

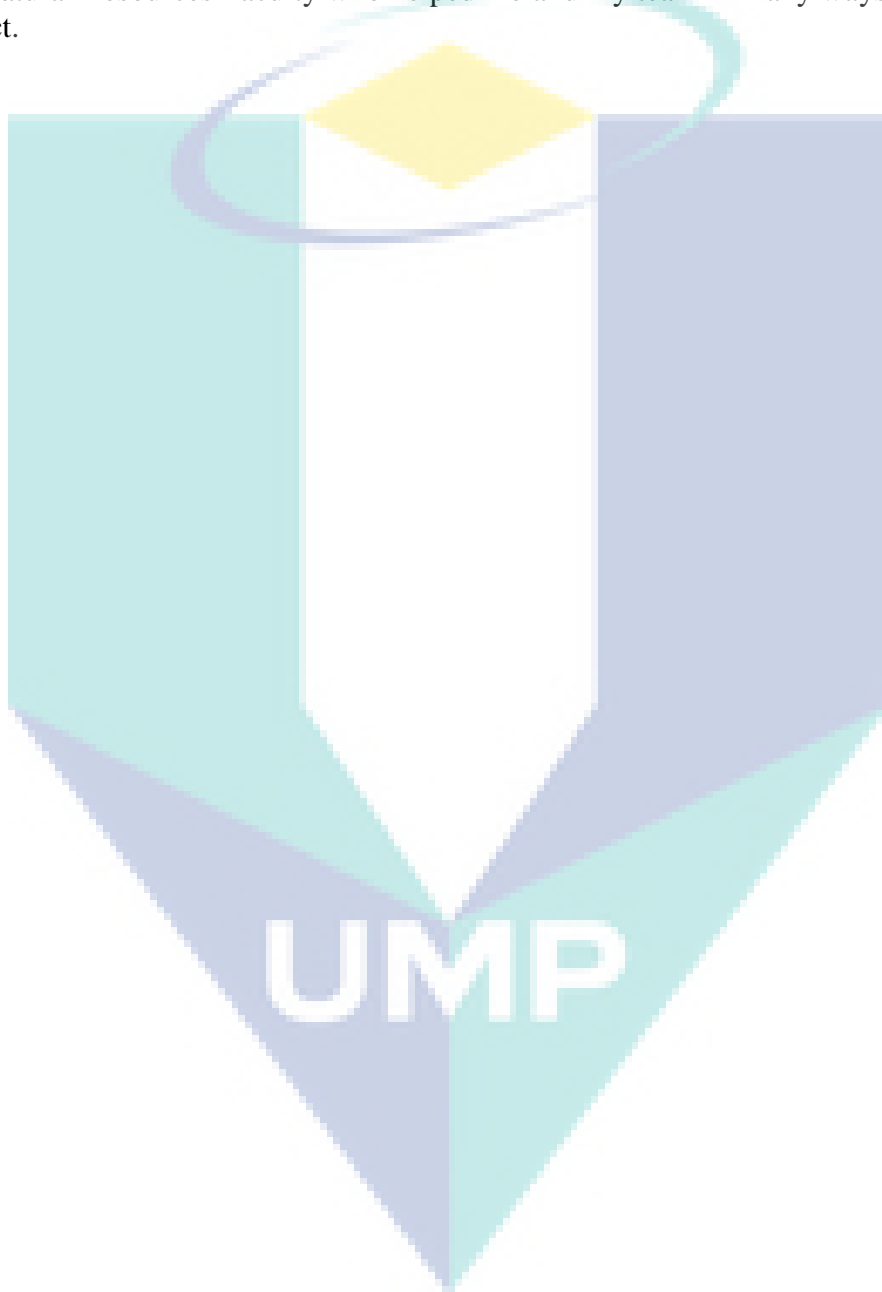
DEVELOPMENT OF A MONITORING AND
CONTROL SYSTEM FOR OPERATIONAL
STUDY OF DIVIDING WALL COLUMN PILOT



UNIVERSITI MALAYSIA PAHANG

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ABSTRAK

Permintaan bahan api fosil semakin meningkat dari tahun ke tahun tetapi sumber bahan api fosil berkurangan. Pada masa kini, para penyelidik cuba mencari sumber alternatif baru bagi mengurangkan kebergantungan terhadap bahan api fosil. Hidrogen merupakan salah satu bahan api alternatif yang menarik untuk dikaji. Tujuan kajian ini adalah untuk menilai terhadap kesan alam sekitar dan ekonomi bagi dua laluan proses penghasilan hidrogen iaitu metana (Kes1) dan etanol (Kes 2). Proses simulasi telah dijalankan dalam kajian ini dengan menggunakan perisian Aspen Plus versi 8.6. Reaksi stim reformasi metana dan etanol disimulasi berasaskan kepada tindakbalas kinetik. Data kinetik telah diperolehi melalui kajian literatur. Tindakbalas dilakukan dalam perisian Aspen Plus dengan menggunakan blok RPlug dengan menyusun kembali model kinetik Langmuir-Hinselwood-Watson (LHHW) dan model kinetic *power-law*. Pada masa yang sama, penulenan bagi hidrogen turut menggunakan kaedah simulasi. Pengesahsahihan data telah menunjukkan keputusan yang hampir sama dalam literatur. Selain itu juga, analisis sensitiviti juga telah dijalankan untuk melihat kesan beberapa parameter seperti suhu, tekanan, berat pemangkin dan nisbah masukan ke dalam rektor untuk kedua-dua kajian kes. Selepas itu, penilaian terhadap alam sekitar dan ekonomi telah dibuat. Data yang diperolehi telah digunakan untuk membuat perbandingan antara kedua-dua kajian kes. Penilaian kitaran hayat (LCA) telah digunakan dalam kajian ini untuk menilai kesan alam sekitar menggunakan perisian GaBi menggunakan kaedah ReCiPe untuk menilai impak alam sekitar bagi semua proses yang terlibat dalam kajian ini. Unit berfungsi bagi LCA dalam kajian ini adalah 1 kg untuk hidrogen. Secara keseluruhannya, 16 kategori impak telah dikaji dan hanya 3 menunjukkan kategori yang banyak memberi impak iaitu perubahan iklim, pengurangan fosil dan pengurangan air. Perbebasan gas rumah hijau tinggi untuk kes 2 iaitu 30.84 kg CO₂ eq. berbanding dengan kes 1 iaitu 9.44 kg CO₂ eq. Manakala, pengurangan fosil tinggi kes 2 iaitu 12.54 kg oil eq. berbanding kes 1 sebanyak 4.044 kg oil eq. Kes 2 juga menyebabkan penyusutan sumber air yang tinggi sebanyak 23.35 m³ eq berbanding kes 1 sebanyak 4.01 m³ eq. Penilaian ekonomi terhadap kedua-dua kajian kes telah dibuat. Kos modal untuk penghasilan hidrogen bagi kes 1 adalah kurang berbanding dengan kes 2 dengan perbezaan 7.92%. Manakala, kos utiliti untuk kes 1 lebih rendah berbanding kes 2 dengan perbezaan sebanyak 12.81%. Secara keseluruhannya, kes 1 iaitu hidrogen daripada metana adalah lebih mesra alam dan lebih jimat dalam kos CAPEX dan OPEX berbanding kes 2 walaupun daripada sumber tenaga yang boleh diperbaharui iaitu etanol.

ABSTRACT

Dividing wall column (DWC) provides a good alternative for processes using conventional distillation column due to possible saving in both energy and capital cost. So far, about 40 divided wall columns (DWCs) are in operation worldwide, about 30 of them within the BASF group. The potential of DWC is however restricted and not applied to broad range of the separation processes due to the challenges in design, simulate, operation and control. The internal configuration of DWC leads to changes in the controllability and operating mode thus becoming a potential hurdle for commercial implementation. In this work we perform several studies related to control and operation of DWC. Moreover, DWC exhibits a multiple input multiple output (MIMO) characteristics. For good process control it is important to consider the probability of pairing the controlled variables and manipulated variables and applying an effective feedback controller to each selected pairs. An appropriate controller pairing is important to reject the disturbances as well as maintaining the product specifications. In our knowledge, there are few research focuses of VDWC particularly for oleochemical products fractionation. Therefore, our aim in this work is to analyze the controllability of a VDWC for fractionation of oleochemical fatty acid.



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CHAPTER 1

FRACTIONATION OF OLEOCHEMICAL FATTY ACID USING VACUUM DIVIDING WALL COLUMN: A CONTROLLABILITY ANALYSIS

1.1 Abstract

Dividing wall column (DWC) provides a good alternative for oleochemical fractionation. However, the internal configuration and multiple input multiple output (MIMO) system of DWC leads to complexity in operation and control. This work aims to analyse the controllability of fractionating oleochemical fatty acid using vacuum dividing wall column (VDWC). To achieve this, Aspen Plus and Aspen Dynamics were used to develop a rigorous steady state and dynamic model of the column. Five manipulated variables (MVs) were considered namely reflux flowrate (L), distillate flowrate (D), bottom flow rate (B), side-stream flowrate (S) and vapour boilup (V) while controlled variables (CVs) were the product compositions. Pairing of MV and CV to determine the best 3×3 control configuration was performed using relative gain array (RGA) and singular value analysis (SVA). The selected control structure was tested on PID controllers for several regulatory and servo problem. The results of RGA and SVA shows that DSV was the best control configuration. Performance analysis was found to be successful in rejecting the disturbances as well as obtaining good set point tracking. However, distillate and bottom composition shows poor controllability compare to middle composition.

1.2 Introduction

Dividing wall column (DWC) provides a good alternative for processes using typical distillation (DC) column due to possible saving in both energy and capital cost. Because of its advantages, extensive research has been done and the first industrial application of DWC's was implemented in 1985 by BASF [1]. So far, about 40 DWCs are in operation worldwide and about 30 of them within the BASF group [2]. The potential of DWC is however restricted and not applied to broad range of the separation processes due to the challenges in design, simulate, operation and control [3,4]. In the

oleochemical industry particularly in Malaysia, mostly used typical (DC) for its product fractionation. In our recent study, fractionating oleochemical fatty acids using DWC reduces around 20% of capital and operating cost compared to typical DC [5].

The integration of two columns into one shell leads to changes in the operating mode and controllability thus becoming a potential hurdle for commercial implementation of DWC [6]. Moreover, distillation columns exhibit a multiple-input multiple-output (MIMO) system in which manipulated variables (MVs) affect multiple controlled variable (CVs) [7]. To provide good process control in such system it is crucial to consider the probability of pairing CV and MV and applying an effective feedback controller to each selected pair. A good pairing will ensure effective rejection of process disturbances as well as maintaining the product specifications. Despite various research on DWC, study on oleochemical fractionation using vacuum dividing wall column (VDWC) received less attention. Therefore, this study aims to analyze the controllability of VDWC for fractionation of oleochemical fatty acid. Our scopes of work in paper include development of steady state and dynamic model in Aspen Plus and Aspen Dynamics. Determination of suitable 3×3 control configuration using steady state relative gain array (RGA) and singular value analysis (SVA) and test the performance of the selected controller configuration to disturbances in feed flowrate, feed composition as well as set point change.

1.3 Methodology

The process under study involves fractionation of oleochemical fatty acids which constitute of three carbon chains namely C10, C12 and C14. The boiling point of C10, C12 and C14 are 270 oC, 299 oC and 326 oC respectively. To avoid product degradation, the column temperature was operated below 270 oC at pressure between 0.01 to 0.1 bar. The feed information is listed in Table 1. The feed comprises mostly of C12 and C10 is the most least. C10 will be fractionated at the distillate stream whereas C12 and C14 will be fractionated at side and bottom stream respectively. The product purity for each streams were set to 99 mole%.

The steady state VDWC model was developed in Aspen Plus. NRTL was chosen as the thermodynamic model due to the polarity of the fatty acid as well as low

operating pressure [5]. Instead of typical two or three model configuration, this work employs four RADFRAC model blocks to mimic the four internal and hydrodynamic behaviour of an actual DWC internal sections. Furthermore, four column configuration was usually applied for dynamic simulation. The product purity for each streams were set to 99 mole%. Design of the VDWC was based on the work by Othman & Rangaiah [5]. The design parameters are shown in Table 1.1.

Table 1.1 VDWC design parameters.

Reflux ratio	46.8
Stages (A/B/C/D) ^a	10 / 11 / 11 / 18
Feed stage	5 (at B)
Pressure, mbar	40
Feed flowrate, kg/h	6000
Mass fraction (C10/C12/C14)	0.05 / 0.71 / 0.24
Feed temperature, °C	30

^aA = Rectifying section, B = Pre-fractionation section, C = Middle section D = Stripping section.

1.3.1 Analysis tool

DWC implies a multi-input-multi-output (MIMO) control scheme. According to Koko and Barakat [8] there are seven degree of freedom (DOF) of DWC corresponding to seven MVs namely reflux flowrate (L), vapour boilup flowrate (V), side stream flowrate (S), distillate flowrate (D), bottom flowrate (B), liquid split ratio (RI) and vapour split ratio (Rv). However, RI and Rv are not suggested to be the MV because it could cause a serious operation and control problem [9,10]. In addition, perfect level control in reboiler and condenser were assumed. This reduces the DOF to three. The controlled variables (CVs) were xC10, xC12 and xC14 represent mole fraction of distillate, side and bottom stream, respectively. For a 3×3 configuration there are nine possible pairings.

In order to screen suitable pairing of MV-CV, relative gain array (RGA) was applied. RGA has been widely used among the researcher [9,10, 11,12] to examine the proposed control system in the distillation column and DWC. In RGA, the best control

loop pairing was determined by the steady-state gain (K). For a 3×3 system, the steady-state gain matrix is denoted by:

$$K_{ij} = \begin{pmatrix} K_{11} = \frac{\Delta y_1}{\Delta u_1} \Big|_{u_{2,3}} & K_{12} = \frac{\Delta y_1}{\Delta u_2} \Big|_{u_{1,3}} & K_{13} = \frac{\Delta y_1}{\Delta u_3} \Big|_{u_{1,2}} \\ K_{21} = \frac{\Delta y_2}{\Delta u_1} \Big|_{u_{2,3}} & K_{22} = \frac{\Delta y_2}{\Delta u_2} \Big|_{u_{1,3}} & K_{23} = \frac{\Delta y_2}{\Delta u_3} \Big|_{u_{1,2}} \\ K_{31} = \frac{\Delta y_3}{\Delta u_1} \Big|_{u_{2,3}} & K_{32} = \frac{\Delta y_3}{\Delta u_2} \Big|_{u_{1,3}} & K_{33} = \frac{\Delta y_3}{\Delta u_3} \Big|_{u_{1,2}} \end{pmatrix} \quad (1.1)$$

Where y indicates the output and u indicates the input. K_{11} in the matrix indicates the changes on y_1 when u_1 is altered while u_2 and u_3 are constant. K_{12} denotes the changes on y_1 when u_2 is altered but u_1 and u_3 are kept constant and so on. From the steady-state matrix, RGA (Λ) can be calculated using the following relationship:

$$\Lambda = K \otimes (K^{-1}) \quad (1.2)$$

Where \otimes denoted as the element by element multiplication. T denotes the transpose of the steady-state gain matrix. If $\Lambda_{ij} = 0$, it means that y_i does not respond to m_j thus m_j could not be used to control y_i . While $\Lambda_{ij} = 1$ means that y_i only responds to m_j and not interact with other manipulated variables. If $0 < \Lambda_{ij} < 1$ or $\Lambda_{ij} > 1$, then an interaction occurs because more than one y_i react to m_j . Last but not least, if $\Lambda_{ij} < 0$, it means that the interaction exists between the related manipulated and controlled variables is in opposite direction and cause instability. The least interaction, where the arrangement which gives a RGA with diagonal element values close to unity will be selected as the best pairing. For RGA, four possible control configuration schemes were studied namely DB/LSV, LB/DSV, DV/LSB and LV/DSB.

Alternatively, singular value analysis (SVA) was also applied. Singular value analysis (SVA) is an alternative to RGA for design of multivariable control systems. Singular values arise from the decomposition of K:

$$K = W\Sigma V^T \quad (1.3)$$

where Σ is the diagonal matrix of singular values. W and V are unitary matrices. The columns of W are referred to as the input singular vectors whereas the columns of V are the output singular vectors. The final matrix property of interest in the condition number (CN). If K is non-singular, the CN number of K is a positive number defined as the ratio of the largest and smallest nonzero singular values:

$$CN = \frac{\sigma_1}{\sigma_r} \quad (1.4)$$

If the CN value is small, then the multivariable effects of uncertainties are not likely to be serious. For SVA, three CVs were manipulated by three out of five MVs with total nine possible pairings. In this work, both RGA and SVA were applied to screen possible MV-CV for the VDWC. RGA was conducted first followed by SVA. The steady state gains for these analysis were obtained through the developed Aspen Plus steady state model. The best possible matching from RGA and SVA analysis will be selected for closed loop performance analysis

1.3.2 Dynamic model

In dynamic model, modifications were made to the steady state model. Pressure changes along the column were considered by fitting all four column stages were with Mellapak 350Y packing. Reflux drum and column sump were sized assuming residence time of 5 min and 10 minutes, respectively. Valves, pumps and compressors were added to the dynamic model flowsheet to achieve pressure consistency. Because of the system dependency on pressure, pressure driven mode was selected. The default control loops were reflux drum level control, column sump level control, top column pressure control and bottom column temperature control. Pressure check was made to ensure pressure consistency prior exporting to Aspen Dynamics.

1.3.3 Closed loop response

The selected controller configuration from the previous step were added to the dynamic model flowsheet. PID controller was adopted in this work. No measurement delay was included. The controller settings were determined using conservative Ziegler-Nichols (ZN) open loop test tuning method. Fastest loop was tuned first and then closed, followed by the second controller tuning while the first controller remains closed. This continues until all control loops were tuned. The controller was tested to several scenarios. First was a regulatory problem in which the process was subjected to feed rate change and change in feed compositions. For the latter three scenarios were considered each with different sets of feed compositions. Second was the servo problem where set point change was introduced to the distillate, side and bottom product composition.

1.4 Results and discussions

The RGA controllability indices results for all four control configurations is given in Table 1.2. RGA values with less than 0 were excluded due to caused instability whilst value close to 1 is preferred. The most suitable pairing of MV-CV are $D-x_{C10}$, $S-x_{C12}$ and $V-x_{C14}$ as their value were close to 1 compared to the other indices. This indicate each MV has a good inner interaction with its correspond CV whilst minimally affect other variables. Table 1.2 shows the CN results from SVA analysis. Pairing no 7 ($D-x_{C10}$, $S-x_{C12}$, $V-x_{C14}$) and 9 ($D-x_{C10}$, $V-x_{C12}$, $B-x_{C14}$) are having the smallest CN value and therefore are preferred as it shows minimal dependency to other MVs. Pairing 7 is consistent with the 3×3 RGA, but not pairing 9. Therefore, DSV control loop arrangement was selected and the updated controller loop of the VDWC is shown in Figure 1.1a.

Table 1.2 RGA steady state controllability indices for 3×3 control problem. The CVs are xC10, xC12 and xC14.

Controlled variables, x_i	Manipulated variables, m_j		
	L	S	V
xC10	-3.3070	4.3080	-0.0010
xC12	4.2978	-3.4826	0.1848
xC14	0.0092	0.1746	0.8162
	L	S	B
xC10	-3.0273	4.0208	0.0065
xC12	0.6892	-0.4059	0.7166
xC14	3.3381	-2.6149	0.2768
	D	S	V
xC10	0.9916	-0.0949	0.1033
xC12	-0.1153	1.1151	0.0002
xC14	0.1237	-0.0202	0.8965
	D	S	B
xC10	-13.2389	-3.4140	17.6529
xC12	0.7780	-0.2493	0.4713
xC14	13.4609	4.6632	-17.1242

Table 1.3 CN values for steady state controllability analysis for 3×3 control problem. The controlled variables are xC10, xC12 and xC14.

Pairing	Controlled variables	Manipulated variables a	CN
1	xC10, xC12, xC14	LDS	2765.00
2	xC10, xC12, xC14	LSV	147.84
3	xC10, xC12, xC14	LSB	222.00
4	xC10, xC12, xC14	LDV	139.46
5	xC10, xC12, xC14	LDB	18.81
6	xC10, xC12, xC14	LVB	1004.33
7	xC10, xC12, xC14	DSV	3.83
8	xC10, xC12, xC14	DSB	33.03
9	xC10, xC12, xC14	DVB	4.22

^aIn each pairing, the first controlled variables is paired with the first manipulated variable, and so on i.e. in pair 1, xC10 is paired with L, xC12 is paired with D and xC14 is paired with S.

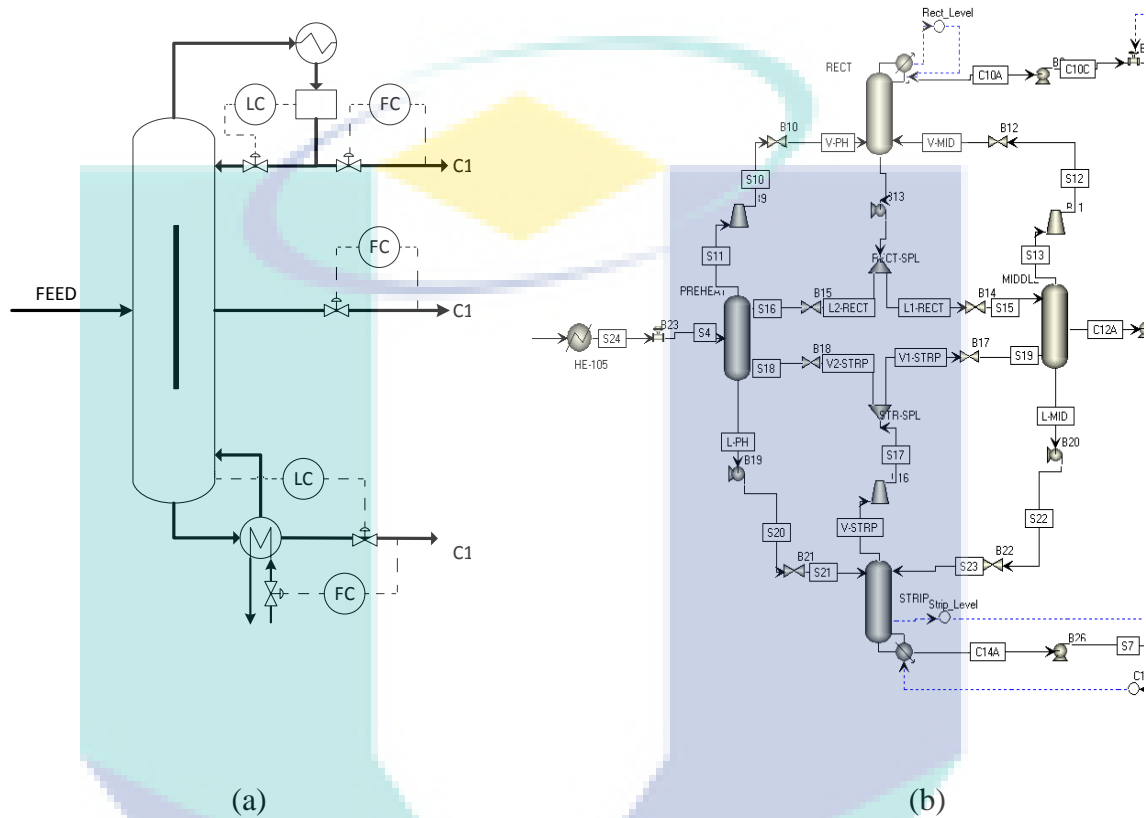


Figure 1-1 (a) DSV control configuration (b) Aspen Dynamics VDW layout for DSV control configuration

The controller loops were added to the dynamic model. Figure 1.1b shows the Aspen Dynamic flowsheet. The dynamic flowsheet layout consists of five control loops corresponding to the DSV configuration as well as level controller for sump and reflux tank. The controller tuning for each controller loop was performed using the Tuning Option. The value is shown in Table 1.3. Time variation of the product composition when subjected to 2% and 4% changes in the feed rate is shown in Figure 1.3a & 1.3b, respectively. We see from the simulations that middle composition (C12) provides much better control compared to distillate (C10) and bottom composition (C14). Bottom composition oscillates more than distillate composition. Variation in feed flowrate seems to have minimal effect on the middle composition. This is due to the large amount of C12 in the feed stream and minimal effect in the internal flows. Overall, the feed rate change disturbance was able to be rejected.

Figure 3c on the other hand, shows the time profile when the process was subjected to several feed composition variation scenarios namely Scenario 1, 2 and 3. From the results, distillate composition has poor controllability compared to the middle and bottom composition with high overshoot and long settling time. Bottom composition incur some oscillation but has a lower overshoot and faster settling time compare to distillate composition. Since large portion of the feed contain C12, variation of C10 and C12 in the feed affect the internal flows and purity which eventually effect the controllability of the distillate and bottom stream. Middle composition is however unaffected by variation in feed composition with good controllability. Overall, variation of feed stream has minimal effect on middle composition.

Table 1.4 PID tuning results based on ZN tuning method.

Loop	K	τ_I	τ_D
D	107.4	90.2	8.5
S	111.0	5.6	-
V	4.0	23.1	2.1
Reflux level	1	20	-
Sump level	1	20	-

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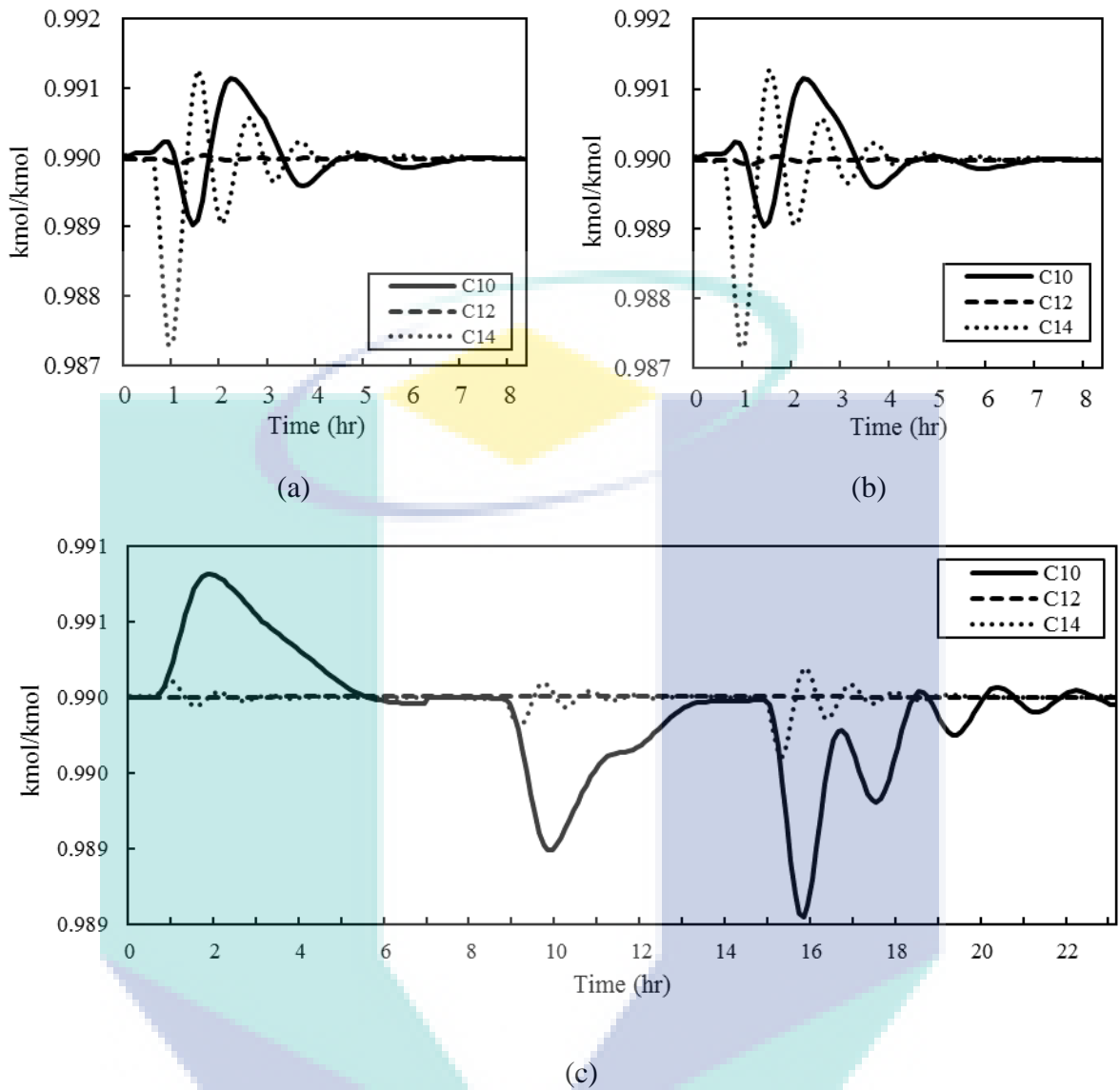


Figure 1-2 Closed loop response to (a) 2% step change in feed flow (b) 4% step change in feed flow (c) variation in feed composition

Figure 3 shows the result of set point tracking in all product stream with change of 0.001. We can see that distillate and bottom composition control shows some oscillation and settle after almost five hours. Middle composition on the other hand, has very good response with almost no overshoot and fast settling time. This is reasonable and consistent with previous results due to the large amount of C12 in the feed stream and has minimal effect in the internal flows.

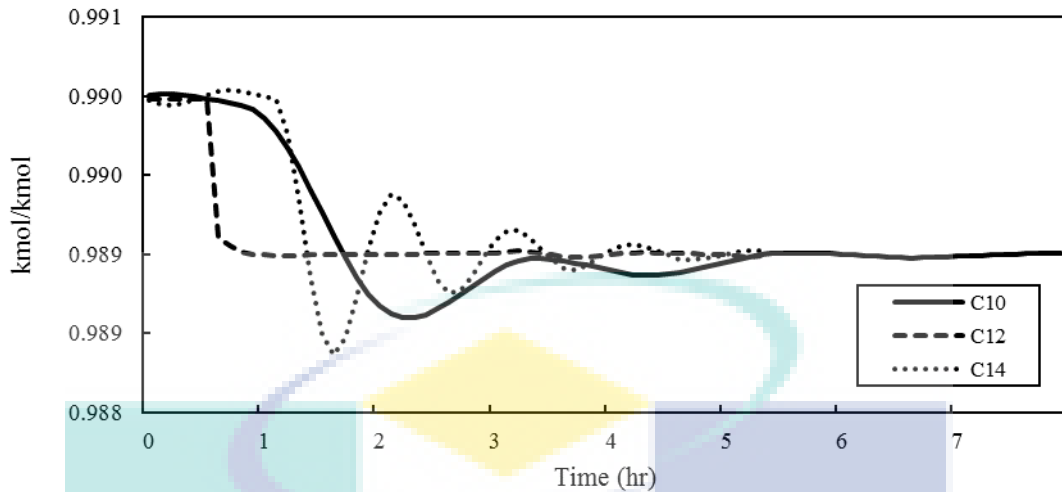


Figure 1-3 Closed loop response to set point change of 0.001 in distillate, middle and bottom composition.

1.5 Conclusions

VDWC controllability analysis for fractionating oleochemical fatty acid has been studied in this work. Steady state and dynamic model of the column have been successfully developed using Aspen Plus and Aspen Dynamics. Pairing of 3×3 control configuration was performed using relative gain array (RGA) and singular value analysis (SVA). Both approach resulted in DSV configuration being the most suitable configuration. The configuration was applied to the dynamic model and the controller performance was tested to feed rate and feed composition variations as well as set point change. Performance analysis was found to be successful in rejecting the disturbances as well as obtaining good set point tracking. However, distillate and bottom composition shows poor controllability compare to middle composition mainly due to the composition proportion of the feed.

References

- [1] Jobson M 2005 Dividing wall distillation comes of age Chemical Engineer no. 766 pp 30-31
- [2] Adrian T, Schoenmakers H and Boll M 2004 Model predictive control of integrated unit operations: Control of a divided wall column Chemical

- Engineering and Processing: Process Intensification 43(3) p 347-355
- [3] Dejanović I, Matijašević L and Olujić Ž 2010 Dividing wall column – a breakthrough towards sustainable distilling. Chemical Engineering and Processing: Process Intensification 49(6) p 559-580
- [4] Yildirim Ö, Kiss A A and Kenig E Y 2011 Dividing wall columns in chemical process industry: a review on current activities Separation and Purification Technology 80(3) p 403-417
- [5] Othman M R and Rangaiah G P 2019 Performance Analysis of Dividing Wall Column Model for Fractionation of Oleochemical Fatty Acid Journal of Oil Palm Research In Press
- [6] Kiss A A and Rewagad R R 2011 Energy efficient control of a BTX dividing-wall column Computers & Chemical Engineering 35(12) p 2896-2904
- [7] Sankaranarayanan D and Deepakkumar G 2014 Implementing the Concept of Relative Gain Array for the Control of MIMO System: Applied to Distillation Column. Annals University Regional Centre, Coimbatore
- [8] Koko I O and Barakat T A 2016 Modelling and Control Analysis of Dividing Wall Distillation Columns University of Khartoum Engineering Journal Oct 23 5(1)
- [9] Mutalib M A and Smith R 1998 Operation and control of dividing wall distillation columns: Part 1: Degrees of freedom and dynamic simulation Chemical Engineering Research and Design 1;76(3) p 308-18
- [10] Cho Y, Kim B, Kim D, Han M and Lee M 2009 Operation of divided wall column with vapor sidedraw using profile position control Journal of Process Control 1;19(6) p 932-41
- [11] Wang S J and Wong D S 2007 Controllability and energy efficiency of a high-purity divided wall column Chemical Engineering Science 1;62(4) p 1010-25
- [12] Strandberg J P 2011 Optimal operation of dividing wall columns. PhD thesis, Norwegian University of Science and Technology, Department of Chemical Engineering

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CHAPTER 2

TEMPERATURE CONTROL OF VACUUM DIVIDING WALL COLUMN – CASE STUDY ON OLEOCHEMICAL FATTY ACID FRACTIONATION

2.1 Abstract

Analysis of oleochemical compositions in distillation column often have large process delays. Inferential control is commonly used by means of stage temperature as the measured variable which provide more responsive composition control. This work aims to evaluate the performance of temperature control in vacuum dividing wall column (VDWC) for fatty acid oleochemical fractionation. Sensitivity analysis was used to determine the relationship between stage and temperature difference for changes in the manipulated variables. The most sensitive tray was selected and implemented to a DSV control configuration in Aspen Dynamics following the work by Othman (2019b). PID controller were adopted with different PI and PID settings using Ziegler-Nichols (ZN) and Internal Model Control (IMC) method. Both methods were compared based on the settling time and overshoot. The best setting was then fine-tuned before tested to set point tracking without any disturbances. From the sensitive analysis, temperature at stage 6, 29 and 34 were selected to be inferred to distillate, middle and bottom product composition respectively. PID controller setting based on ZN method provide the best setup with fastest settling time and smallest overshoot and provide good performance for set point tracking.

2.2 Introduction

Oleochemical industry particularly in Malaysia mostly uses typical distillation column (DC) for its product fractionation. In process design perspective dividing wall column (DWC) shows very promising alternative to DC which able to reduce around 20% of capital and operating cost (Othman, 2019a). However, one of the potential hurdles for commercial implementation of DWC is the challenges in design, simulate, operation and control (Dejanovic, Matijasevic, & Olujic, 2010; Yildirim et al., 2011) as well as complexity in operating and controllability due to the introduction of a wall within the column internal (Kiss & Rewagad, 2011). Various research has been

conducted for DWC control. One of the most common controlled variable in distillation column is composition. However, composition is difficult to measure. For such variable, inferential control is often implemented which uses easily measure process variables i.e. temperature, pressure and flow to infer more difficult process variables such as compositions and molecular weight. Parrish & Brosilow (1985) stated that for higher order and long-dead-time processes, inferential control systems will generally outperform conventional feedback control systems. Because of that, inferential control has excellent performances such as disturbance resisting and set point tracking. However, the application is restricted when strong load disturbance exists or stable control accuracy and response speed are highly required in the system. Besides, it can be much less expensive in terms of capital and operating cost as well as can provide measurement that are not available any other way. According to Ansari and Tade (2008) inferential models which are based on fast and continuously available temperature, pressure and flow measurements reduce the negative impact of the sample intervals and time delays with minimum compromise on accuracy. The resulting continuous and fast response keeps the product qualities on specifications and minimizes the quality giveaway. In order to establish composition inferential control, one needs to know the particular correlation between process variables and product composition. According to Marlin (1995), a good inferential control is when inferred variable is closely related to true variable so that controlling inferred controlled variable will maintain true controlled variable close to desired value. The use of tray temperature to infer composition is widely used in distillation column (Luyben, 2005).

Various studies have been conducted for temperature inferential control in DWC. For example Wang et al. (2018) investigated temperature inferential control of DWC for separating ethanol, n-propanol, and n-butanol ternary mixture. Yuan et al. (2017) studied inferential temperature control for benzene–toluene–o-xylene in DWDC system. Ignat & Woinaroschy (2011) on the other hand analyzed the controllability of inferential temperature of 4 point control structure for a case study of separation of a ternary nonideal methanol – ethanol – 1-propanol mixture in a DWC. Most of inferential control studies of DWC focuses on petrochemical processes. Study on vacuum dividing wall column (VDWC) inferential control particularly for oleochemical industries however received less attention. Moreover, oleochemical products were analyzed using analytic apparatus i.e. HPLC which had large process delay. This

practically hindered composition as the controlled variable. Hence, temperature tray could be adopted to infer product composition. Therefore, this work aims to evaluate the performance of stage temperature control in vacuum dividing wall column (VDWC) for oleochemical application. Oleochemical fatty acid was used as the case study.

2.3 Methodology

2.3.1 Steady state and dynamic modelling

The process under study involves fractionation of oleochemical fatty acids which constitute of three carbon chains namely C10, C12 and C14 with boiling point of 270 °C, 299 °C and 326 °C respectively. To avoid product degradation, the column temperature was operated below 270 °C at pressure between 0.01 to 0.1 bar. The product purity for each streams were set to 99 mole%. Due to the polarity of the fatty acid as well as low operating pressure NRTL thermodynamic model and its variances can be used. In this work NRTL was chosen. For process flowsheeting, four RADFRAC model blocks were used for both steady state and dynamic modelling using Aspen Plus and Aspen Dynamics, respectively. Steady state model was used for sensitivity analysis to study the relationship between tray temperature and product composition as well as to determine the tray number to be inferred for controlling the product composition. Dynamic model was used for controllability analysis of the inferential control configuration. The control configuration used in the dynamic model was based on Othman (2019b). Othman conducted a controllability analysis of VDWC for oleochemical fatty acid fractionation using relative gain array (RGA) and singular value analysis (SVA). From his findings, it was found that DSV control configuration was the best 3x3 control pairing due to the low interaction between control loops. Therefore, DSV control configuration was adopted for this work. Figure 2.1 shows the DSV control configuration.

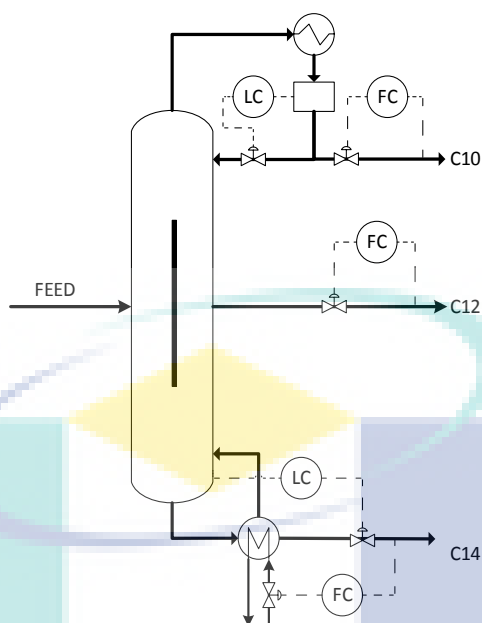


Figure 2-1 DSV control configuration

2.3.2 Steady state sensitivity analysis

In this work, sensitivity analysis was applied to establish a column temperature profile for different operating points which was then compared with base case data. Table 2.1 shows the base case condition. In this work, several operating points were considered by changing the manipulated variables. Three conditions were considered (1) $\pm 10\%$ change in distillate flowrate, D (2) $\pm 10\%$ change in middle flowrate, S and (3) ± 60 change in reboiler duty, V. From the column profile, temperature deviation from the base case (ΔT) were plotted. From this plot, one can determine the most sensitive tray. The identified tray for each product were then implemented in Aspen Dynamics and evaluated for its controllability performance.

Table 2.1 Base case steady state condition.

Parameters	
Flowrate, kg/h	
- Feed	6240
- Distillate	316.11
- Side stream	4405.92
- Bottom	1517.96
Product composition, mol%	

- C10 at distillate	99
- C12 at side stream	99.8
- C13 at bottom	99
Reflux ratio	60.65
Reboiler duty, kW	2275.5

2.3.3 Tuning and closed loop response

The selected inferential variable from the previous step were added to developed DSV based control configuration by replacing the controlled variables from composition to the designated tray temperature. PID controller was adopted in this work. No measurement delay was included. Ziegler-Nichols (ZN) tuning method is a heuristic method of tuning PID controller which is widely used for tuning. Meanwhile, Internal Model Control (IMC) tuning method is also adopted because of its ability to compensate disturbances and model uncertainty. However, ZN setting result in very good disturbance response for integrating processes, but are otherwise known to result in rather aggressive setting and also give poor performance (Skogestad, 2004). On the other hand, IMC setting it result in poor disturbance response for integrating processes but are robust and generally give very good responses for set point changes (Rivera, Morari, & Skogestad, 1986). Therefore, in this work both ZN and IMC setting was compared before further fine-tuned. The controller setting was subjected to set point change for distillate, side and bottom product composition. The controller performance was tabled in term of their settling time and overshoot. Tuning with fastest settling time and small overshoot were selected before further fine-tuned.

2.4 Results and discussions

Sensitivity analysis for distillate, middle and bottom sections are performed individually. For distillate section, Figure 2.2a gives the temperature deviation for $\pm 10\%$ change in distillate flow. The temperature deviation for increase flow rate was negative, while positive for decrease flow rate. The plot also shows several sensitive trays. For flowrate increment tray number 2 to 7 and 28 to 34 were the most sensitive whereas for flowrate decrement tray 2 to 8 and 21 to 31 were the most sensitive. In this

work tray 6 were selected as the preferred temperature tray to be inferred to distillate product stream. Note that, For the tray number other than this region, it would be a very poor inferential variable, because the sensor error and low magnitude noise would invalidate any correlation drawn from simulation. Besides, a small temperature deviation indicate that valve saturation can easily occur and operability region could be limited.

Figure 2.2b gives the temperature deviation for 10% change in the middle stream. The temperature deviation also shows the same pattern as in (a) but with fewer peaks. For flowrate increment tray number 21 to 31 were the most sensitive while tray number 28 to 38 were the most sensitive whereas for decrease in middle flowrate. Tray 29 were selected as the preferred temperature tray to be inferred to middle product stream. Figure 2.2c gives the temperature deviation for 10% change in the reboiler duty. The temperature deviation was apparent for 60% decrease in reboiler duty with different peak at the stripping section and rectifying section while not so much change for reboiler duty increment. For reboiler duty increment tray number 4 to 8 and 29 and 37 were the most sensitive Tray 34 were selected as the preferred temperature tray to be inferred to middle product stream due its location near the reboiler which quickly affected by changes in reboiler duty. Figure 2.3a shows updated inferential control loop of DSV based control configuration of the VDWC. The loop was implemented in Aspen Dynamics as shown in Figure 2.3b. For PID controller setting, ZN method was compared to IMC. Its performance towards set point tracking were evaluated.

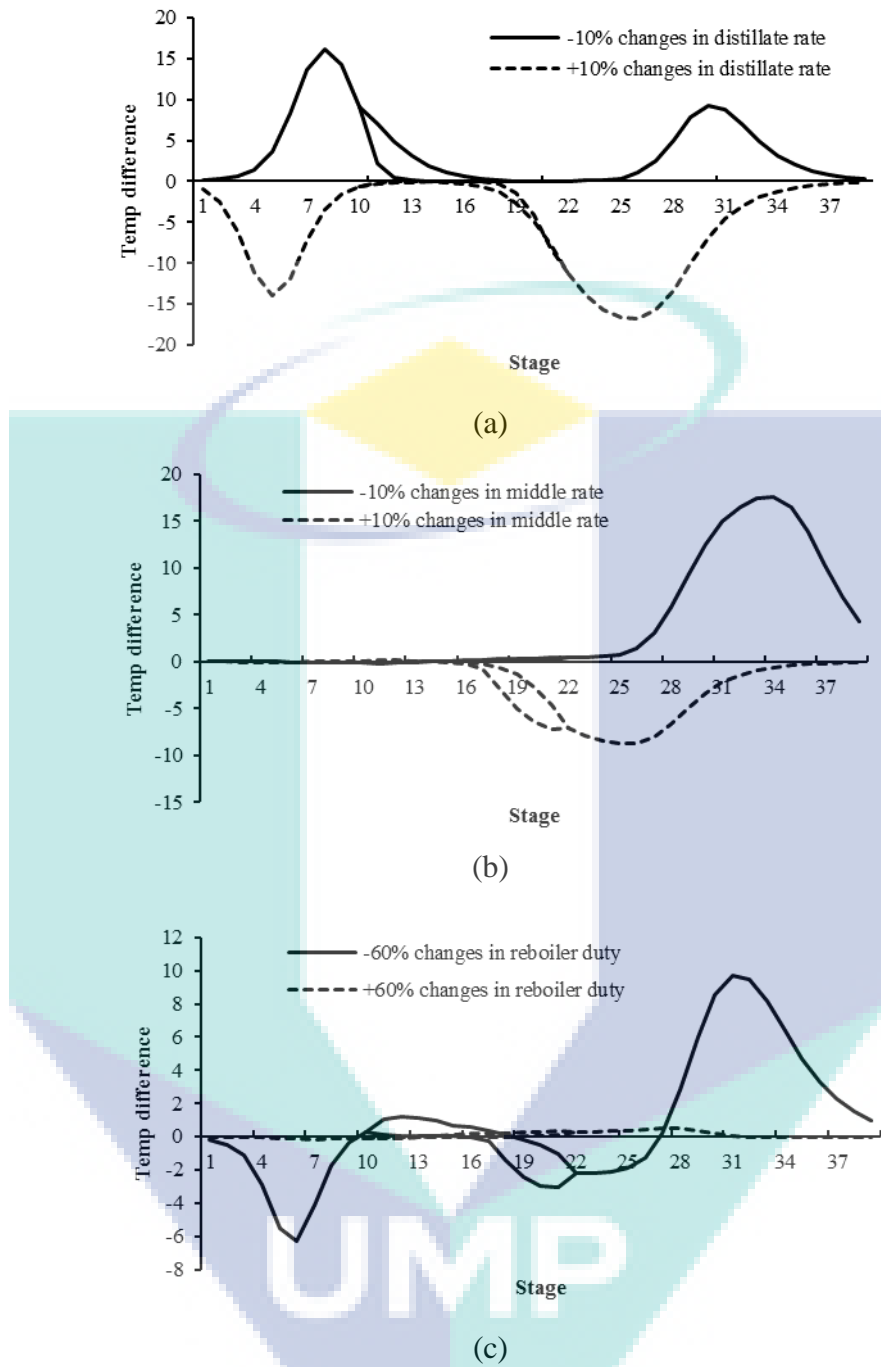


Figure 2-2 Stage temperature sensitivity analysis (a) $\pm 10\%$ changes in distillate flow (b) $\pm 10\%$ changes in middle flow (c) $\pm 60\%$ changes in reboiler duty

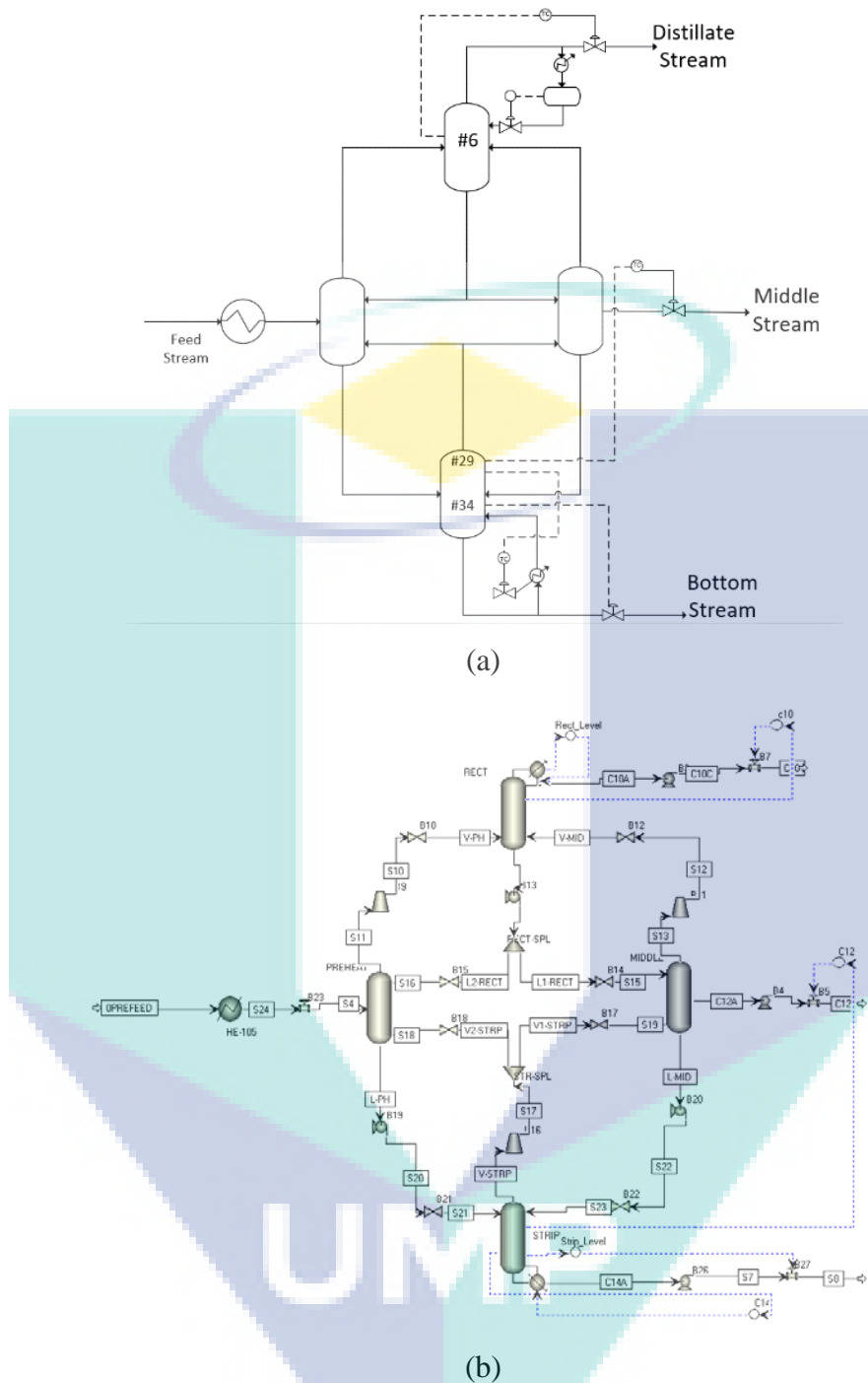


Figure 2-3 a) Inferential control using identified tray in DSV control configuration (b) the corresponding flowsheet in Aspen Dynamics

Table 2.2 shows the ZN and IMC tuning value and well as its corresponding controller performance for set point tracking. Overall, in terms of settling time and overshoot both approach able to compromise and meet the satisfactory target. PID controller based on ZN tuning however provides better performance in term of settling time and overshoot. Hence, PID-ZN setting was adopted and further fine-tuned. Table 2.3 and Figure 2.4 shows the tuning values and well as the controller performance for

set point tracking after fine-tuned. Both distillate and bottom stream shown fast response with minimal overshoot. However, for middle stream when the settling time is longer than other controller loop. This due to the large amount of C12 which is require longer time for heating.

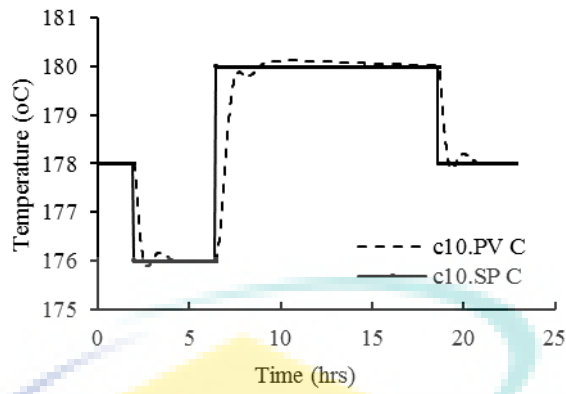
Table 2.2 Control settings for the proposed inferential control in DSV control configuration with the comparison between Ziegler-Nicols and IMC method as well as comparison between PI and PID

Control Loop		Ziegler Nichols		IMC
		PI	*PID	PID
Distillate Stream, C10	Gain	86.87513	144.7919	2.350773
	Integral Time	35.47682	26.63425	375.1321
	Derivative Time	0	4.26148	5.251207
	Settling Time	6.09 hours	2.64 hours	3.42 hours
	Overshoot	0.45 %	5 %	0.45 %
Middle Stream, C12	Gain	5.742162	95.7021	22.86846
	Integral Time	7150.78564	2000	2171.743
	Derivative Time	0	858.9532	295.5417
	Settling Time	1983 hours	321.1 hours	359.1 hours
	Overshoot	0 %	0%	0 %
Bottom Stream, C14	Gain	0.921668	1.536113	0.393431
	Integral Time	60.83798	45.67416	39.798
	Derivative Time	0	7.307865	7.038206
	Settling Time	16.83 hours	11.62 hours	30.31 hours
	Overshoot	44.2 %	36.35 %	70.7 %

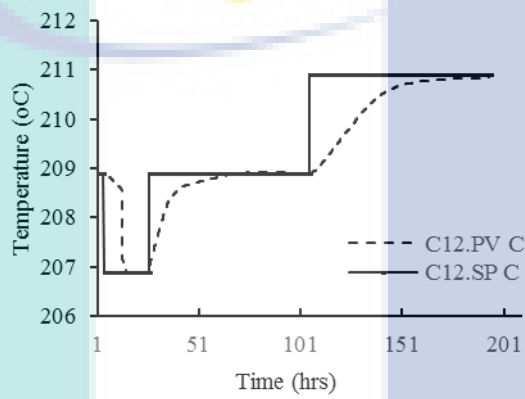
Table 2.3 After tuning of ZN PID controller for distillate, middle and bottom stream

Control Loop		Before tuning	After tuning
Distillate Stream, C10	Gain	144.7919	6.6
	Integral Time	26.63425	600
	Derivative Time	4.26148	4.2618
	Settling Time	2.64 hours	1.82 hours
	Overshoot (< 5 %)	5 %	4.75%
Middle Stream, C12	Gain	2521.756	2.2
	Integral Time	35.80275	39
	Derivative Time	5.72844	5
	Settling Time	46.7	50.62 hours
	Overshoot (< 5 %)	12.4 %	0 %
Bottom Stream, C14	Gain	1.536113	80
	Integral Time	45.67416	3
	Derivative Time	7.307865	7.03826
	Settling Time	11.62 hours	1.31 hours
	Overshoot (< 5 %)	36.35 %	3.05%

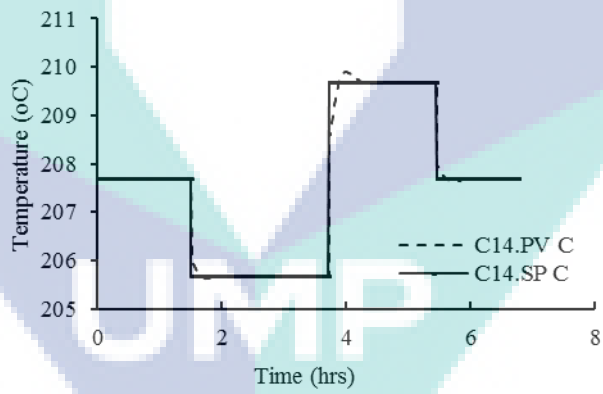
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(a)



(b)



(c)

Figure 2-4 Set-point tracking for tray temperature change in (a) stage 6 (b) stage 29 (c) stage 34

2.5 Conclusions

The sensitivity analysis was successfully determining the best tray temperature location for distillate at 6th stage, middle at 29th stage and bottom at 34th stage based on the most temperature deviation from base case. The comparison from two tuning methods; ZN and IMC, with different controller setting of PI and PIC reveal that PID-ZN setting was preferred with good controller response in term of better settling time and minimum overshoot. The fine-tuned has improved the quality of controller response target thus give good set point tracking in the stage temperature.

References

- [1] Ansari, R. M., & Tadé and M. O. 2008. *On-Line Inferential Models in Nonlinear Multivariate Control Applications*. 4(1–2), 71–83
- [2] Cho Y, Kim B, Kim D, Han M and Lee M 2009 *Operation of divided wall column with vapor sidedraw using profile position control* (Journal of Process Control) p 932-41
- [3] Dejanović, Igor & Matijašević, Ljubica & Olujić, Ž. 2010. *Dividing wall column—A breakthrough towards sustainable distilling*. vol 49 (Chemical Engineering and Processing). p 559-580
- [4] Ignat, Radu & Woinaroschy, Alexandru. 2011. *Dynamic analysis and controllability of dividing-wall distillation columns using a four points control structure*. vol 73(UPB Scientific Bulletin, Series B: Chemistry and Materials Science).
- [5] Illner, M., & Othman, M. R. 2015. *Modeling and Simulation of a Dividing Wall Column for Fractionation of Fatty Acid in Oleochemical Industries*. (Perintis E-Journal)
- [6] Marlin, T. E. 1995. *Process Control: Designing Processes and Control Systems for Dynamic Performance*. McGraw-Hill.
- [7] Adrian T, Schoenmakers H and Boll M 2004 *Model predictive control of integrated unit operations: Control of a divided wall column* *Chemical Engineering and Processing: Process Intensification* vol 43 (Elsevier) p 347-355
- [8] Kano, M., Showchaiya, N., Hasebe, S., & Hashimoto, I. 2001. *Inferential control of distillation compositions: Selection of model and control configuration*. vol

34 (IFAC Proceedings) p 347–352.

- [9] Kiss, Anton & R. Rewagad, Rohit. 2011. *Energy efficient control of a BTX dividing-wall column*. vol 35 (Computers & Chemical Engineering) p 2896-2904
- [10] Yildirim, Ömer & Kiss, Anton & Kenig, Eugeny. 2011. *Dividing wall columns in chemical process industry: A review on current activities*. (Separation and Purification Technology). P 403-417
- [11] Kano, M., Miyazaki, K., Hasebe, S., & Hashimoto, I. 2000. *Inferential control system of distillation compositions using dynamic partial least squares regression*. (Journal of Process Control), p 157–166
- [12] Kim, Kwangil, et al. 2012 “*Two-Point Temperature Control Structure Selection for Dividing-Wall Distillation Columns*”. vol. 51, no. 48, (Industrial & Engineering Chemistry Research), pp. 15683–95
- [13] Kiss A A and Rewagad R R 2011 *Energy efficient control of a BTX dividing-wall column* (Computers & Chemical Engineering) p 2896-2904
- [14] Koko I O and Barakat T A 2016 *Modelling and Control Analysis of Dividing Wall Distillation Columns vol 5. No.1* (University of Khartoum Engineering Journal)
- [15] Luan, Shujun, et al..2013 *A Simplified Temperature Difference Control Scheme*.vol 52.no.7 (Industrial & Engineering Chemistry Research) pp. 2642–60,
- [16] Luyben, W. L. 2006. *Evaluation of criteria for selecting temperature control trays in distillation columns*. vol 16, (Journal of Process Control) p115–134.
- [17] Othman M R and Rangaiah G P 2019a *Performance Analysis of Dividing Wall Column Model for Fractionation of Oleochemical Fatty Acid* (Journal of Oil Palm Research In Press)
- [18] Othman M.R. 2019b *Controllability Analysis of A Vacuum Dividing Wall Column For Oleochemical Fatty Acid Fractionation*. IOP:MSE
- [19] Othman, Mohamad, and Umarul Imran. 2015 *Feasibility Study of Fractionating Fatty Acid Using Dividing Wall Column (DWC): Modelling Approach*.
- [20] Parrish, J. R., & Brosilow, C. B. 1985. *Inferential control applications*. *Automatica*, vol 2 , p 527
- [21] Qian, Xing, et al. *Comparison of Stabilizing Control Structures for Dividing Wall Columns*. vol. 49 (IFAC-PapersOnLine), pp. 729–34

- [22] Rivera, D., Morari, M., & Skogestad, S. 1986. *Internal Model. 4. PID Controller Design* vol 25 (Industrial & Engineering Chemistry, Process Design and Development,) p 252-265.
- [23] Skogestad, Sigurd. 2004. *Simple Analytic Rules for Model Reduction and PID Controller Tuning.* vol 13 (Journal of Process Control) p 291-309.
- [24] Strandberg J P 2011 *Optimal operation of dividing wall columns.* PhD thesis, (Norwegian University of Science and Technology)
- [25] Tyreus, B. D., & Luyben, W. L. 1992. *Tuning PI controllers for integrator/dead time processes.* 31(11), (Industrial & Engineering Chemistry Research) p 2625–2628
- [26] Wang, Jianxin & Yu, Na & Chen, Mengqi & Cong, Lin & Sun, Lanyi. 2017. *Composition control and temperature inferential control of dividing wall column based on model predictive control and PI strategies.* vol 26 (Chinese Journal of Chemical Engineering).
- [27] Yuan, Yang et al. “Asymmetrical temperature control of a BTX dividing-wall distillation column.” (2017).

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The logo of Universiti Malaysia Pahang (UMP) is a large, stylized letter 'U' composed of several overlapping triangles in shades of blue and teal. The letters 'UMP' are written in a bold, white, sans-serif font across the center of the 'U' shape.

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CHAPTER 3

FRACTIONATION OF OLEOCHEMICAL FATTY ACID USING VACUUM DIVIDING WALL COLUMN: A CONTROLLABILITY ANALYSIS

3.1 Introduction

In oleochemical fatty acid production, the main process that occur to split the component from feed into fatty acid is separation. The separation or fractionation occur at 2 main stages, first at the beginning section where the splitting of crude palm kernel (CPO) or crude palm oil kernel (CPKO) into product of fatty acid and by product of glycerine and secondly refining fatty acids to remove the heavy ends and volatiles. The pure fatty acid is used as important raw material in the manufacture of soaps, washing powder and other personal care product there it is important to purify the fatty acid as high as a product as possible. These processes are take places in fractional distillation columns or it can be also called fractionator with various type of design & technology by considering factors such as of high vacuum, low pressure drop, low bottoms temperature, minimum hold up, short residence time and high packing efficiency. Thus, steadily increasing requirements on fractionation demands in the oleochemical industry require advanced separation technology. (Faessler et al., 2007) .

Therefore, one of the advanced distillation column is dividing wall distillation column. A DWC is in essence a fully thermally coupled distillation sequence with only one condenser and one reboiler regardless of the number of products. The entire sequence is packed into a single shell by means of one or more vertical partition walls (Dejanović et al., 2010). This condition has resulting the advantages of lower space requirement due to function of two columns combined in one shell, decreased energy requirements, creation of pre fractionator on the feed side and avoid remixing of products.

However, current limitation of applying dividing wall distillation column in oleochemical sector due to limited familiarity, higher requirements on operation, potential corrosion problems and limited flexibility. On top of that, the readily

challenges to control fatty acid distillation column become hindering factors such as process uncertainties because of frequent process changes in term of feed quality and disturbances, process nonlinearity and greater cost on maintenance. Particularly maintenance on instrumentation with countless sensors and controller, which are vital elements for operation to rely on in ensuring smooth control and monitor the process variables.

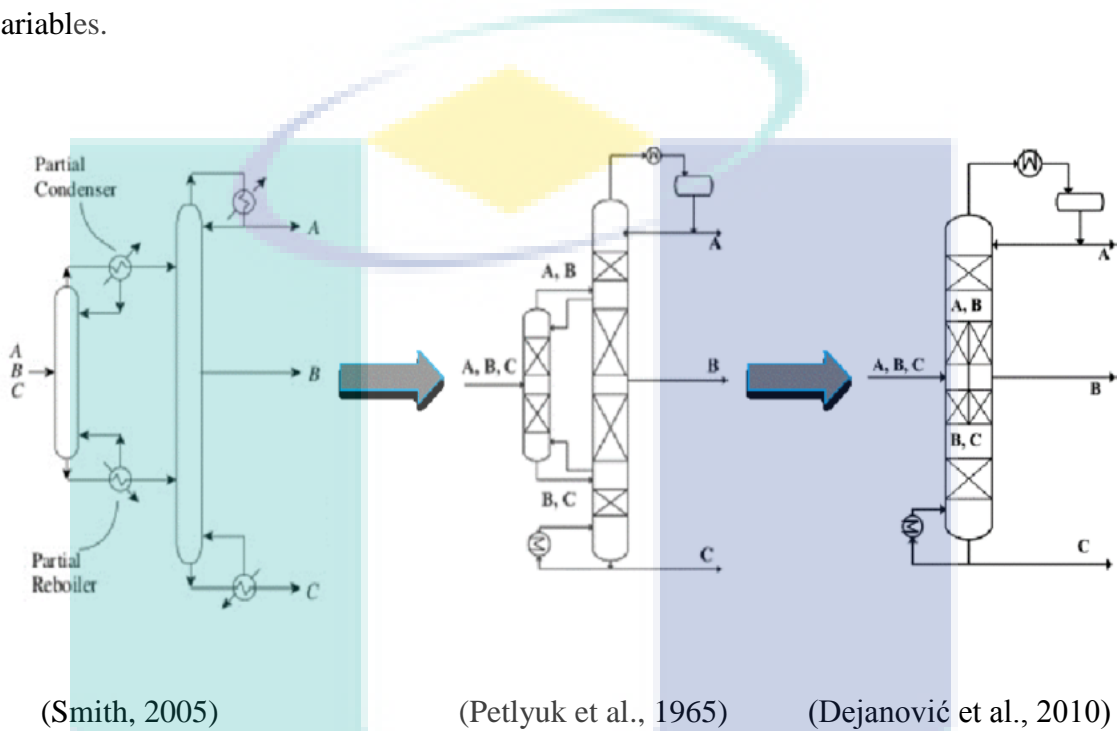


Figure 3-1 Development of Dividing Wall Distillation Column ((Lorenz et al., 2018)

Another limitation happen is in controlling this fractionator to get quality product as required. The current practice to achieve that purpose is by controlling the appropriate temperature of distillation column and supported by offline laboratory analysis of the product, carried out in hourly interval. This approach shall be improved by subscribing latest control strategy with minimum human intervention. Another issue is when inference of temperature with product purity as related by thermodynamic equilibrium is used and it is accurate in the condition where the laboratory analysis is reliable and correct as well as process is not subject to serious disturbance. However, this ideal situation does not always happen. Many processes tend to be have difficulties such as frequent changes in quality of raw materials and interruption of utilities. changes of feedstock composition alter the thermodynamic relationship between temperature and product purity at distillation outlet. The unique relation between the setpoint temperature and product purity needed for success of indirect strategy is

doubtful thus exposing operation to greater uncertainties. This can result in excessive off-specification products especially when operation is rectifying by changing from one operating region to another. Although experienced panel operators or engineer can handle these, dependence on human intervention still allowing to unnecessary uncertainties.

Thus to encounter these shortcomings, inferential models which is based on fast and continuously temperature, pressure, flow measurement is explore to reduce the negative impact of the sample intervals and time delays with minimum compromise on accuracy. (Ansari and Tade, 2008) Inferential measurement is a powerful methodology that allows difficult to measure primary variables be inferred from other easily measure secondary variables. The example is use temperature, flow or pressure to estimate the product purity generated by distillation column. If the model is accurate, the output used as feedback for automatic control. In essence, inferential measurement systems mimic what experience process operators and engineers do in operating process plant with elimination of human inconsistencies factor with continuous and fast response thus keep the product qualities on specifications and minimizes the quality giveaway. Besides, sensors used in inferential control tend to have high reliability because ultimately the purpose is as well to replace expensive and less reliable sensors.

3.1.1 Background

Various research has been conducted for DWC control involving controllability analysis as well as inferential control. Luyben performed a simulation and proposed a four-point control structure for a three-product dividing wall column. (Ling and Luyben, 2009) proposed new control structure that controls these purities and also minimizes energy consumption by controlling a composition of the heaviest component in the prefractionator. (van Diggelen et al., 2010) compared conventional PID controller with obtained H_{∞} controller synthesis and μ -synthesis Several work by (Rewagad and Kiss, 2012) ,(Adrian et al., 2004) and (Dohare et al., 2015) have also been reported for the use of model predictive control for dividing wall column . Most of this studies focuses on petrochemical processes. Study on vacuum dividing wall column (VDWC) control particularly for oleochemical industries however received less attention. Therefore, it is our focused in this work to evaluate the performance of inferential control strategy for

high product purity in dividing wall column (VDWC) for oleochemical application. Oleochemical fatty acid was used as the case study.

3.1.2 Problem Statement

Inferential control of distillation composition was investigated by many researchers, yet limited found apply in dividing wall distillation column oleochemical fatty acid fractionation. For instance (Kano et al., 2001) analyse the inferential control using methanol, ethanol and propanol. (Wang et al., 2018) has investigated temperature inferential control of DWC for separating ethanol, n-propanol, and n-butanol ternary mixture. (Yuan et al., 2017) has studied inferential temperature control for the process medium of benzene–toluene–o-xylene in DWDC system. (Ignat and Woinaroschy, 2011), has analyzed the controllability of inferential temperature of 4 point control structure for a case study of separation of a ternary nonideal methanol – ethanol – 1-propanol mixture in a DWC. Therefore, the gap found to be closed by apply inferential control for DWC in oleochemical

Second issue is to establish good inferential control for composition control require the most accurate process variable to be inferred as controlled variable either as temperature, pressure or flow. The study shows in most cases temperature is used such as (Kano et al., 2001) and (Hori and Skogestad, 2007). The control scheme introduced by Shiren et al. (1997) utilizes the easily available tray temperatures to estimate the product compositions and uses the estimated compositions in feedback control. (Ignat and Woinaroschy, 2011) investigated control of DWC with inferential temperature measurements. The key point of configuring an effective temperature inferential control scheme lies in the selection of the sensitive stage or its possible combinations with reference stages (i.e., temperature difference (TD) and double temperature difference (DTD)) as the controlled variable (CV) for each control loop (Luyben, 1969; Yu and Luyben, 1984; Mejdell and Skogestad, 1991). Although a number of criteria were already proposed and used frequently for the selection of sensitive and reference stages, no easy-to-use methods are currently available for the effective discrimination of sensitive stages and their possible combinations with reference stages and this makes the derivation of temperature inferential control systems a considerably challenging issue, especially for the highly integrated DWDCs. For that (Luyben, 2006) has

suggested criteria for selecting temperature control trays in distillation columns. However most case refer to conventional single distillation column whereby thermally coupled distillation column such as DWC it generates stronger interaction between control loops of column making controllability worse than any conventional scheme (Mizey et al., 1998) but in some cases it can exceed conventional system in controllability (Hernandez et al., 2005). These uncertainties therefore perceive as gap to validate on the selection of the best temperature tray using DWC in oleochemical application which is still remain unverified.

As suggested by previous work related in establishing inferential control for composition control, PI control is used (Kano et al., 2001). Since the temperature is slow response process derivative is required. (Ignat and Woinaroschy, 2011) in his study of control DWC use PID loops with inferential temperature measurements. According to him, PID controllers have the advantage of a short development time, and small development effort. This is a gap to be further study by comparing PI with PID.

The ZN settings are too aggressive for most process control applications, where oscillations and overshoot are usually not desired. On the other hand, the IMC-settings in are known to result in a poor disturbance response for integrating processes but are robust and generally give very good responses for setpoint changes. (Skogestad, 2003). IMC has drawn a great interest to be implemented for the unstable process due to its effectiveness design principle [Shamsuzzoha and Lee, 2008]. IMC also have shown to be more robust when compared to the conventional controller and renowned for its setpoint tracking capabilities [Fieg et al., 1996; Marlin, 2000]. The IMC philosophy is based on the Internal Model Principle which states that the control can only be achieved by using the model that able to represent the control system. Therefore, comparing between IMC with ZN is required to find the best controller setting in order to give best controller response.

3.1.3 Motivation

The motivation behind of this project is based on the fact that promising cost saving ranging from 20-30 % as studied by Nguyen (2015) in term of CAPEX and OPEX for applying DWC in fatty acid fractionation in Malaysia. Besides, this

breakthrough shall contribute to technology advancement in fatty acid fractionation in Malaysia as none of 18 oleochemical operating companies in Malaysia yet to subscribe this technology as result of low confidence level in controllability and operational excellence.

3.1.4 Objective

A good inferential control is it has coherent inferential model which can estimate a product composition from on-line measured process variable which in this case is temperature. However, this paper primarily scope limited in evaluating the temperature controller loops which consist best measurement location and best controller setting use for inferential temperature control for product purity.

3.1.5 Scope of Study

The scope of work addressed in this project is as follows:

1. Identify stage temperature using sensitivity criterion method
2. Propose controller loop and controller tuning setting via comparison ZN vs IMC, PI vs PID
3. Register $\pm 2\%$ (setpoint tracking) for distillate, middle stream and bottom stream to evaluate performance of controller response using settling time and overshoot as the criteria.

3.2 Inferential Control Structure

In general, inferential control is the controlled variables that are difficult to measure are estimated from some easy to measure process variables and then used in feedback control. Inferential control has many excellent performances such as disturbance resisting and set point tracking, however, the application is restricted when strong load disturbance exists or stable control accuracy and response speed are highly required in the system. It configuration basically uses easily measure process variables (T, F, P) to infer more difficult quantities such as compositions and molecular weight. With that it can substantially reduce analyzer delay as supported by (Parrish and

Brosilow, 1985) that state in high-order, long-dead-time processes, inferential control systems will generally outperform conventional feedback control systems.

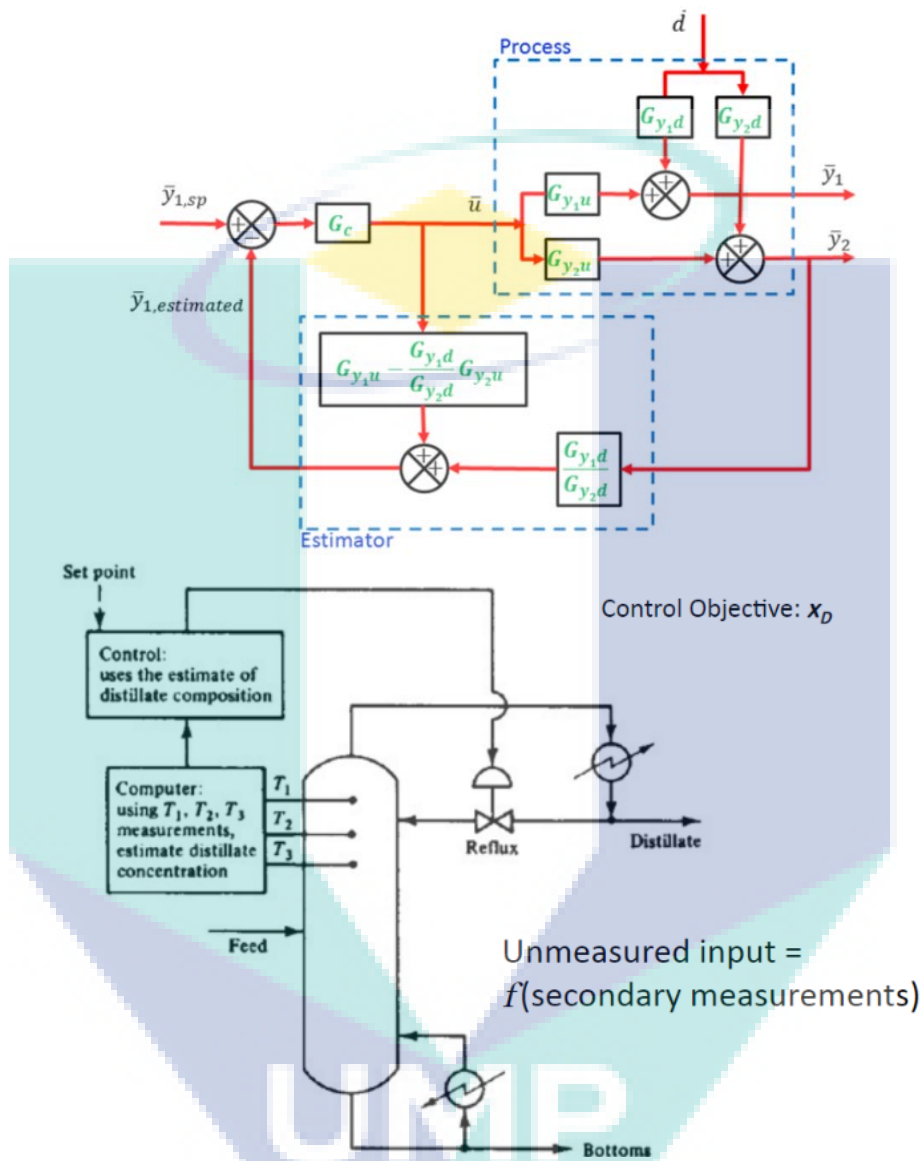


Figure 3-2 Inferential Control (Stephanopoulos, 1985)

Besides, it can be much less expensive in terms of capital and operating cost as well as can provide measurement that are not available any other way. According to (Ansari and Tadé, 2008) inferential models which are based on fast and continuously available temperature, pressure and flow measurements reduce the negative impact of the sample intervals and time delays with minimum compromise on accuracy. The resulting continuous and fast response keeps the product qualities on specifications and minimizes the quality giveaway.

In order to establish inferential control, the need to know a particular correlation what process variable correlates really well with product composition. The basic inferential control features consist of software sensors where the inferred values of the primary variable are used as feedback signals to an external controller such as PI controller. According to (Marlin, 1995) the good inferential control is when inferred variable is closely related to true variable so that controlling inferred controlled variable $CV_i(s)$ will maintain true controlled variable $CV_i(t)$ close to desired value.

According to (Luyben, 2005)), temperatures are widely used to provide inferential control of compositions. Temperature sensors are inexpensive, reliable and introduce only small measurement lags in the control loop. In a binary system with constant pressure, temperature is uniquely related to composition. This is not true in multi-component systems, but temperatures at appropriate locations in a distillation column can often provide fairly accurate information about the concentrations. In addition according to (Hori and Skogestad, 2007) Strandberg and Skogestad temperature is a good indicator of composition and is easy to measure. Temperature control is fast and can keep the compositions (and split) in the column close to nominal value and hence preventing “drift” in the event of disturbances. Therefore (Luan et al., 2013) put the attention on right location of temperature control whereby the two temperature controls located respectively in the rectifying and stripping section of the column to maintain the top and bottom product qualities. The two temperature difference control loops arranged respectively each side of dividing wall with their temperature measurement arranged above and below the locations for introducing feed to prefractionator and withdrawing product from main column.

3.3 DWDC application in oleo chemical industry

According to (Nguyen, 2015) Divided wall columns integrate two (or more) different separation units into one single device with one (or more) vertical partitions in the central section. Dividing wall splits a single column into two parts: a pre-fractionator section and a main column. It uses only one reboiler and one condenser.

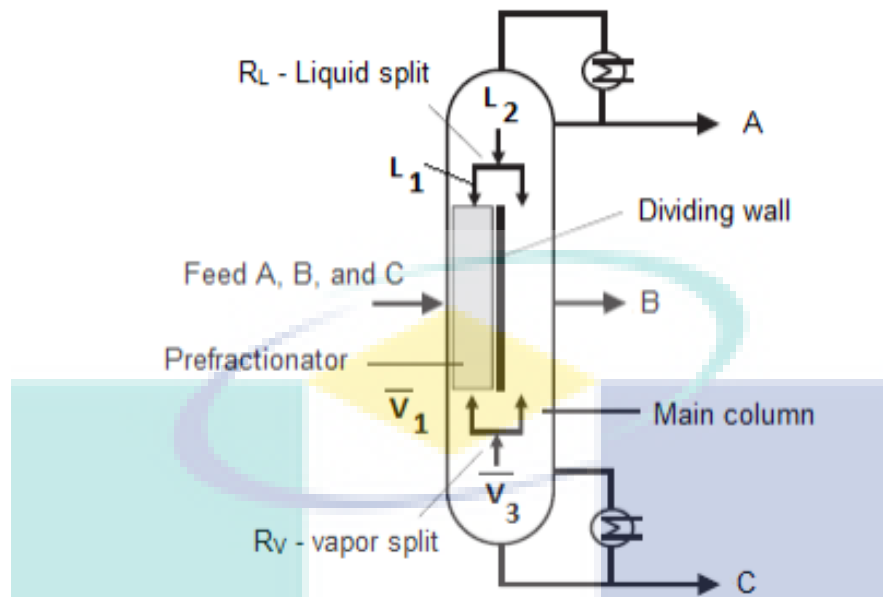


Figure 3-3 Separation for ternary mixture in the divided wall column (Rewagad and Kiss, 2012)

Figure 3.3 shows a divided wall column for separation of a ternary mixture. Considering separation of a ternary mixture A, B, and C, in which the component B is the distributed component. The feed is introduced into the prefractionator while distillate, side, and bottom products are removed from the main column. Component B is distributed between the top and bottom of the prefractionator section. The top of the prefractionator section contains mainly component A, a part of component B and a little component C. The bottom of the prefractionator section contains mainly component C, a part of component B and a little of component A. The upper part of the main column separates components A and B and the lower part of the main column separates components B and C. The liquid stream (L_2) from the condenser and vapor stream (V_3) from the reboiler are split on the two sides of the dividing wall. The liquid split R_L is the ratio between the liquid stream L_1 and liquid stream L_2 while the vapor split R_V is ratio between the vapor stream V_1 and the vapor stream V_3 . Divided wall columns can save both energy demand and capital cost. In fact, depending on the type of applications, desired purities of products, and relative volatilities of component, energy and capital costs are often reduced by 20 to 50% compared to traditional configurations (Kaibel, 2014). The DWC offers the following advantages:

(1) Lower capital investment

For separation of the ternary mixture shown in figure 2.1, the traditional sequences require at least two columns with two re-boilers and two condensers. However, the divided wall column needs only one column, one re-boiler and one condenser. Therefore, it leads to savings in investment cost.

(2) Reduced energy requirements

The conventional arrangement for separating a ternary mixture uses a direct sequence with two columns to obtain three pure products as shown in Figure 3.4

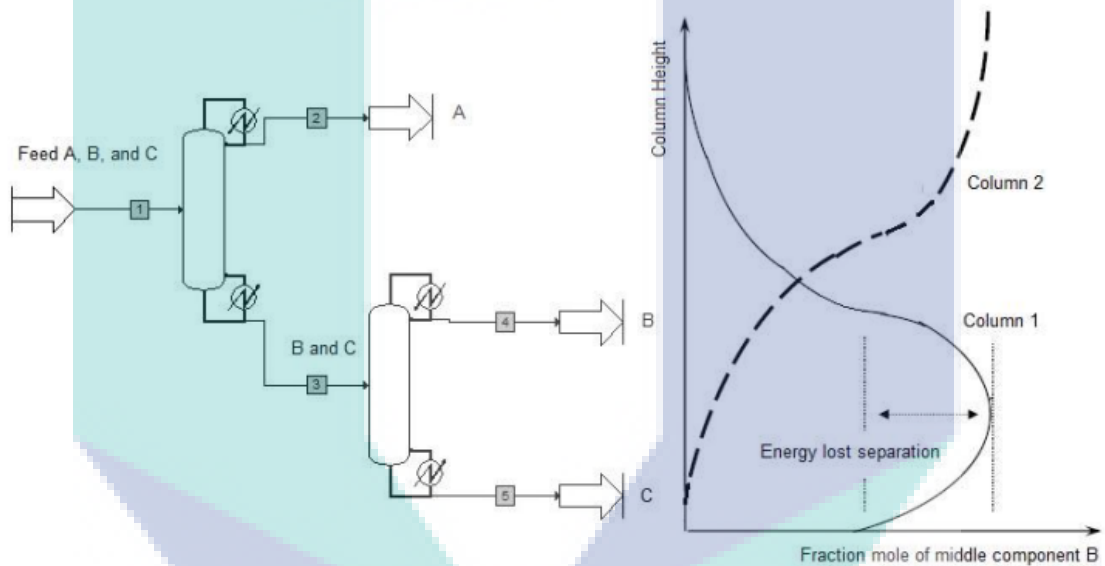


Figure 3-4 Energy is lost separating middle component B in the conventional arrangement (Pendergast et al., 2009)

(3) High purity for all products

Compared with a simple side-draw column, a higher purity of middle product can achieve in the divided wall column. Therefore, when a high purity middle component is desired, a divided wall column should be considered.

(4) Less construction volume

For multicomponent mixture separations, a divided wall column has only one reboiler and one condenser to obtain pure products. Therefore, the system needs less construction volume than traditional sequences. Moreover it does not need pipes connecting the two columns. Although a divided wall column may offer the potential for a savings in both capital and energy costs, the dividing wall columns have some main drawbacks. They are:

(1) Higher columns owing to the increased number of theoretical stages.

A divided wall column will be taller and have a larger diameter than of the two conventional columns.

(2) Increased pressure drop due to the higher number of theoretical stages.

A divided wall column operates with one reboiler and one condenser. Therefore, the condenser operates at the lowest temperature while the reboiler operates at the highest temperature. However, compared to the direct or indirect sequences with two columns, the reboiler of first column and the condenser of second column operate at middle range temperatures.

(3) Only one operating pressure is available.

A divided wall column operates at only one operating pressure. In comparison, traditional sequences may operate with different operating pressures in the two columns.

3.4 Controllers tuning and closed loop response

3.4.1 Feedforward Control

Feedback is reactive: there must be an error before corrective actions are taken. However, in some circumstances it is possible to measure a disturbance before it enters the system and this information can be used to take corrective action before the disturbance has influenced the system. The effect of the disturbance is thus reduced by measuring it and generating a control signal that counteracts it. This way of controlling a system is called feedforward. Feedforward is particularly useful to shape the response

to command signals because command signals are always available. Since feedforward attempts to match two signals, it requires good process models; otherwise the corrections may have the wrong size or may be badly timed.

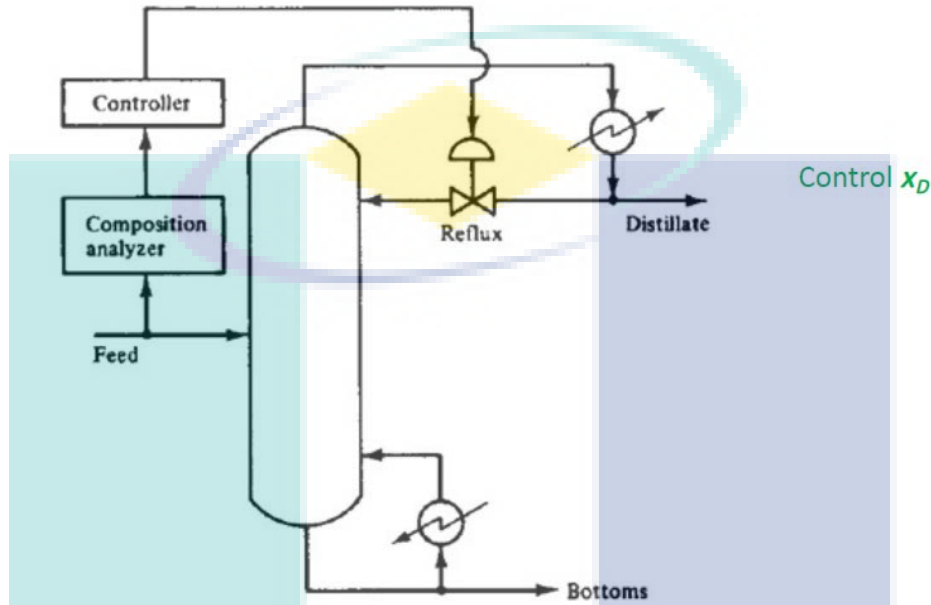


Figure 3-5 Feedforward control scheme in distillation column (Stephanopoulos, 1985)

3.4.2 PID Feedback Control

The distinguishing feature of the PID controller is the ability to use the three control terms of proportional, integral and derivative influence on the controller output to apply accurate and optimal control. A PID controller, which continuously calculates an error value $e(t)$ as the difference between a desired setpoint $SP=r(t)$ and a measured process variable $PV=y(t)$ and applies a correction based on proportional, integral, and derivative terms. The controller attempts to minimize the error over time by adjustment of a control variable $u(t)$, such as the opening of a control valve, to a new value determined by a weighted sum of the control terms.

Term P is proportional to the current value of the $SP - PV$ error $e(t)$. For example, if the error is large and positive, the control output will be proportionately large and positive, taking into account the gain factor "K". Using proportional control alone will result in an error between the setpoint and the actual process value, because it

requires an error to generate the proportional response. If there is no error, there is no corrective response.

Term I accounts for past values of the SP – PV error and integrates them over time to produce the I term. For example, if there is a residual SP – PV error after the application of proportional control, the integral term seeks to eliminate the residual error by adding a control effect due to the historic cumulative value of the error. When the error is eliminated, the integral term will cease to grow. This will result in the proportional effect diminishing as the error decreases, but this is compensated for by the growing integral effect.

Term D is a best estimate of the future trend of the SP – PV error, based on its current rate of change. It is sometimes called "anticipatory control", as it is effectively seeking to reduce the effect of the SP – PV error by exerting a control influence generated by the rate of error change. The more rapid the change, the greater the controlling or dampening effect.

Tuning – The balance of these effects is achieved by loop tuning to produce the optimal control function. The tuning constants are shown below as "K" and must be derived for each control application, as they depend on the response characteristics of the complete loop external to the controller. These are dependent on the behaviour of the measuring sensor, the final control element (such as a control valve), any control signal delays and the process itself. Approximate values of constants can usually be initially entered knowing the type of application, but they are normally refined, or tuned, by "bumping" the process in practice by introducing a setpoint change and observing the system response.

The overall control function can be expressed mathematically as

$$u(t) = K_p e(t) + K_i \int_0^t e(t') dt' + K_d \frac{de(t)}{dt}, \quad \text{Eq. 3.1}$$

where K_p , K_i and K_d , all non-negative, denote the coefficients for the proportional, integral, and derivative terms respectively (sometimes denoted P, I, and D). In the standard form of equation, K_p , K_i and K_d , are respectively replaced by K_p / T_i and $K_p T_d$; the advantage of this being that T_i and T_d have some understandable physical meaning as they represent the integration time and derivative time respectively

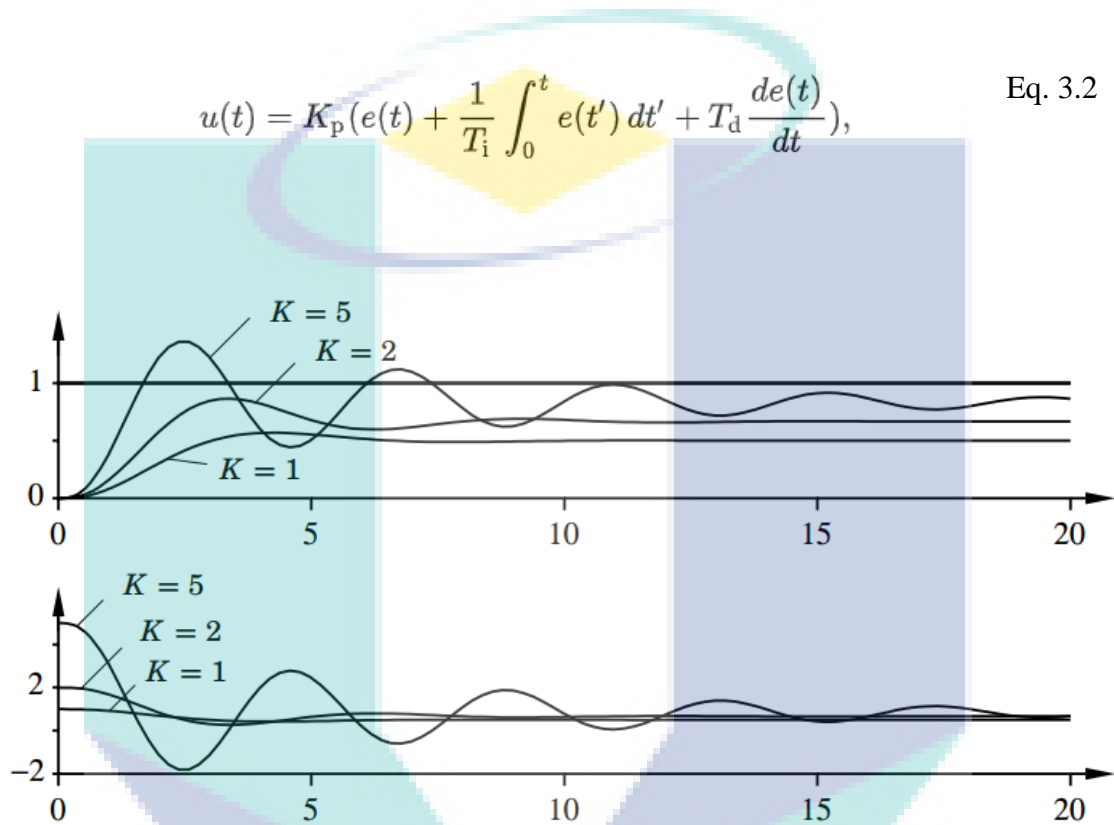


Figure 3-6 Simulation of closed-loop system with proportional control. The process transfer function is $P(s) = 1/(s+1)^3$ (° Astrom, 2002)

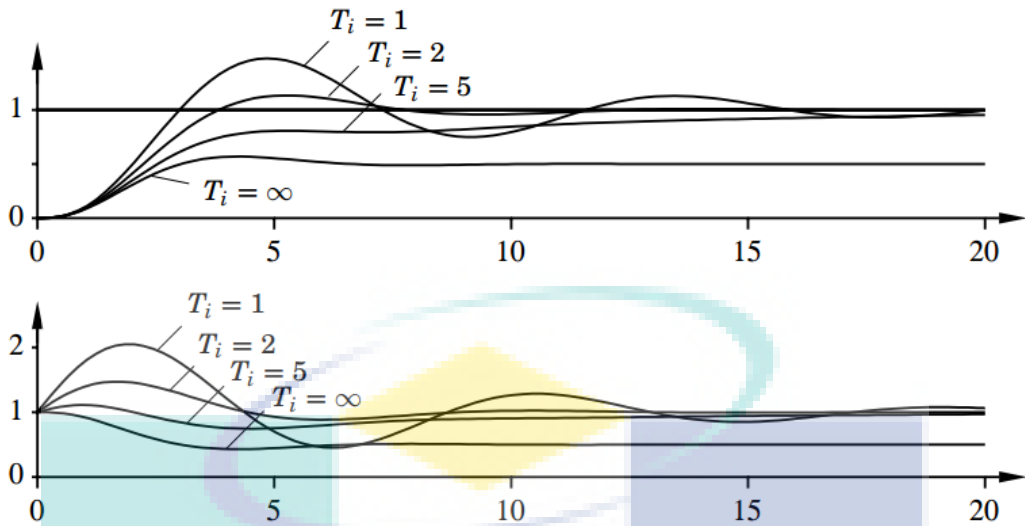


Figure 3-7 Simulation of closed-loop system with proportional and integral control. The process transfer function is $P(s) = 1/(s+1)^3$ and the controller gain is $K=1$ (° Astrom, 2002)

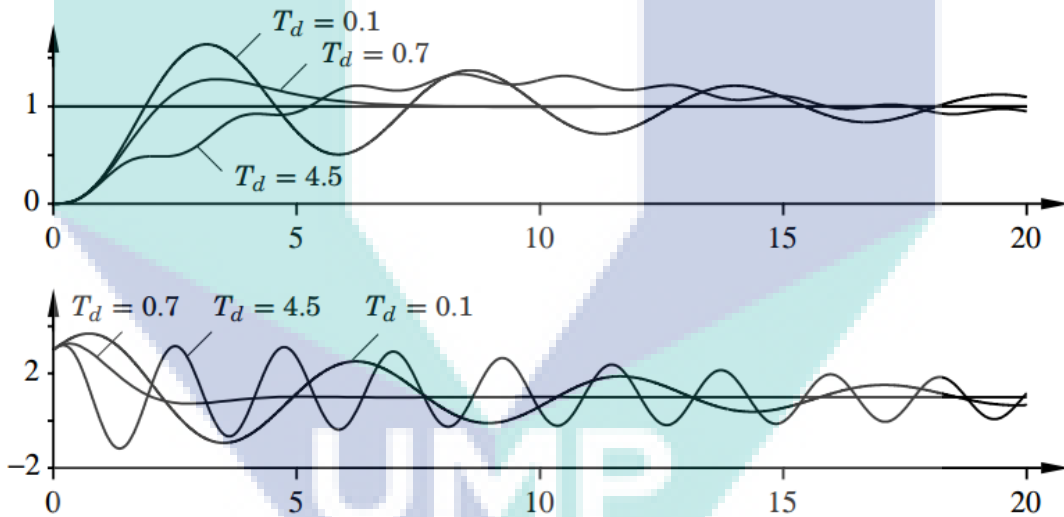


Figure 3-8 Simulation of closed-loop system with proportional, integral control and derivative. The process transfer function is $P(s) = 1/(s+1)^3$ and the controller gain is $K=1$ and $T_i=2$ (° Astrom, 2002)

3.4.3 Process Models

Controller settings of proportional, integral and derivatives can be derived from process models which consist of 3 components which are process gain, time constant and dead time.

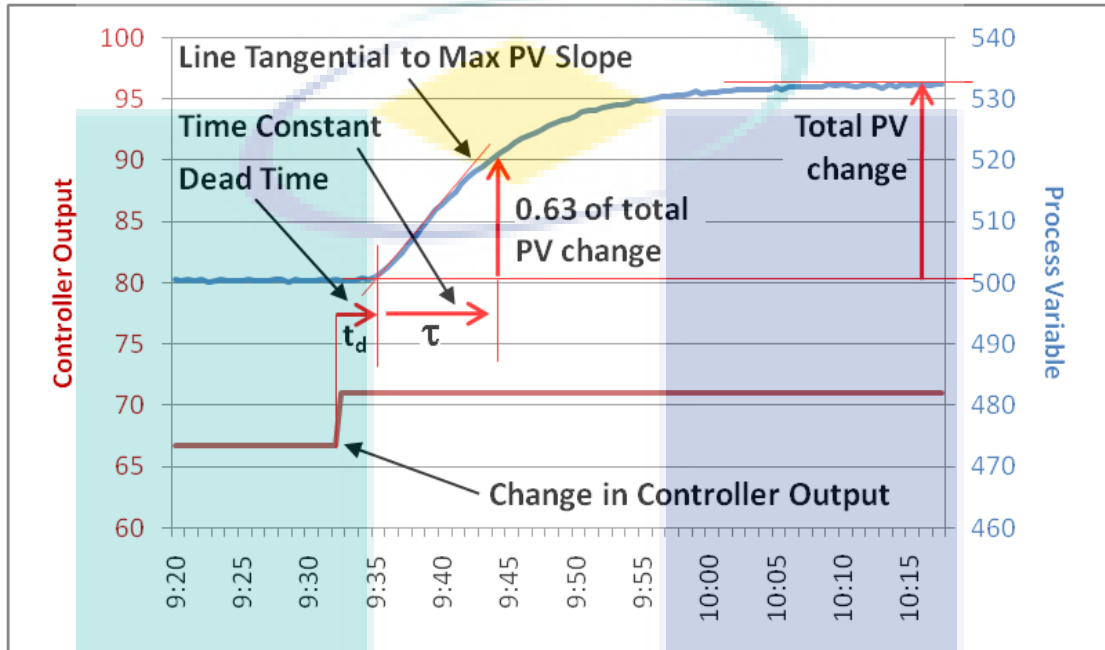


Figure 3-9 Step test result to acquire process model value (Dr. David Corrigan, 2012)

The process gain is the change in the output y induced by a unit change in the input u . The process gain is calculated by evaluating the change in output which can be referred as change in controller output divided by the change in input which can be referred as total PV change in percentage at steady state initial and final conditions.

$$\text{Process Gain} = \Delta \text{ Output} / \Delta \text{ Input} \quad \text{Eq. 3.3}$$

The process gain affects the magnitude of the response, regardless of the speed of response. For Time Constant, it refers to how fast the process variable responds to changes in the manipulated variable. Given a change in $u(t) = \Delta u$, the solution to the linear first-order differential (without time delay) becomes:

$$y(t) = \left(e^{-t/\tau_p} \right) y(0) + \left(1 - e^{-t/\tau_p} \right) K_p \Delta u \quad \text{Eq. 3.4}$$

If the initial condition $y(0)=0$ and at $t=\tau_p$, the solution is simplified to the following.

$$y(\tau_p) = \left(1 - e^{-\tau_p/\tau_p} \right) K_p \Delta u = \left(1 - e^{-1} \right) K_p \Delta u = 0.632 K_p \Delta u \quad \text{Eq. 3.5}$$

The time constant is therefore the amount of time needed for the output to reach $(1-\exp(-1))$ or 63.2% of the way to steady state conditions. The process time constant affects the speed of response.

In higher order process such as temperature the higher order time constants contribute to the dead time. τ and t_d can be found more accurately for higher order system (e.g. temperature) by using the ‘two points’ method. The 2 points refer to

- The time it takes the PV to reach 63.2% of its final value, $t_{0.632}$
- The time it takes the PV to reach 28.3% of its final value, $t_{0.283}$

For dead time, it refers to how much time passes before the process variable is effected by a change in the manipulated variable (MV). For single order process, the formula is,

$$\tau = 1.5 \times (t_{0.632} - t_{0.283}) \quad \text{Eq. 3.6}$$

For higher order process such as temperature,

$$t_d = (t_{0.632} - \tau) \quad \text{Eq. 2-7}$$

3.4.4 Ziegler Nichols Tuning Method

The Ziegler–Nichols tuning method is a heuristic method of tuning a PID controller. It was developed by John G. Ziegler and Nathaniel B. Nichols. It is performed by setting the I (integral) and D (derivative) gains to zero. The "P"

(proportional) gain, K_p is then increased (from zero) until it reaches the ultimate gain K_u at which the output of the control loop has stable and consistent oscillations. K_u and the oscillation period T_u are used to set the P, I, and D gains depending on the type of controller used:

The ultimate gain K_u is defined as $1/M$, where M = the amplitude ratio, $K_i = K_p/T_i$ and $K_d = K_p T_d$ which has the following transfer function relationship between error and controller output:

$$u(s) = K_p \left(1 + \frac{1}{T_i s} + T_d s \right) e(s) = K_p \left(\frac{T_d T_i s^2 + T_i s + 1}{T_i s} \right) e(s) \quad \text{Eq. 3.8}$$

Table 3.1 Ziegler Nichols Tuning Chart

K_c	τ_I	τ_D		
P control	$K_u/2$			
PI control	$K_u/2.2$	$P_u/1.2$		
PID control	$K_u/1.7$	$P_u/2$		P_u

The Ziegler–Nichols tuning creates a "quarter wave decay". This is an acceptable result for some purposes, but not optimal for all applications. This tuning rule is meant to give PID loops best disturbance rejection. It yields an aggressive gain and overshoot— some applications wish to instead minimize or eliminate overshoot, and for these this method is inappropriate.

In control theory, overshoot refers to an output exceeding its final, steady-state value. For a step input, the percentage overshoot (PO) is the maximum value minus the step value divided by the step value. In the case of the unit step, the overshoot is just the maximum value of the step response minus one. For second order systems, the percentage overshoot is a function of the damping ratio ζ and is given by,

$$PO = 100 \cdot e^{\left(\frac{-\zeta\pi}{\sqrt{1-\zeta^2}}\right)} \quad \text{Eq. 3.9}$$

The damping ratio can also be found by:-

$$\zeta = \frac{\left(\ln \frac{PO}{100}\right)^2}{\pi^2 + \left(\ln \frac{PO}{100}\right)^2} \quad \text{Eq. 3.10}$$

3.4.5 IMC Controller Tuning Method

The tuning parameters of an ideal PID controller based on IMC approach are given as: -

$$\bar{G}_c = \frac{(\tau_p s + 1)\left(1 + \frac{t_d}{2}s\right)}{K(\tau_f s + 1)} \quad \text{Eq. 3.11}$$

$$K_p = \frac{2\frac{\tau_p}{t_d} + 1}{K\left(2\frac{\tau_f}{t_d} + 1\right)} \quad \text{Eq. 3.12}$$

$$\tau_i = \frac{t_d}{2} + \tau_p \quad \text{Eq. 3.13}$$

$$\tau_d = \frac{\tau_p}{2\frac{\tau_p}{t_d} + 1} \quad \text{Eq. 3.14}$$

Since 1942, well over one hundred controller tuning methods have been developed, each trying to accomplish a certain objective or fill a specific niche. One of these methods is the Internal Model Control (IMC) tuning method sometimes called Lambda tuning. It offers a stable and robust alternative to other techniques, such as the

famous Ziegler-Nichols method, that usually aim for speed at the expense of stability. The IMC tuning method was developed for use on self-regulating processes. Most control loops, e.g., flow, temperature, pressure, speed, and composition, contain self-regulating processes. One obvious exception is a level control loop, which contains an integrating process. The IMC tuning method offers the following advantages are once tuned using the IMC tuning rules, the process variable will not overshoot its set point after a set point change.

The IMC tuning rules are much less sensitive to possible errors made when determining the process dead time through step tests. It is easy to under- or over-estimate dead time on lag dominant processes because of their relatively short dead times. Ziegler-Nichols and many other tuning rules can give poor results when the dead time is measured incorrectly. Besides, the tuning is very robust, meaning that the control loop will remain stable even if the process characteristics change substantially from the ones used for tuning. An IMC-tuned control loop absorbs disturbances better and passes less of them on to the rest of the process. This is a very attractive characteristic for using IMC tuning in highly interactive processes.

Unfortunately, the IMC tuning method has a drawback, too. It sets the controller's integral time equal to the process time constant. If a process has a very long time constant, the controller will consequently have a very long integral time. Long integral times make recovery from process disturbances very slow.

The IMC tuning rules are designed for use on a non-interactive controller algorithm. The tuning rules presented here are for a PI controller. Although IMC tuning rules have also been developed for PID controllers, there is theoretically no difference in the speed of response of the PI and PID tuning rules, so there is little sense in adding the complexity of derivative control. The effect of increasing each of the parameters of Proportional, Integral and Derivative is summarised in the table below.

3.4.6 Set Point Tracking

The principal objective of a feedback controller is typically either disturbance rejection or setpoint tracking. A controller designed to reject disturbances will take

action to force the process variable back toward the desired setpoint whenever a disturbance or load on the process causes a deviation. Set point tracking is one of the goals of process control where the controller adjusts to the desired value of a certain parameter (which is called the set point). It is desired that a controller changes smoothly and rapidly to set point changes. As study by (Thornhill et al., 2003) the manipulated variable can be exploited in controller performance assessment. Therefore, this can achieve by tuning the controller setting to optimize combination that gave best response curve with criteria such as no overshoot and short settling time. The definition of performance criteria is as below.

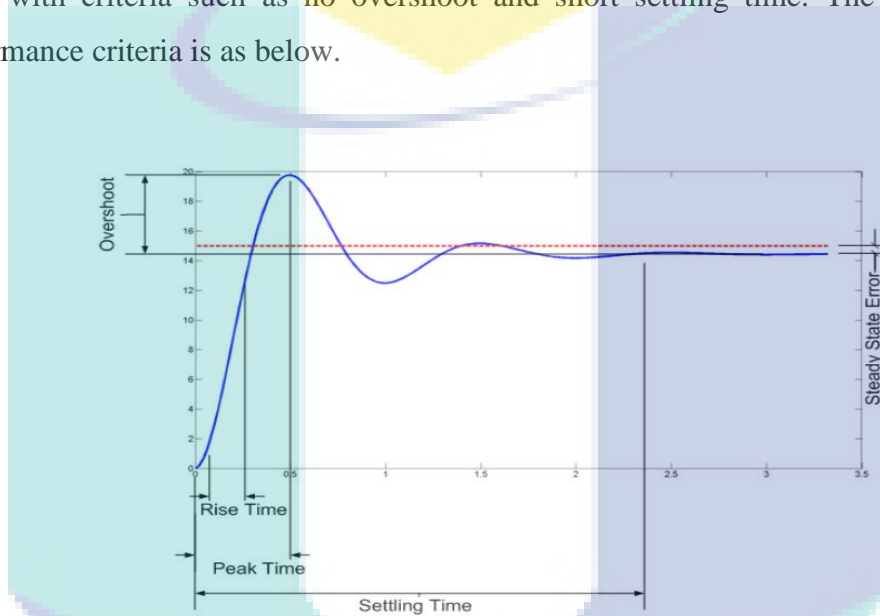


Figure 3-10 A typical step response for 2nd order system with complex poles and no finite zeros (Dr. David Corrigan, 2012)

$$\text{Overshoot \%} = 100 \times \exp \left\{ \frac{-\zeta\pi}{\sqrt{1-\zeta^2}} \right\} \text{ for } 0 < \zeta < 1 \quad \text{Eq. 3.15}$$

$$t_r \approx \frac{1}{\omega_n} (2.3\zeta^2 - 0.078\zeta + 1.12) \text{ for } 0 < \zeta < 1$$

$$t_p = \frac{\pi}{\omega_n \sqrt{1-\zeta^2}}$$

$$t_s \approx -\frac{\ln(\text{tolerance})}{\zeta\omega_n} \text{ for } \zeta \ll 1$$

- Rise Time (t_r) - the time taken for the output to go from 10% to 90% of the final value.
- Peak Time (t_p) - the time taken for the output to reach its maximum value.
- Overshoot - $(\text{max value} - \text{final value})/\text{final value} \times 100$.
- Settling Time (t_s)- The time taken for the signal to be bounded to within a tolerance of $x\%$ of the steady state value.
- Steady State Error e_{ss} - The difference between the input step value (dashed line) and the final value.

For a 2nd System it is possible to write down expressions for the parameters of the step response (for $0 < \zeta < 1$) as follows. Besides, the Integral of the absolute value of the error (IAE), integral of the time-weighted absolute, value of the error (ITAE) can be used as criteria to evaluate the performance of control structures (Wang and Ward, 2013) as well as weighted integral of squared error by (Nishikawa et al., 1984).

3.5 Potential of DWDC in Oleochemical via Inferential control

According to (Othman and Imran, 2015) In Malaysia, oleochemical fractionation of fatty acid typically imply series of distillation column but after studies performed it show feasible to apply DWC for fatty acid fractionation with significant reduction in operating cost while maintaining high product purity. Yet the performance need to be validated further prior industrial scale up either via pilot plant or via modelling and simulation based. According to (Niggemann and Fieg, 2012), the process model is compared with experimental data from production scale dividing wall and thus validated process model serves as a virtual plant and can be used to develop and test future process control structures. Moreover, knowledge gained help to increase the acceptance of dividing wall column in industry, particularly in this case oleochemical industry in Malaysia. According to (Illner and Othman, 2015) , a unique four column configuration model was used to represent the four sections in DWC. Using Aspen Plus as computer aided process-engineering tool, a rigorous steady state simulation performed. The results show that a more logical and realistic model for describing the

process operation was obtained, compared to a common more two or three-column approach.

3.6 Summary

Many studies have look on the advantages for apply DWC in industry in term of its operability either by pilot plant and simulation based. Besides, the parameters for smooth control of DWC is explored and identified their correlation with product composition. Various control strategies have been compare with respect to DWC application. However, the research gap is identified for implementation of DWC with inferential control in fatty acid fractionation is limited because of uncertainties in term of control performance. Therefore, the goals of this study is to evaluate the temperature controller loops which consist best measurement location and best controller setting use for inferential temperature control for product purity, as a part of effort to establish reliable inferential control strategy that can become primary of choice in operation for product purity control rather than conventional composition control.

3.7 Methodology

3.7.1 Specification of thermodynamics model

Based on (Illner and Othman, 2015) the modelling set-up by using 4 column to give equivalent DWC configuration as illustrated.

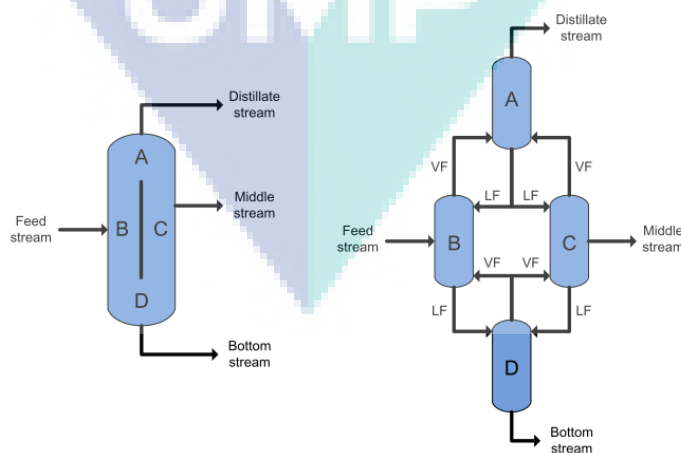


Figure 3-11 4 column to give equivalent DWC configuration (Othman et al., 2015)

The thermodynamic model SRK (Soave-Redlich-Kwong) is used for modelling work base on non-polarity of fatty acid (Illner and Othman, 2015). The simulated capacity of the column specifies by 6000 kg/hr of PKO-based fatty acid. The feed based on palm kernel oil (PKO) is specify as in Table 1 with triglyceride and unsaponifiable as residue which are represented by methyl oleate and n-hentriacontane.

Table 3.2 : PKO-based fatty acid compound and its mole fraction defined in Aspen Plus

Dynamics		
Component Name	Molecular formula	Mole Fraction
Water	H ₂ O	0.00035
Caproic acid	C ₆ H ₁₂ O ₂	0.0012
Caprylic acid	C ₈ H ₁₆ O ₂	0.033
Capric acid	C ₁₀ H ₂₀ O ₂	0.034
Lauric acid	C ₁₂ H ₂₄ O ₂	0.474
Myristic acid	C ₁₄ H ₂₈ O ₂	0.162
Palmitic acid	C ₁₆ H ₃₂ O ₂	0.079
Oleic acid	C ₁₈ H ₃₄ O ₂	0.1562
Linoleic acid	C ₁₈ H ₃₂ O ₂	0.026
Stearic acid	C ₁₈ H ₃₆ O ₂	0.0188
Triglyceride (methyl-oleate)	C ₁₉ H ₃₆ O ₂	0.0099
Unsaponifiables (n-hentriacontane)	C ₃₁ H ₆₄	0.00555

3.7.2 Steady state value of simulation

Table 3.3: Steady state values for simulation

Parameters	Value	Unit
Flow rate of feed stream	6000	kg/hr
Light cut purity (C ₆ -C ₁₀)	>99	% mol /% wt
Middle cut purity (C ₁₂ -C ₁₄)	>99	% mol /% wt
Heavy cut purity (C ₁₆ -C ₁₈ , TG, wax)	>99	% mol /% wt

3.7.3 Distillation Model

3.7.4 Model Design Basis

Table 3.4: Distillation model

	Section A	Section B	Section C	Section D
Stage Number	21	30	30	35
Diameter (meter)	2	2	2	2
Internal Structure	Packed type with packing factor 72 m-1			
Temperature	Reboiler temperature below 240°C			
Pressure	Top colum Pressure : 20 mbar Total column pressure drop : 12.25 mbar			
HETP	0.3	0.3	0.3	0.3
Feed Tray	N/A	10th stage	N/A	N/A
Middle Draw Off	N/A	N/A	3rd stage	N/A
Product purity	> 99.0 mole %	N/A	> 99.0 mole %	> 99.0 mole %

3.7.5 Assumption

To use the standard shortcut method, the component net flow model, and simplified model of a divided wall column, assume that:

- The relative volatility of components is constant;
- The vapor and liquid flows in each section of the divided wall column are constant;
- The pressure of the system is constant;
- The heat transfer across the dividing wall is neglected;
- The heat losses from the column walls are negligible;
- Vapor-liquid equilibrium is achieved on each stage;
- The heavy non-key component is assumed to go completely to the bottom of section II and the light non-key component is assumed to go completely to the top of section III;

3.7.6 Case Study

Case study of this paper is evaluating the inferential temperature controller loops which includes best measurement location and best controller setting use for product purity control using dividing wall distillation column for fatty acid fractionation.

3.7.7 Steady state and dynamic modelling

The process under study involves fractionation of oleochemical fatty acids which constitute of three carbon chains namely C10, C12 and C14 with boiling point of 270 °C, 299 °C and 326 °C respectively. To avoid product degradation, the column temperature was operated below 270 °C at pressure between 0.01 to 0.1 bar. The product purity for each streams were set to 99 mole%. Due to the polarity of the fatty acid as well as low operating pressure NRTL thermodynamic model and its variances can be used. In this work NRTL was chosen. For process flowsheeting, four RADFRAC model blocks were used for both steady state and dynamic modelling using Aspen Plus and Aspen Dynamics, respectively. Steady state model was used for sensitivity and correlation analysis to study the relationship between tray temperature and product composition as well as to determine the tray number to be inferred for controlling the product composition. Dynamic model was used for controllability analysis of the inferential control configuration. The control configuration used in the dynamic model was based on Othman (2019b). Othman conducted a controllability analysis of VDWC for oleochemical fatty acid fractionation using relative gain array (RGA) and singular value analysis (SVA). From his findings, it was found that DSV control configuration was the best 3x3 control pairing due to the low interaction between control loops. Therefore, DSV control configuration was adopted for this work. Figure 1 shows the DSV control configuration.

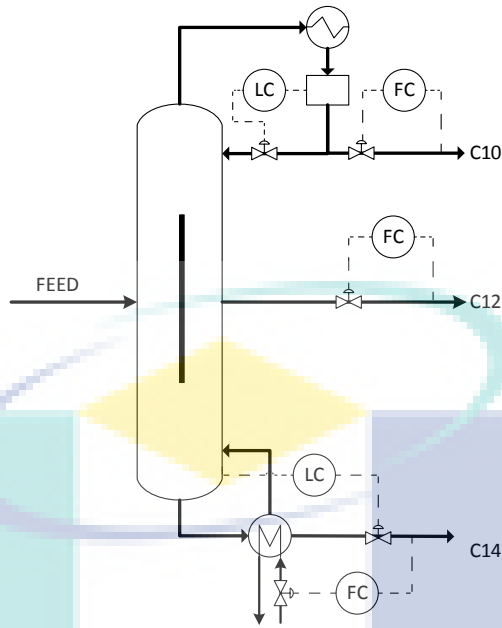


Figure 3-12 DSV control configuration

Table 3.5 Base case steady state condition.

Parameters	
Flowrate, kg/h	
- Feed	6048.41
- Distillate	292.836
- Side stream	4280.08
- Bottom	1427.24
Product composition, mol%	
- C10 at distillate	99
- C12 at side stream	99
- C13 at bottom	99
Reflux ratio	46.8
Vapor boilup ratio	1.17126

3.7.8 Tuning and closed loop response

The selected inferential variable from the previous step were added to developed DSV based control configuration by replacing the controlled variables from composition to the designated tray temperature. PID controller was adopted in this work. No measurement delay was included. Ziegler-Nichols (ZN) tuning method is a

heuristic method of tuning PID controller which is widely used for tuning. Meanwhile, Internal Model Control (IMC) tuning method is also adopted because of its ability to compensate disturbances and model uncertainty. However, ZN setting result in very good disturbance response for integrating processes, but are otherwise known to result in rather aggressive setting and also give poor performance (Skogestad, 2003). On the other hand, IMC setting it result in poor disturbance response for integrating processes but are robust and generally give very good responses for set point changes (Rivera et al., 1986). Therefore, in this work both ZN and IMC setting was compared before further fine-tuned. The controller setting was subjected to set point change for distillate, side and bottom product composition. The controller performance was tabled in term of their settling time and overshoot. Tuning with fastest settling time and small overshoot were selected before further fine-tuned.

3.7.9 Analysis

Settling time and overshoot were used as criteria to evaluate the performance of control.

3.8 Results and Discussions

Sensitivity analysis for distillate, middle and bottom sections are performed individually. For distillate section, Figure 1a gives temperature deviation for $\pm 5\%$ change in distillate flow. The temperature deviation for 5% increase of distillate flow was negative, while positive for 5% decrease. These curve shows that tray number two until seven were sensitive to changes for 5% increase in distillate flow while tray number six until ten sensitive to changes of 5% decrease distillate flow.

For middle section, Figure 1b gives temperature deviation between column tray number and change of side stream flow. The solid line is for -10% of distillate flow and dashed line is +10 % in distillate flow. However, the temperature deviation for both plus and minus 10 % in distillate flow give similar value, thus overlapped in the graph. These curve show two sensitive regions to changes plus and minus 10% side stream flow which refer to tray from number 4 until number 11 and from tray number 22 until 30. For bottom section, Figure 5 gives temperature deviation between column tray number and change of distillate flow as the selected manipulated variable. The solid line

is for -10% of distillate flow and dashed line is +5 % in distillate flow. As expected, the temperature deviation between column tray number and increase 5 % of distillate flow are positive, while they are negative for decrease 10% of distillate flow. These curve show 2 sensitive regions to changes minus 10% distillate flow which refer to tray from number 3 until number 8 and from tray number 17 until 31 while tray from number 27 until number 38 sensitive to changes of plus 5% distillate flow. Temperatures on the mentioned trays in the column are quickly affected by changes in distillate flow, so distillate flow with identified tray temperature is dynamically feasible. For the tray number other than this region, it would be a very poor inferential variable, because the sensor error and low magnitude noise would invalidate any correlation drawn from simulation. Besides, a small temperature deviation indicate that valve saturation can easily occur and operability region could be limited.

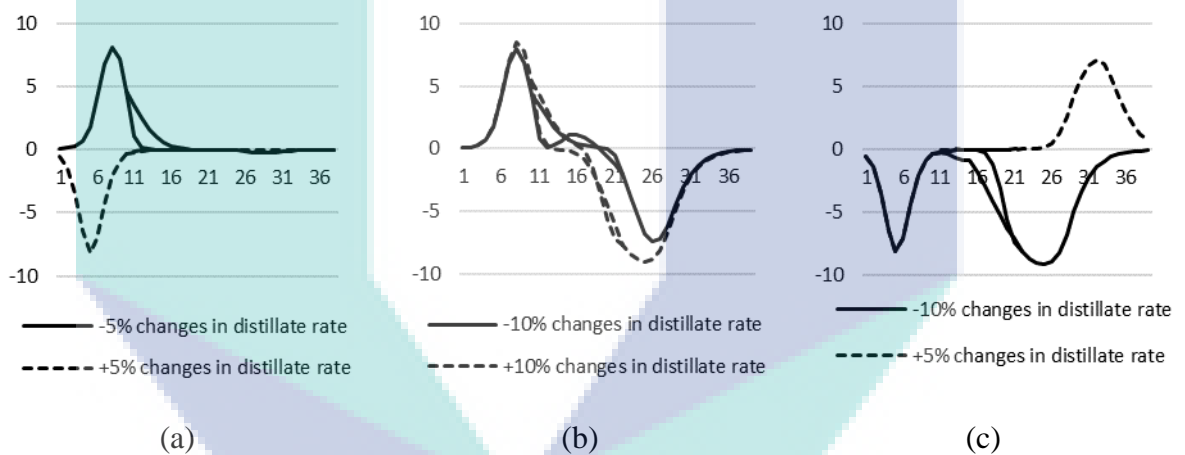


Figure 3.13: Tray temperature sensitivity analysis (a) $\pm 5\%$ changes in distillate flow (b) $\pm 10\%$ changes in side stream flow (c) $\pm 10\%$ changes in bottom flow

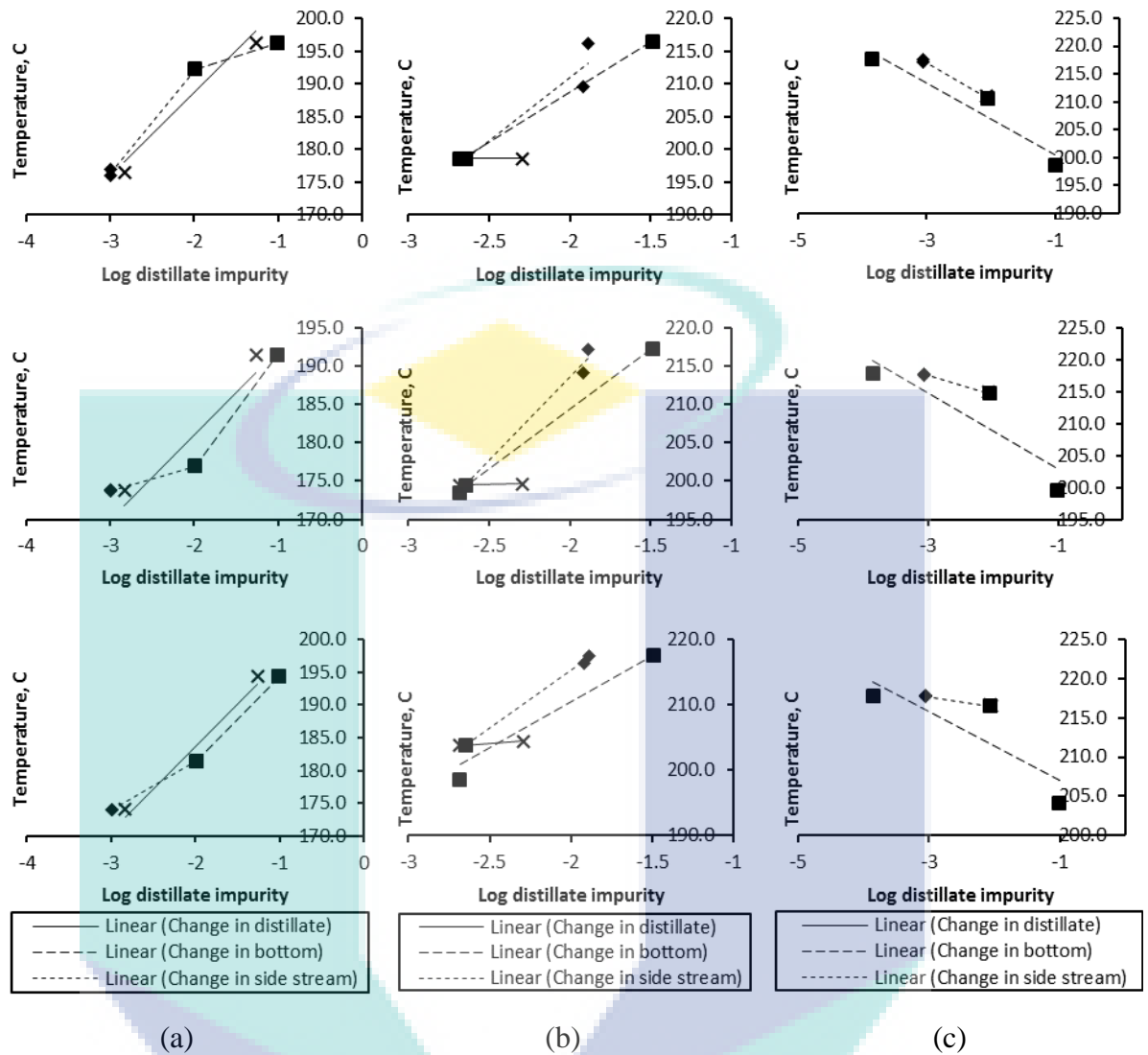


Figure 3-13 Correlation analysis slope for (a) C10 (b) C12 (c) C14

However, to further select the most sensitive tray to be used as inferred variable for distillate purity, middle purity and bottom purity, slope analysis on selected trays chosen from sensitive region is done individually. For distillate as shown in Figure 2, the location of the tray with the largest slope is Stage 6. Hence slope analysis suggest the use of Stage 6 for inferred variable for distillate purity (C10). For middle as shown in Figure 4, the steepest slope is happening at tray 8 with value of slope at 15.212. Hence tray 8 is satisfy to be chosen as tray to be inferred for C12 purity control at side draw for the column. For bottom as shown in Figure 6, it has steepest slope with the value of 9.1033. Hence slope analysis suggested that tray 25 will be a good inferential measurement location to be inferred for purity control as it has shown significant sensitivity towards changes made. It should be remembered that these are steady-state

result and tell us nothing about dynamics. For controller setting Ziegler-Nichols method is compared to IMC. PI and PID are also compared. The tuning setting for all controllers is shown in Table 1. The comparison in term of settling time and overshoot between PI vs PID and ZN vs IMC is shown for distillate, middle and bottom stream respectively below.

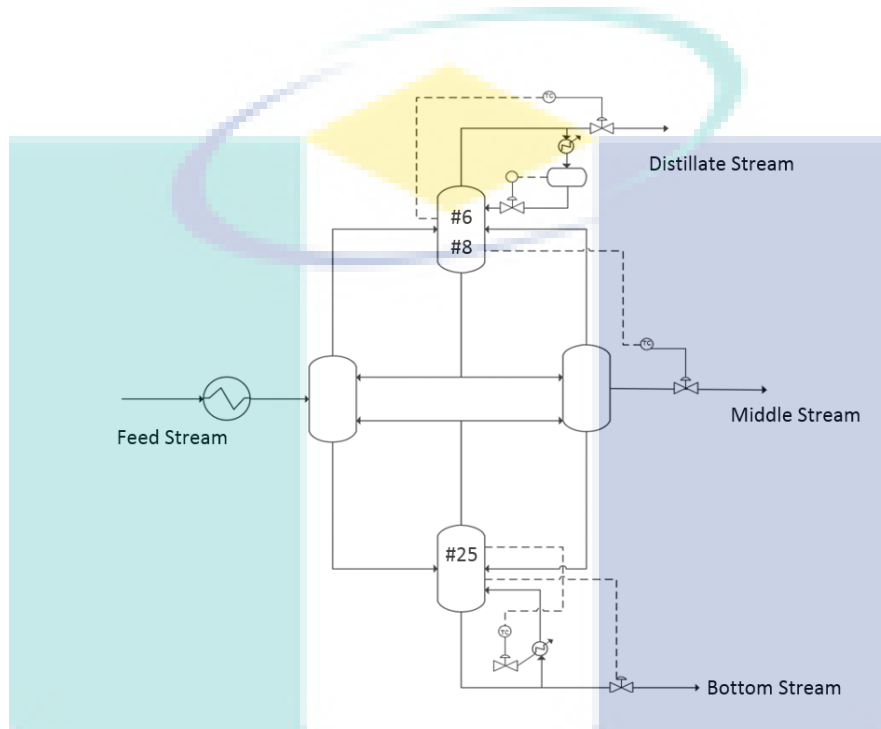


Figure 3-14 Inferential control using identified tray in DSV control configuration

Table 3.6 Control settings for the proposed inferential control in DSV control configuration with the comparison between Ziegler-Nichols and IMC method as well as comparison between PI and PID

Control Loop		Ziegler Nichols		IMC
		PI	*PID	PID
Distillate Stream, C10	Gain	86.87513	144.7919	2.350773
	Integral Time	35.47682	26.63425	375.1321
	Derivative Time	0	4.26148	5.251207
Result	Settling Time	6.09 hours	2.64 hours	3.42 hours
	Overshoot	0.45 %	5 %	0.45 %
Middle	Gain	5.742162	95.7021	22.86846

Stream, C12	Integral Time	7150.78564	2000	2171.743
	Derivative Time	0	858.9532	295.5417
Result	Settling Time	1983 hours	321.1 hours	359.1 hours
	Overshoot	0 %	0%	0 %
Bottom Stream, C14	Gain	0.921668	1.536113	0.393431
	Integral Time	60.83798	45.67416	39.798
Result	Derivative Time	0	7.307865	7.038206
	Settling Time	16.83 hours	11.62 hours	30.31 hours
Result	Overshoot	44.2 %	36.35 %	70.7 %

From the table 1, the comparison result shown Ziegler Nichlols tuning method is preferred compared to IMC as overall result in term of settling time and overshoot is compromise and meet the satisfactory target. By using Ziegler Nichlos, comparison between different controller setting between PI and PID shows PID showing best result in term of settling time and overshoot. Hence, this ZN PID setting is used as highlighted in the Table 1 for fine tuning prior testing under the condition of setpoint tracking. The graphical of comparison between PI vs PID of ZN and comparison between ZN vs IMC for distillate, middle and bottom stream as illustrated below. The fine tuning effort revealed positive result as shown in the Table 2 which has improved performance in term of settling time and overshoot from initial ZN PID setting selected before. The graphical response curve for all stream illustrated in Figure 14.

Table 3.7 After tuning of ZN PID controller for distillate, middle and bottom stream

Control Loop		Before Tuning	After tuning
Distillate Stream, C10	Gain	144.7919	6.6
	Integral Time	26.63425	600
	Derivative Time	4.26148	4.2618
Result	Settling Time	2.64 hours	1.82 hours
	Overshoot (< 5 %)	5 %	4.75%
Middle Stream, C12	Gain	95.7021	360
	Integral Time	2000	60

	Derivative Time	858.9532	5
Result	Settling Time	321.1 hours	3.77 hours
	Overshoot (< 5 %)	0%	0.28%
Bottom Stream, C14	Gain	1.536113	80
	Integral Time	45.67416	3
	Derivative Time	7.307865	7.03826
Result	Settling Time	11.62 hours	1.31 hours
	Overshoot (< 5 %)	36.35 %	3.05%

Set Point Tracking

Both distillate and bottom stream shown excellent result as indicate in Figure 15 and Figure 16 where tracking of PV upon the setpoint changes reveal satisfactory settling time and overshoot. Both increase and decrease of setpoint did not tell any poor setpoint tracking performance. Thus it tell us that the location of identified tray and the controller setting using ZN PID incorporating fine tune effort can be used to infer product purity.

However, for middle stream when the setpoint tracking is test for - 2°C, it demonstrated poor set point tracking as it unable to achieve the setpoint at 185°C from 187°C as shown in Figure15.. For reduction to187°C from 189°C, it require longer period of time to settle at setpoint. No concrete reason discovered in this study for under performance of C12.

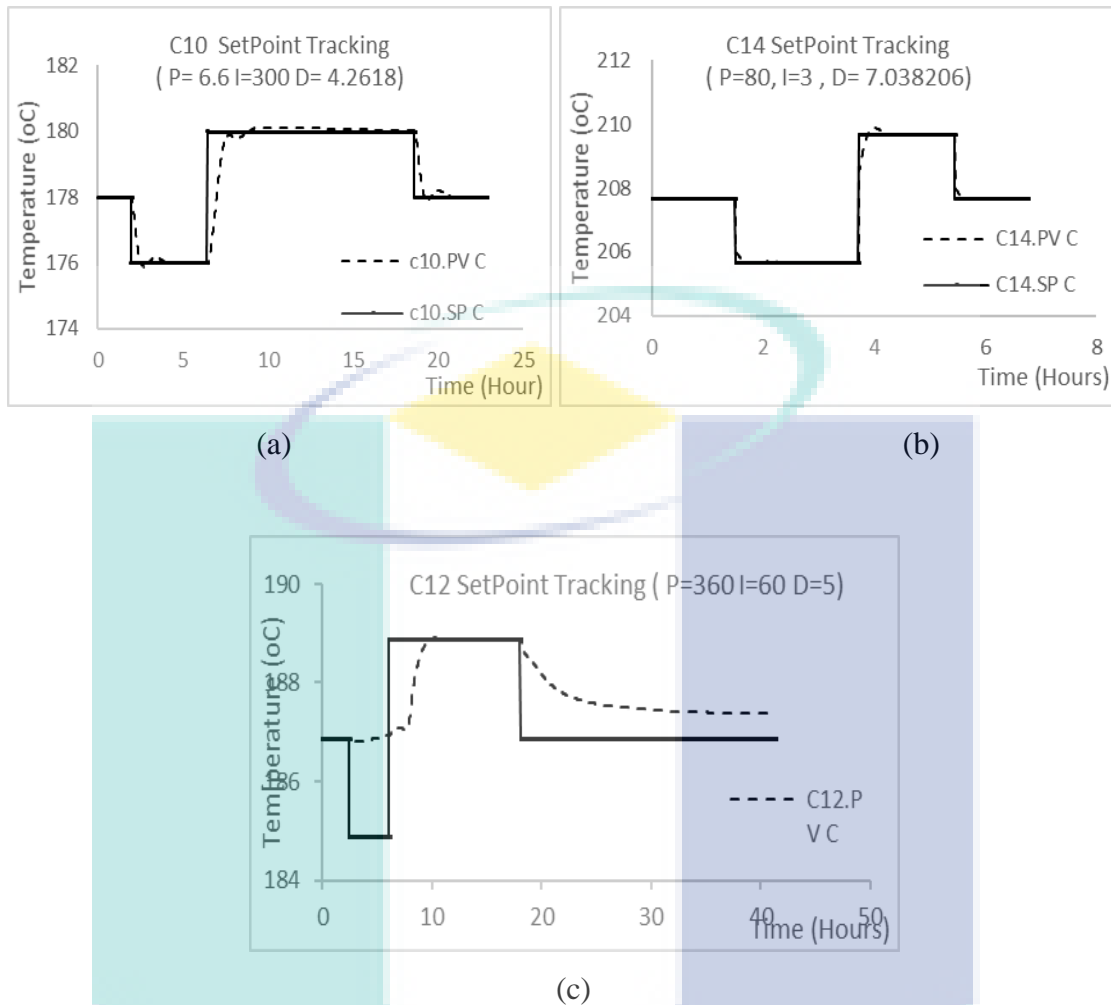


Figure 3-15 Setpoint tracking for + 2 oC (a) distillate stream (b) bottom stream and (c) middle stream

3.9 Conclusions

The sensitivity analysis was successfully determining the best tray temperature location for distillate at 6th tray, middle at 8th tray and bottom at 25th tray based on the most temperature deviation from base case. Correlation analysis has supported those trays number by give constant slope for distillate flowrate (D) change. It indicates strong relationship between tray temperature and composition of products to changes in distillate flowrate (D). The comparison from 2 tuning methods which are ZN and IMC and different controller setting of PI and PIC reveal ZN PID is preferred with satisfactory controller response in term of better settling time and minimum overshoot. The fine-tuned has improved the quality of controller response as per target thus give good setpoint tracking for distillate and bottom stream.

The potential application of this study is the setting of the controller can be used for actual controller DWC for oleochemical process to ensure stability for control specifically for inferential temperature control. However, to achieve a good inferential control performance, the use of composition estimator is important to solve collinearity as suggested by (Kresta et al, 1994). Therefore, rejection of disturbance such as feed change can be solved as it involved changes of temperature profile in the column which require a new set point of temperature inserted to maintain product purity. The future scope is in order to implement inferential control in real time it is recommend to incorporate estimator that best suit for oleochemical application with all results reveal from this study for a better performance of inferential control for product purity in DWC.

References

- °Astrom KJ. 2002. Control System Design.
- Adrian T, Schoenmakers H, Boll M. 2004. Model Predictive Control of Integrated Unit Operations: Control of a Divided Wall Column. *Chem. Eng. Process. Process Intensif.* **43**:347–355.
- Ansari RM, Tade MO. 2008. On-Line Inferential Models in Nonlinear Multivariate Control Applications. *Dev. Chem. Eng. Miner. Process.* **4**:71–83. <http://doi.wiley.com/10.1002/apj.5500040106>.
- Dejanović I, Matijašević L, Olujić Ž. 2010. Dividing wall column—A breakthrough towards sustainable distilling. *Chem. Eng. Process. Process Intensif.* **49**:559–580. <https://linkinghub.elsevier.com/retrieve/pii/S0255270110000814>.
- van Diggelen RC, Kiss AA, Heemink AW. 2010. Comparison of Control Strategies for Dividing-Wall Columns. *Ind. Eng. Chem. Res.* **49**:288–307. <https://pubs.acs.org/doi/10.1021/ie9010673>.
- Dohare RK, Singh K, Kumar R. 2015. Modeling and model predictive control of dividing wall column for separation of Benzene–Toluene–o-Xylene. *Syst. Sci. Control Eng.* **3**:142–153. <https://doi.org/10.1080/21642583.2014.996301>.
- Dr. David Corrigan. 2012. Characterising the Response of a Closed Loop System.
- Faessler P, Kolmetz K, Seang KW, Lee SH. 2007. Advanced fractionation technology for the oleochemical industry. *Asia-Pacific J. Chem. Eng.* **2**:315–321.

- <http://doi.wiley.com/10.1002/apj.25>.
- Hori ES, Skogestad S. 2007. Selection of Control Structure and Temperature Location for Two-Product Distillation Columns. *Chem. Eng. Res. Des.* **85**:293–306. <https://linkinghub.elsevier.com/retrieve/pii/S0263876207730514>.
- Ignat R, Woinaroschy A. 2011. Dynamic analysis and controllability of dividing-wall distillation columns using a four points control structure. *UPB Sci. Bull. Ser. B Chem. Mater. Sci.* **73**.
- Illner M, Othman MR bin. 2015. Modeling and Simulation of a Dividing Wall Column for Fractionation of Fatty Acid in Oleochemical Industries. *Perintis E-Journal* **5**.
- Kaibel B. 2014. Dividing-Wall Columns. In: . *Distillation*. Elsevier, pp. 183–199. <https://linkinghub.elsevier.com/retrieve/pii/B978012386878700005X>.
- Kano M, Showchaiya N, Hasebe S, Hashimoto I. 2001. Inferential control of distillation compositions: Selection of model and control configuration. *IFAC Proc. Vol.* **34**:347–352. <https://linkinghub.elsevier.com/retrieve/pii/S147466701733848X>.
- Ling H, Luyben WL. 2009. New Control Structure for Divided-Wall Columns. *Ind. Eng. Chem. Res.* **48**:6034–6049. <https://pubs.acs.org/doi/10.1021/ie801373b>.
- Lorenz H-M, Staak D, Grützner T, Repke J-U. 2018. Divided Wall Columns: Usefulness and Challenges. In: . *Chem. Eng. Trans.*, Vol. 69, p.
- Luan S, Huang K, Wu N. 2013. Operation of Dividing-Wall Columns. 1. A Simplified Temperature Difference Control Scheme. *Ind. Eng. Chem. Res.* **52**:2642–2660. <http://pubs.acs.org/doi/10.1021/ie3029762>.
- Luyben WL. 2005. Effect of Feed Composition on the Selection of Control Structures for High-Purity Binary Distillation. *Ind. Eng. Chem. Res.* **44**:7800–7813. <https://pubs.acs.org/doi/10.1021/ie0580147>.
- Luyben WL. 2006. Evaluation of criteria for selecting temperature control trays in distillation columns. *J. Process Control* **16**:115–134. <https://linkinghub.elsevier.com/retrieve/pii/S0959152405000569>.
- Marlin TE. 1995. *Process Control: Designing Processes and Control Systems for Dynamic Performance*. McGraw-Hill.
- Nguyen TD. 2015. Conceptual design, simulation and experimental validation of divided wall column: application for non-reactive and reactive mixture.
- Niggemann G, Fieg G. 2012. Validation of Dividing-Wall Columns Based on Experimental Data and Dynamic Simulations: Pilot-Plant and Production-Scale Columns. *Ind. Eng. Chem. Res.* **51**:931–943.

- <http://pubs.acs.org/doi/10.1021/ie201598q>.
- Nishikawa Y, Sannomiya N, Ohta T, Tanaka H. 1984. A method for auto-tuning of PID control parameters. *Automatica* **20**:321–332. <https://linkinghub.elsevier.com/retrieve/pii/0005109884900475>.
- Othman M, Imran U. 2015. Feasibility study of fractionating fatty acid using dividing wall column (DWC): Modelling Approach. In: .
- Othman MR, Amran UI, Rangaiah GP. 2015. Process Optimization of DWC for Fatty Acid Fractionation using Taguchi Methods of Experimental Design. *Chem. Eng. Trans.* **45**:925–930.
- Parrish JR, Brosilow CB. 1985. Inferential control applications. *Automatica* **21**:527–538. <https://linkinghub.elsevier.com/retrieve/pii/0005109885900020>.
- Pendergast J, Vickery D, Au-Yeung P, Anderson J. 2009. Consider dividing wall columns **72**:23–27.
- Petlyuk F.B., Platonv V.M., Slavinskii D.M. 1965. Thermodynamically optimal method for separating multicomponent mixtures **5**:555–561.
- Rewagad RR, Kiss AA. 2012. Dynamic optimization of a dividing-wall column using model predictive control. *Chem. Eng. Sci.* **68**:132–142. <https://linkinghub.elsevier.com/retrieve/pii/S0009250911006555>.
- Rivera DE, Morari M, Skogestad S. 1986. Internal model control: PID controller design. *Ind. Eng. Chem. Process Des. Dev.* **25**:252–265. <https://doi.org/10.1021/i200032a041>.
- Skogestad S. 2003. Simple analytic rules for model reduction and PID controller tuning. *J. Process Control* **13**:291–309. <https://linkinghub.elsevier.com/retrieve/pii/S0959152402000628>.
- Smith R. 2005. Chemical Process Design and Integration. Wiley, Chichester, UK, Hoboken, USA. 29 p.
- Stephanopoulos G. 1985. Chemical process control: An introduction to theory and practice.
- Thornhill N, Shah S, Huang B. 2003. Controller Performance Assessment in Set Point Tracking and Regulatory Control. *Thornhill, N.F. Shah, S.L. Huang, B. Controll. Perform. Assess. set point Track. Regul. Control. Int. J. Adapt. Control Signal Process.* **17** (7-9). pp. 709-727. ISSN 08906327 **17**.
- Wang J, Yu N, Chen M, Cong L, Sun L. 2018. Composition control and temperature inferential control of dividing wall column based on model predictive control and

- PI strategies. *Chinese J. Chem. Eng.* **26**:1087–1101.
<https://linkinghub.elsevier.com/retrieve/pii/S1004954117312041>.
- Wang W-J, Ward JD. 2013. Control of Three Types of Dividing Wall Columns. *Ind. Eng. Chem. Res.* **52**:17976–17995. <http://pubs.acs.org/doi/10.1021/ie401477p>.
- Yuan Y, Huang K, Chen H, Zhang L, Wang S. 2017. Asymmetrical temperature control of a BTX dividing-wall distillation column. *Chem. Eng. Res. Des.* **123**:84–98.
<https://linkinghub.elsevier.com/retrieve/pii/S0263876217302538>.

