be boiled and thereby evaporated to the top segment of the column via the reaction segment. This phenomenon was actually found to be so while carrying out the experiments of this study.

3.3. Tuning Parameters

With reference to Equation (2), and using the transfer function model equations developed for the controllers, the values obtained from the calculation of the tuning parameters of the controllers are as shown in Tables 6 and 7. Table 6 shows the tuning parameters of the controllers using Ziegler-Nichols PID control tuning technique while shown in Table 7 are the tuning parameters calculated with Cohen-Coon PID control tuning technique for the three controllers involved in the control study of this work.

Table 6. Ziegler-Nichols control tuning parameters

Controller	K_c	τ_I (min)	τ_D (min)
PIDC1	19.0144	0.6788	0.1697
PIDC2	2.8790	0.2543	0.0636
PIDC3	14.6186	0.4234	0.1059

Table 7. Cohen-Coon control tuning parameters

Controller	K_c	τ_I (min)	τ_D (min)
PIDC1	19.0144	0.8348	0.1234
PIDC2	2.8790	0.3121	0.0462
PIDC3	14.6186	0.5211	0.0770

From the tables, the proportional gain of the controllers using the two tuning techniques (Ziegler-Nichols and Cohen-Coon) were observed to be the same but the integral time and the derivative time were found to be different.

3.4. Decouplers

The decouplers, calculated in form of a decoupling matrix, used in this work for the control of reactive packed distillation column were estimated with reference to the model equations given in Equations 3, 4 and 5 using the relationship shown in Equation (9)

$$\boldsymbol{K}_I = \begin{bmatrix} k_{p_{11}} & k_{p_{12}} & k_{p_{13}} \\ k_{p_{21}} & k_{p_{22}} & k_{p_{23}} \\ k_{p_{31}} & k_{p_{32}} & k_{p_{33}} \end{bmatrix}^{-1} \quad (9)$$

to be approximately

$$\boldsymbol{K}_I = \begin{bmatrix} -0.0005 & 0.0000 & -0.0013 \\ -0.2788 & 0.0010 & 0.0300 \\ -0.0065 & -0.0000 & 0.0007 \end{bmatrix} \quad (10)$$

The applications of decouplers to the system resulted in the elimination of the effects of interactions among the variables involved in the control of the MIMO reactive packed distillation column. Thus, the control of the MIMO system was accomplished like that of SISO systems. This made the control of the column easier and more efficient, as can be seen from the results of the control studies outlined and discussed below.

3.5. Control Studies

3.5.1. Set-Point Tracking

The results obtained from the set-point tracking simulation of control studies of this work are as shown in Figures 8 – 10. It should be noted from

the results that the values of the segment temperature are given in form of deviation variables. As can be observed from Figure 8, the responses of the top segment temperatures obtained using the two tuning techniques were able to reach the set point at almost the same time (that is, their response or settling times were almost the same) and with very close rise time. However, the overshoot observed from Ziegler-Nichols tuning technique was found to be higher than that of Cohen-Coon tuning technique.

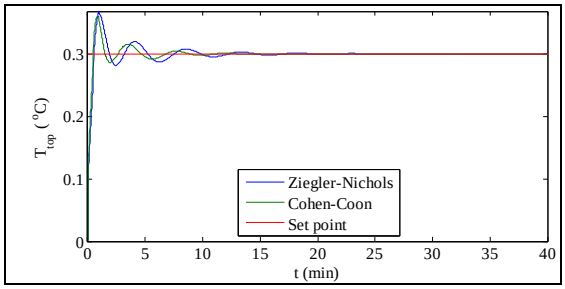

Figure 8. Dynamic responses of top segment temperature to a 0.3 unit set-point change

Also observed from the responses of the top segment temperature shown in Figure 8 was that they were able to get to the set point at approximately 25 min of simulation but the simulation was run up to 40 min because of the responses of the other two segments (reaction segment temperature and bottom segment temperature) that could not actually settle at that time, and since the system was really a MIMO type, the simulation was carried out for all of the segment temperatures with the same simulation module of Simulink. In other words, the simulation time was determined by the slowest-settling segment temperature of the process.

Similar observations obtained from the responses of the top segment temperatures for the two tuning techniques investigated in this work were also observed from the responses of the reaction segment temperature as can be seen in Figure 9. The responses of Ziegler-Nichols tuning technique had higher overshoot than that of the Cohen-Coon tuning technique but the responses of the two techniques were able to get to the steady-state at almost the same time and with approximately the same rise time.

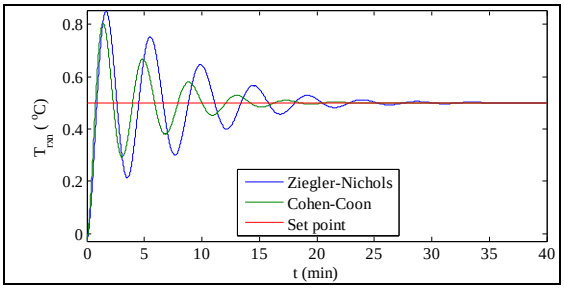

Figure 9. Dynamic responses of reaction segment temperature to a 0.5 unit set-point change

As shown in Figure 10, the nature of the results obtained from the responses of the bottom segment temperatures were not very different from those of the other two (top segment and reaction segment temperatures) discussed before because, for this case too, the overshoot obtained when Ziegler-Nichols was used as the tuning technique was also higher than of the Cohen-Coon tuning technique.

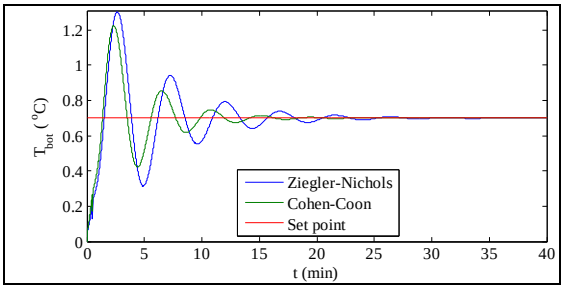

Figure 10. Dynamic responses of bottom segment temperature to a 0.7 unit set-point change

In order to determine the better one among the two tuning techniques applied for the decoupling PID control of the reactive packed distillation column for the set-point tracking case, the performance criteria, Integral Absolute Error (IAE) and Integral Squared Error (ISE) of the controllers were calculated with the aid of Simulink and the results obtained from the calculations are as shown in Table 8 below.

Table 8. Performance criteria of set-point tracking control system

Method	**IAE**			**ISE**		
	$\mathbf{T_{top}}$	$\mathbf{T_{rxn}}$	$\mathbf{T_{bot}}$	$\mathbf{T_{top}}$	$\mathbf{T_{rxn}}$	$\mathbf{T_{bot}}$
Z-N	0.20	2.27	3.02	0.01	0.45	0.98
C-C	0.14	1.36	2.02	0.01	0.25	0.62

As can be seen from Table 8 above, the IAE values of Cohen-Coon tuning technique were found to be lower than those of the Ziegler-Nichols tuning technique for the top segment, the reaction segment

and the bottom segment temperatures. Similarly, the ISE values of Cohen-Coon tuning technique were discovered to be lower than those of the Ziegler-Nichols tuning technique except in the case of the top segment temperature where they (the two tuning techniques) had the same ISE values, as seen in Table 8. These results of the IAE and ISE values were found to be in support of the observations obtained from the graphical responses of the temperatures.

3.5.2. Disturbance Rejection

As said earlier, the temperature control of this process was tested not only for set-point tracking but also for disturbance rejection. As such, the control simulation of this study was also carried out for disturbance rejection (the disturbance variable was chosen to be the reboiler duty) and the results of the simulations are as shown in Figures 11 and 12.

Figure 11 shows the responses of the top segment temperatures for a unit step change in the reboiler duty for the two tuning techniques studied in this work. As can be seen from the figure, the response of the Cohen-Coon tuning technique was observed to be a little bit, though not much, faster in reaching the set-point of the process than that of the Ziegler-Nichols technique.

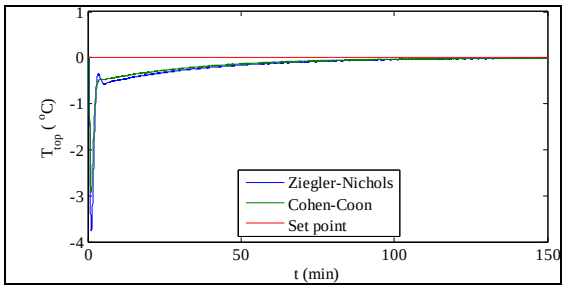

Figure 11. Dynamic responses of top segment temperature to a unit step change in disturbance variable

Shown in Figure 12 are the dynamic responses of the reaction segment temperature to the disturbance variable. Just as it was observed in the case of the set-point tracking, the overshoot of the Ziegler-Nichols tuning technique was found to be higher than that of the Cohen-Coon tuning technique but the responses of the two PID control tuning techniques were discovered to settle almost at the same time.

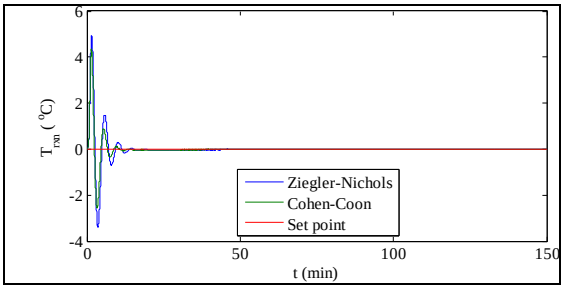

Figure 12. Dynamic responses of reaction segment temperature to a unit step change in disturbance variable

The differences between the two control tuning techniques were also shown clearly and numerically by calculating the IAE and ISE values and the results of the calculations are as tabulated below (Table 8). From the table, it was observed that the IAE and ISE values of Cohen-Coon tuning technique were lower than those of the Ziegler-Nichols for both the top segment temperature and the reaction segment temperature.

Table 9. Performance criteria of disturbance rejection control system

Method	IAE		ISE	
	T_{top}	T_{rxn}	T_{top}	T_{rxn}
Z-N	26.06	16.14	21.05	37.86
C-C	22.19	11.65	13.76	23.73

This smaller natures of the IAE and ISE values for the responses of the segment temperatures of the Cohen-Coon tuning technique than those of the Ziegler-Nichols tuning technique were also in support of what were observed from the graphical responses of the segment temperatures.

4. Conclusions

The good results obtained from the simulations of decoupling PID set-point tracking and disturbance rejection control studies of reactive packed distillation column using Ziegler-Nichols and Cohen-Coon tuning techniques have shown that decoupling PID control can be successfully applied to the system. In addition, due to its relatively lower values of IAE and ISE, Cohen-Coon tuning technique was discovered to be better than Ziegler-Nichols tuning technique for this process.

5. Acknowledgements

Abdulwahab Giwa wishes to acknowledge the support received from the Scientific and Technological Research Council of Turkey (Türkiye Bilimsel ve Teknolojik Araştırma

Kurumu - TÜBİTAK) for his Ph.D. Programme. In addition, this research was supported by the Scientific Research Project Office of Ankara University (Ankara Üniversitesi Bilimsel Araştırma Proje Ofisi) under Project No. 09B4343007.

6. Nomenclatures

τ	Time constant of the process model (min)
τ_D	Derivative time of the controller (min)
τ_I	Integral time of the controller (min)
C-C	Cohen-Coon
F	Feed ratio (mL s^{-1} F_a / mL s^{-1} F_e)
F_a	Acetic acid feed rate (mL/s)
F_e	Ethanol feed rate (mL/s)
G(s)	Model transfer function
g(s)	Submodel transfer function
IAE	Integral Absolute Error
ISE	Integral Squared Error
K_c	Proportional gain of the controller
K_I	Decoupling matrix
K_p	Static gain of the process model
MIMO	Multi-Input Multi-Output
PID	Proportional-Integral-Derivative
PIDC	Proportional-Integral-Derivative Control(ler)
PRBS	Pseudo-Random Binary Sequence
P_u	Ultimate period (min/cycle)
Q	Reboiler duty (kJ/s)
R	Reflux ratio
RPDC	Reactive Packed Distillation Column
SISO	Single-Input Single-Output
t	Simulation time (s)
T_{bot}	Bottom segment temperature (^oC)
t_d	Delay time (min)
T_d	Delay time (min)
T_{rxn}	Reaction segment temperature (^oC)
T_{top}	Top segment temperature (^oC)
Z-N	Ziegler-Nichols

7. References

[1] Al-Arfaj, M.A. and Luyben, W.L., "*Comparison of Alternative Control Structures for an Ideal Two-Product Reactive Distillation Column"*, Industrial and Engineering Chemistry Research, 2000, 39: 3298-3307.

[2] Al-Arfaj, M.A. and Luyben, W.L., "*Comparative Control Study of Ideal and Methyl Acetate Reactive Distillation"*, Chemical Engineering Science, 2002a, 57: 5039-5050.

[3] Al-Arfaj, M.A. and Luyben, W.L., "*Design and Control of an Olefin Metathesis Reactive Distillation Column"*, Chemical Engineering Science, 2002b, 57: 715-733.

[4] Bock, H., Wozny, G. and Gutsche, B., 1997. "*Design and Control of a Reaction Distillation Column Including the Recovery System"*, Chemical Engineering and Processing, 36: 101- 09.

[5] Cheng, Y. and Yu, C., "*Effects of Feed Tray Locations to the Design of Reactive Distillation and its Implication to Control"*, Chemical Engineering Science, 2005, 60: 4661-4677.

[6] Giwa, A. and Karacan, S., "*Simulation and Optimization of Ethyl Acetate Reactive Packed Distillation Process Using Aspen Hysys"*, The Online Journal of Science and Technology, 2012, 2: 57-63.

[7] Grüner, S., Mohl, K.D., Kienle, A., Gilles, E.D. Fernholz, G. and Friedrich, M., "*Nonlinear Control of a Reactive Distillation Column"*, Control Engineering Practice, 2003, 11: 915-925.

[8] Khaledi, R. and Young, B.R., "*Modeling and Model Predictive Control of Composition and Conversion in an ETBE Reactive Distillation Column"*, Industrial and Engineering Chemistry Research, 2005, 44: 3134-3145.

[9] Kumar, A. and Daoutidis, P., "*Modeling, Analysis and Control of Ethylene Glycol Reactive Distillation Column"*, AIChE Journal, 1999, 45: 51-68.

[10] Mathworks, MATLAB, *The Language of Technical Computing*, The MathWorks, Inc., Natick, 2012.

[11] Monroy-Loperena, R., Perez-Cisneros, E. and Alvarez-Ramirez, J., "*A Robust PI Control Configuration for a High-Purity Ethylene Glycol Reactive Distillation Column"*, Chemical Engineering Science, 2000, 55: 4925-4937.

[12] Prakash, K.J.J, Patle, D.S. and Jana, A.K., "*Neuro-Estimator Based GMC Control of a Batch Reactive Distillation"*, ISA Transactions, 2011, 50: 357-363.

[13] Seborg, D.E., Edgar, T.F. and Mellichamp, D.A., *Process Dynamics and Control*, 2nd Edition, John Wiley & Sons, New Jersey, 2004.

[14] Sneesby, M.G., Tade, M.O. and Smith, T.N., "*Two-Point Control of a Reactive Distillation Column for Composition and Conversion"*, Journal of Process Control, 1999, 9: 19-31.

[15] Sneesby, M.G., Tade, M.O., Datta, R. and Smith, T. N., "*ETBE Synthesis via Reactive Distillation. 2. Dynamic Simulation and Control Aspects"*, Industrial and Engineering Chemistry Research, 1997, 36, 1870-1881.

[16] Stephanopoulos, G., *Chemical Process Control: An Introduction to Theory and Practice*, PTR Prentice Hall, New Jersey, 1984.

[17] Völker, M., Sonntag, C. and Engell, S., "*Control of Integrated Processes: A Case*

Study on Reactive Distillation in a Medium-Scale Pilot Plant", Control Engineering Practice, 2007, 15: 863-881.

[18] Vora, N. and Daoutidis, P., "*Dynamics and Control of an Ethyl Acetate Reactive Distillation Column",* Industrial and Engineering Chemistry Research, 2001, 40: 833-849.