EXPERIMENTAL AND THEORETICAL STUDIES OF A DIVING WALL COLUMN USED FOR THE SEPARATION OF HIGH PURITY PRODUCTS IN BIODIESEL PRODUCTION

MOHAMAD RIZZA BIN OTHMAN (LEADER)
NOORLISA BINTI HARUN (MEMBER)
WAN HANISAH BINTI W. IBRAHIM (MEMBER)
BADHRULHISHAM BIN ABDUL AZIZ (MEMBER)

E-mail: rizza@ump.edu.my

Research vote no.:
RDU130601

Faculty of Chemical & Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG

2017

ACKNOWLEDGEMENTS

I would like to express my gratitude to my team members for their support in completing this research project. My appreciation also to my undergraduate students for their effort. Without their dedicated work, this research may not be completed. Thank you again and may Allah swt bless our efforts.

Dr.-Ing. Mohamad Rizza bin Othman
Project leader
UMP Gambang
January 2016

ABSTRACT

Typically, fractionation of oleochemical products in Malaysia used conventional distillation columns (CDCs) and none so far imply DWC (dividing wall column) mainly due to the lack of understanding of its design, control and operation. In this research project, a systematic simulation based approach for the design of DWC have been proposed. A four column configuration model were developed to represent DWC internal sections and was succesfully simulated using Aspen Plus. Moreover, sensitivity analysis and optimization work shows that the model were found to be more accurate in representing the behaviour of DWC. Apart from that, our techno-economic and environmental feasibility study shows that compared to CDC, DWC saves up to 6% in capital cost and more than 30% savings for utility cost. In addition to the theoritical study, a DWC pilot plant have been built at Universiti Malaysia Pahang. The pilot plant was design on borosilicate glass components and operated under vacuum condition with temperature up to 200oC. The feed capacity is in the range of 5 - 7 kg/h for different type of oleochemical products. We also proposed a novel AHP-HAZOP for process hazard analysis and implement it to the pilot plant. With the realization of the pilot plant, future exciting work will be explore particularly on the operation and controllability. We believe such study integral work between theoritical and experimental scale is important to gain the operation experience for future industrial implementation.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1.1 Background

Distillation is one of the most common separation methods, as it is widely understood and used to a great extent in mixture separation techniques. Although the thermodynamic efficiency of distillation processes is low, the ease and confidence in operation makes it one of the most preferred separation methods Sangal et al. (2013). However, Sangal et al. (2013) also argue that distillation columns consume a substantial part of the entire energy requirement for chemical industry. Furthermore, the U.S. Department of Energy reported that there are more than 40,000 distillation columns in North America alone and these distillation columns are estimated to consume 40 % of the total energy to operate plants in the refining and bulk chemical industries Demirel (2013). Therefore, reducing its energy consumption could significantly achieve overall plant energy savings and increase the plant profitability. This motivates researchers to focus on developing and improving the efficiency of distillation processes. One of them is the dividing wall column (DWC). Over the years, DWC have attracted many researchers and have been successfully implemented in process industries especially for separating ternary mixtures. It is a promising energy saving alternative for separating multi-component mixtures Serra et al. (1999) from hydrocarbons, alcohols, aldehydes, ketones, acetals, amines and others Yildirim et al. (2011). In recent development, the application of DWCs have been extended to conduct azeotropic, extractive and reactive distillation Yildirim et al. (2011). Yildirim et al. (2011) predicted that future implementations of the DWC technology will be realized in developing countries with emerging markets rather than in countries with established distillation networks.

The DWC column configuration is thermodynamically equivalent to the Petyluk column configuration. Instead of an external pre-fractionator in the Petyluk design, a vertical wall is introduced in the middle part of the main distillation column of the DWC that create a feed pre-heater and draw off section in the middle section. When multicomponent mixtures are introduced into the column, the low boiling component goes overhead as distillate and the high boiling components goes out as bottom product, while the intermediate boiling component draws off in the side stream. The reflux is split between the two wall of the divided sections in the column and the vapor flow is split according to the pressure drop in both section Kiss and Ignat (2012) thus prevents the lateral mixing of liquid and vapor streams. Moreover, the wall also divides the space in the column prevents contamination of the side stream by the feed stream.

For separating ternary mixtures, DWC possesses a significant advantage in energy saving compared conventional two-column system. A single DWC for ternary mixture separation save a second column, whereby internals, reboiler, condenser, control and maintenance are significantly reduced. Julius Montz GmbH claimed that, by using DWC, investment cost can be cut by 20% to 30% and operating cost by around 25%. In particular, energy consumption can be reduced by up to 50%, which leads to lower carbon dioxide emission and smaller column diameter due to reduction in internal flows Barroso-Muñoz et al. (2011).

1.2 Research objectives and scopes

The main objective of this research is to perform integral analysis to comprehend the design and operation configuration of DWC in separating ternary mixtures into high purity products.

The specific objectives of this research are:-

- To design and fabricate an experimental DWC rig for separation of multicomponent mixtures.
- To study the effect of different column loadings, feed conditions, liquid distribution and dividing wall configuration to the hydrodynamic behaviour (pressure drops and operational limit), thermodynamic behaviour (temperature profile) and composition profile of the DWC.
- To formulate a model that describes the process characteristics of DWC i.e. heat transfer and liquid and vapour distribution.
- To validate the process model to experimental data and increasing its acceptance of the simulation results.

1.3 Overview of research contributions

1.3.1 Undergraduate research project report

1. Shaktiaaruvin a/l V Ravit. "3D design of heterogeneous catalytic biodiesel reactor and its plant using AVEVA PDMS". 2016. URP Thesis, FKKSA, UMP.
2. Nur Mahirah bt Katmin. "Conceptual 3D design of batch distillation column by using AVEVA PDMS". 2016. URP Thesis, FKKSA, UMP.

3. Nor Shaiful Irwan bin Shariman. "Simulation of dividing wall column for fractionation of fatty alcohol in oleochemical industries : A pilot plant case study". 2016. URP Thesis, FKKSA, UMP.

4. Widad Najihah binti Adanan. "Equation oriented modelling of dividing wall column using MOSAIC". 2015. URP Thesis, FKKSA, UMP.

5. Mohamad Firdaus bin Sahda. "Development of equilibrium model of vacuum distillation for benzene/oluene separation using MOSAIC software". 2015. URP Thesis, FKKSA, UMP.

6. Mazmajulianna binti Abdull Rahman. "Modelling of reactive distillation using MOSAIC: Comparison of equilibrium stage and non-equlibrium stage models". 2015. URP Thesis, FKKSA, UMP.

7. Marina binti Ismail. "Qualitative risk evaluation of dividing wall column using fault tree analysis (FTA) for integrating occupational safety and health". 2014. URP Thesis, FKKSA, UMP.

8. Rosshila binti Idris. "Quantitative risk evalaution of diviindg wall column using analytical hierarchy process (AHP)". 2014. URP Thesis, FKKSA, UMP.

9. Nurul Hafizah bin Sarimon. "Operator training simulator (OTS) of chemical process using Aspen simulation workbook (ASW): Example to dividing wall column for fatty acid fractionation". 2014. URP Thesis, FKKSA, UMP.

10. Chieng Tiew Hing. "Modelling of ractive distillation column using MOSAIC". 2013. URP Thesis, FKKSA, UMP.

1.3.2 Undergraduate plant design project report

1. Kong Zi ling, Noor Hafiza binti Abdullah, Nuredah Aisyah binti Nor Azman, Fatin Najihah binti Mohd Fauzi, Nur Ayuni binti Marsal. "Design 50 000

mt per year of oleochemical methyl ester from RBD palm kernel oil". 2015. Undergraduate Process Plant Design Report. FKKSA, UMP.

1.3.3 Conference and exhibition presentations

1. R. Idris, C.T. Hing, N. Harun & M.R. Othman. 2016. Development of Equation Oriented Modeling of Advanced Distillation Process Using MOSAIC: RD and DWC Case Study. 3rd International Conference of Chemical Engineering & Industrial Biotechnology (ICCEIB 2016). 28-30 November 2016. Melaka. Malaysia. ORAL
2. H. Abdul Aziz & M.R. Othman. 2016. Application of Analytic Hierarchy Process (AHP) in Prioritizing HAZOP Analysis for Pilot Plant. 3rd International Conference of Chemical Engineering & Industrial Biotechnology (ICCEIB 2016). 28-30 November 2016. Melaka. Malaysia. ORAL
3. M.R. Othman. 2016. Application of Dividing Wall Column for Improved Fractionation of Oleochemical Product from Modelling Work to Pilot Plant. National Seminar on Palm Oil Milling, Refining, Environment and Quality (POMREQ). 29-30 November 2016. The Royale Chulan Hotel, Kuala Lumpur, Malaysia. ORAL
4. R. Idris, C.T. Hing, N. Harun & M.R. Othman. 2016. Development of Equation Oriented Modeling of Advanced Distillation Process Using MOSAIC: RD and DWC Case Study. 3rd International Conference of Chemical Engineering & Industrial Biotechnology (ICCEIB 2016). 28-30 November 2016. Melaka. Malaysia. ORAL
5. H. Abdul Aziz & M.R. Othman. 2016. Application of Analytic Hierarchy Process (AHP) in Prioritizing HAZOP Analysis for Pilot Plant. 3rd International Conference of Chemical Engineering & Industrial Biotechnology (ICCEIB 2016). 28-30 November 2016. Melaka. Malaysia. ORAL

6. M.R. Othman & U.I. Amran. 2015.Feasibility Study of Fractionating Fatty Acid using Dividing Wall Column (DWC): Modelling Approach. 4th Conference on Emerging Energy and Process Technology (CONCEPT) 2015. 15-16 December 2015. A Famosa Resort. Melaka. ORAL

7. M.R. Othman, G.P. Rangaiah. 2015. Process Optimization of a Dividing-Wall Column (DWC) for Fatty Acid Fractionation using Taguchi Methods of Experimental Design. 18th Conference Process Integration, Modelling and Optimisation for Energy Saving and Pollution (PRES). 23-27 August 2015. Kuching, Sarawak, Malaysia. ORAL

8. R Idris, M H Hassim, W H W Ibrahim, M R Othman. 2015. Systematic Prioritization of HAZOP Analysis Using Analytic Hierarchy Process (AHP). SOMCHE 2015. 21-22 Oct 2015. Kuala Lumpur. ORAL.

9. M. Illner & M.R. Othman. 2015. " Simulation and modelling of a DWC for separation of fatty acid in oleochemical industries". SOMCHE 14. 29-30 October 2014. ORAL.

   item C.T. Hing & M.R. Othman. 2014. "Web-based Modelling of Chemical Processes Using MOSAIC : A Reactive Distillation Case Study". MUCET 2014. 10-11 Nov 2014. Melaka, Malaysia. ORAL

1.3.4 Publications

1. H. Abdul Aziz & M.R. Othman. 2017. Application of Analytic Hierarchy Process (AHP) in Prioritizing HAZOP Analysis for Pilot Plant. Chemical Engineering Research Bulletin. 19(2017), pg. 87-95.

2. R. Idris, C.T. Hing, N. Harun & M.R. Othman. 2017. Development of Equation Oriented Modeling of Advanced Distillation Process Using MOSAIC: RD and DWC Case Study. Australian Journal of Basic and Applied Sciences. 11(113), pg. 30-42.

3. Rosshila Idris, Mimi Haryani Hassim, Wan Hanisah Wan Ibrahim, Mohamad Rizza Othman. 2015. Prioritizing HAZOP analysis using analytic hierarchy process (AHP). Clean Technologies and Environmental Policy. Vol. 18(5), pp 13451360. IF (1.93)
4. Mohamad Rizza Othman, Umarul Imran Amran & Gade Pandu Rangaiah. 2015. Process Optimization of DWC for Fatty Acid Fractionation using Taguchi Methods of Experimental Design Chemical Engineering Transactions. Vol. 45, pg. 925-930. IF (1.03)
5. M.R. Othman & M. Illner. 2015. ”Simulation and modelling of a DWC for separation of fatty acid in oleochemical industries”. PERINTIS Journal. Vol. 5., No. 1, pp. 34 44

1.3.5 Keynote speaker invitation

1. M.R. Othman. 2016. Application of DWC for Improved Oleochemical Fractionation: From Comceptual Design to Pilot Plant Validation. International Palm Oil Congress (PIPOC). 14-16 November 2017. Kuala Lumpur Convention Centre in Kuala Lumpur, Malaysia.
2. M.R. Othman. 2016. Application of Dividing Wall Column for Improved Fractionation of Oleochemical Product from Modelling Work to Pilot Plant. National Seminar on Palm Oil Milling, Refining, Environment and Quality (POMREQ). 29-30 November 2016. The Royale Chulan Hotel, Kuala Lumpur, Malaysia.

1.3.6 Award

1. BEST ORAL PRESENTER. M.R. Othman & U.I. Amran. 2015.Feasibility Study of Fractionating Fatty Acid using Dividing Wall Column (DWC): Modelling Approach. 4th Conference on Emerging Energy and Process Technology

(CONCEPT) 2015. 15-16 December 2015. A Famosa Resort. Melaka.

1.3.7 Industrial collaboration

1. Julius Montz GmbH, Germany - provide column design and drawing and technical consultation.
2. SULZER AG, Switzerland - provide structured packing design consultation and material.
3. National Instruments, USA - provide technical assistance for control system.

1.3.8 Industrial presentation

1. FPG Oleochemicals Sdn. Bhd., Lot 3831, Kuantan Port, Bukit Tanjung Gelang, 26080 Kuantan, Pahang, Malaysia
2. IOI Pan Century Sdn. Bhd., Lot 231, Pasir Gudang, Malaysia, Jalan Pekeliling, Kawasan Perindustrian Pasir Gudang, 81700 Pasir Gudang, Johor, Malaysia

1.4 Report layout

In this report an executive summary of selected main contributions will be presented. Overall this reports contains 7 chapters including introduction. Chapter 2 present an introduction to oleochemical fractionation and application of DWC in the oleochemical industry. Chapter 3 focuses on the modelling and simulation of DWC using Aspen Plus. A unique four column configuration were used instead of conventional two or three column configuration. Temperature and composition profile were also studied. Detail of this study have been published as in number 5 of the publication lists. Chapter 4 discuss a topic related to DWC sensitivity analysis and optimization based on experimental design application while Chapter

5 presents on a new methodology of performing process safety analysis (PHA) using a combination of HAZOP analysis and analytic hierarchy process (PHA). Chapter 6 presents on the feasibility of applying DWC compared to typical DC. Also included in the design and development of DWC pilot plant. Chapter 7 will conclude the findings and recommendations for future work.

1.5 References

1. Barroso-Munoz, F.O., Hernandez, S., Segovia-Hernandez, J.G., Hernandez-Escoto, H., Rico-Ramrez, V., Chavez, R.-H., 2011. Implementation and operation of a dividing-wall distillation column. Chemical Engineering & Technology 34(5), 746750.

2. Dejanovic, I., Matijasevic, L., Olujic, Z., 2010. Dividing wall columna breakthrough towards sustainable distilling. Chemical Engineering and Processing: Process Intensication 49 (6), 559580.

3. Demirel, Y., 2013. Sustainable operations for distillation columns. Chemical Engineering & Process Techniques 1005, 115.

4. Kiss, A.A., Ignat, R.M., 2012. Enhanced methanol recovery and glycerol separation in biodiesel productiondwc makes it happen. Applied Energy 99, 146153.

5. Sangal, V. K., Kumar, V., Mishra, M. I., 2013. Optimization of a divided wall column for the separation of c4-c6 normal paran mixture using box-behnken design. Chemical Industry and Chemical Engineering Quarterly 19 (1), 107119.

6. Serra, M., Espuna, A., Puigjaner, L., 1999. Control and optimization of the divided wall column. Chemical Engineering and Processing: Process Intensication 38(4), 549562.

7. Yildirim, O., Kiss, A. A., Kenig, E. Y., 2011. Dividing wall columns in chemical process industry: a review on current activities. Separation and Purification Technology 80 (3), 403417.

CHAPTER 2

DWC IN THE OLEOCHEMICAL INDUSTRY

2.1 Oleochemical

Oleochemicals deal with the physico-chemical transformation of fats and oils from animals and vegetables. However, they also include those derivatives derived from the subsequent modification of carboxylic acid group of fatty acids by chemical or biological means, and other compounds obtained from further reactions of these derivatives (Lim et al., 2010). Oleochemicals are often categorised into basic oleochemicals such as fatty acids, fatty methyl esters, fatty alcohols, fatty amines and glycerol, and their further downstream derivatives obtained from further chemical modifications of these basic oleochemicals. First used in the fabrication of soaps, oleochemical is now part of our daily lives where it is found in a wide variety of sectors: food, cosmetics, pharmaceutical and industrial.

The raw materials of the oleochemical sector are:

- Vegetable oils such as rapeseed oil as well as those derived from soybean,sunflower, safflower, corn, peanut, palm, coco, castor, etc.
- Fats and oils from animals like rendered fats, tallow, lard, fish oil, etc.
- Recycled fats and oils like grease from restaurants, used frying oils, trap greases, etc.

For many of the well-known applications until the beginning of the 20th century, oleochemical had been subjected to the powerful competition of the petrochemical sector. However, for some uses, the physico-chemical properties of oleochemicals still offer some unsurpassed advantages and performances. Nowadays, the uses of oleochemicals are in constant growth considering the strong environmental tendencies and legislations that are being brought to the forefront. A product of non-fossil, renewable and biodegradable raw materials, oleochemicals, often called "Bioproducts", are already fully developed and in use all over the world. The reasons of oleochemical can replace petrochemicals in all their applications First, oleochemicals are derived from renewable resources, as compared to petrochemicals which are obtained from exhaustible or non-renewable petroleum. Secondly, products derived from oleochemicals are more readily biodegradable and hence do not pose a threat to the environment. Thirdly, products derived from petroleum sources use more energy and cause higher emissions of such pollutants as NOx, SO2, CO and hydrocarbons (Ting, 2001).

2.2 Background

Distillation is one of the most common separation methods, as it is widely understood and used to a great extent in mixture separation techniques. Although the thermodynamic efficiency of distillation processes is low, the ease and confidence in operation makes it one of the most preferred separation methods Sangal et al. (2013). However, Sangal et al. (2013) also argue that distillation columns consume a substantial part of the entire energy requirement for chemical industry. Furthermore, the U.S. Department of Energy reported that there are more than 40,000 distillation columns in North America alone and these distillation columns are estimated to consume 40 % of the total energy to operate plants in the refining and bulk chemical industries Demirel (2013). Therefore, reducing its energy consumption could significantly achieve overall plant energy savings and increase the plant profitability. This motivates researchers to focus on developing and improving the efficiency of distillation processes. One of them is the dividing wall column (DWC). Over the years, DWC have attracted many researchers and have been successfully implemented in process industries especially for separating ternary mixtures. It is a promising energy saving alternative for separating multi-component mixtures Serra et al. (1999) from hydrocarbons, alcohols, aldehydes, ketones, acetals, amines and others Yildirim et al. (2011). In recent development, the application of DWCs have been extended to conduct azeotropic, extractive and reactive distillation Yildirim et al. (2011). Yildirim et al. (2011) predicted that future implementations of the DWC technology will be realized in developing countries with emerging markets rather than in countries with established distillation networks.

The DWC column configuration is thermodynamically equivalent to the Petyluk column configuration. Instead of an external pre-fractionator in the Petyluk design, a vertical wall is introduced in the middle part of the main distillation column of the DWC that create a feed pre-heater and draw off section in the middle section. When multicomponent mixtures are introduced into the column, the low boiling component goes overhead as distillate and the high boiling components goes out as bottom product, while the intermediate boiling component draws off in the side stream. The reflux is split between the two wall of the divided sections in the column and the vapor flow is split according to the pressure drop in both section Kiss and Ignat (2012) thus prevents the lateral mixing of liquid and vapor streams. Moreover, the wall also divides the space in the column prevents contamination of the side stream by the feed stream.

For separating ternary mixtures, DWC possesses a significant advantage in energy saving compared conventional two-column system. A single DWC for ternary mixture separation save a second column, whereby internals, reboiler, condenser, control and maintenance are significantly reduced. Julius Montz GmbH claimed that, by using DWC, investment cost can be cut by 20% to 30% and operating cost by around 25%. In particular, energy consumption can be reduced by up to 50%, which leads to lower carbon dioxide emission and smaller column diameter due to reduction in internal flows Barroso-Muñoz et al. (2011).

2.3 DWC in Malaysia

In 2013, Malaysia crude palm oil (CPO) production recorded an increase of 2.3% to 19.22 million tons against 18.79 million tons in 2012. The oleochemical industry in Malaysia is now one of the largest in the world, with capacity representing about 20% of the world's capacity. There are currently 18 oleochemical companies in Malaysia, as shown in Table 2.1, and they have been exporting products to more than 100 countries. From our survey (summarized in Table 1), it is found that all oleochemical plants employ a series of typical distillation columns for fractionation of its oleochemical products. Considering DWC benefits and implementations in the US and Europe, especially for chemical industries, application of DWC to oleochemical industries in Malaysia offers huge opportunity. Possible reasons for not utilizing DWC in oleochemical industries are relatively more complexity and lack of a systematic design approach Dejanović et al. (2010). Nevertheless, with the advancement of process simulators, modelling, design and control of DWC have been extensively studied in the open literature. A sequential-modular modelling is often used for simulating DWC. Note that DWC block is not currently available in commercial process simulators. Hence, several distillation blocks have to be arranged to represent the unique structure of DWC.

In this paper, we propose DWC application for fatty acid fractionation in oleochemical industries, and simulate it using four-column configuration model, representative of the internal sections of DWC. A step-by-step procedure to process model development from short cut to rigorous models is presented. Aspen Plus is used for rigorous steady state simulation of the model. Finally, economic and CO2 emission comparison of DWC with conventional columns, and hydrodynamics of DWC are discussed for industrial application.

Table 2.1

Application of DC and DWC in Malaysian oleochemical industry.

Company	FAC	Gly	FAL	FES	Col. type
					DC / DWC
1. AcidChem International Sdn Bhd	✓	✓			DC
2. Emery Oleochemical Sdn Bhd	✓	✓	✓	✓	DC
3. Natural Oleochemical sdn Bhd	✓	✓			DC
4. Global Biodiesel Sdn Bhd	✓				DC
5. Inno-Wangsa Oils & Fats Sdn Bhd	✓				DC
6. Kwantas Oil Sdn Bhd	✓	✓			DC
7. Nexsol (Malaysia) Sdn Bhd	✓	✓			DC
8. Timuran Enterprise Sdn Bhd	✓	✓			DC
9. Pacific Oleochemical Sdn Bhd		✓			DC
10. IFFCO Malaysia Sdn Bhd		✓			DC
11. Palm Oleo Sdn Bhd		✓			DC
12. Southern Acids Sdn Bhd		✓			DC
13. Carotino Sdn Bhd		✓		✓	DC
14. KL-Kepong Oleomas Sdn Bhd		✓	✓	✓	DC
15. Loreno Sdn Bhd		✓			DC
16. Fatty Chemical (M) sdn Bhd			✓		DC
17. FPG Oleochemical Sdn. Bhd.	✓	✓			DC

*FAC=fatty acid, Gly=glycerol, FAL=fatty alcohol, FES=fatty ester

2.3.1 Fatty acid fractionation

Fractionation of the fatty acid cuts from refined-deodorized-bleached (RBD) palm kernel oil (PKO) is by thermal separation using distillation columns. The main fatty acid cuts from RBD PKO that need to be fractionated are: C6-C10 (precut, PC), C12 (light cut, LC), C14 (middle cut, MC) and C16-C18 (heavy cut, HC). Table 2.2 shows the boiling temperature of each carbon chain. Typically, four distillation columns are needed to fractionate the fatty acid cuts as shown in Figure 3.1a. Because of the distinctive boiling point differences, it is interesting to analyze the possibility of fractionating the cuts using DWC. An intensified process is possible using only two DWCs in series, as shown in Figure 3.1b, to produce four cuts of fatty acids. Note that the operating temperature must be lower than 270 °C to avoid product degradation, and therefore the columns must be operated under vacuum. The feed used in this work is based on information obtained from our industrial partner (please refer to Table 3.1).

Table 2.2
Fatty acids physical properties.

Component name	MW	T_{BP}, C	Dipole moment, debye
Caproic acid	116.16	205.7	1.57092
Caprylic acid	144.214	239.7	1.69983
Capric acid	172.268	270.0	1.67884
Lauric acid	200.321	298.7	1.63987
Myristic acid	228.375	326.2	1.67884
Palmitic acid	256.429	350.0	1.7388
Linoleic acid	280.451	354.9	1.21716
Oleic acid	282.467	359.9	1.43901
Stearic acid	284.483	374.0	1.66985

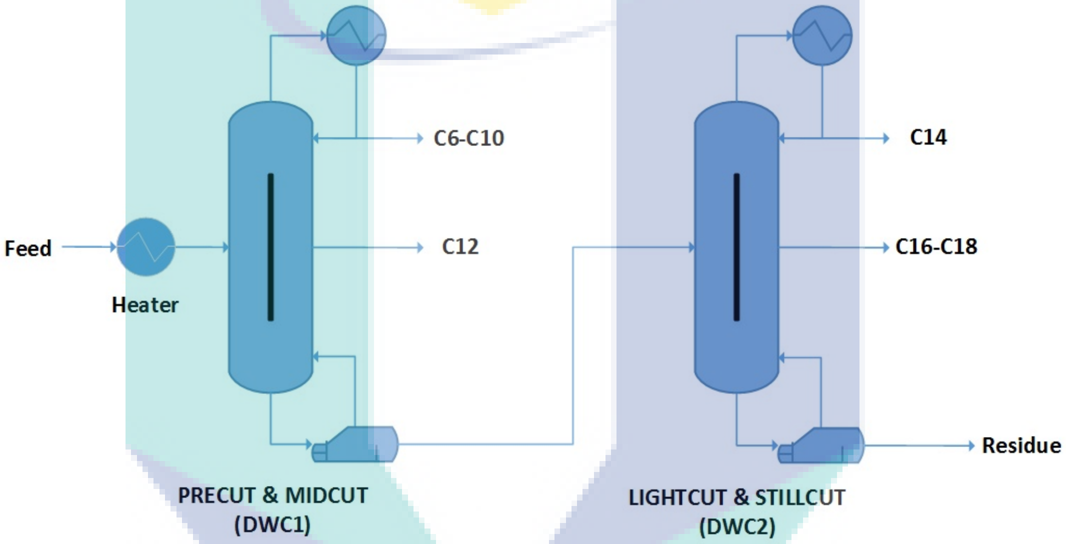


Figure 2.1. Flowsheet configuration for four product fractionation using yypical distillation column

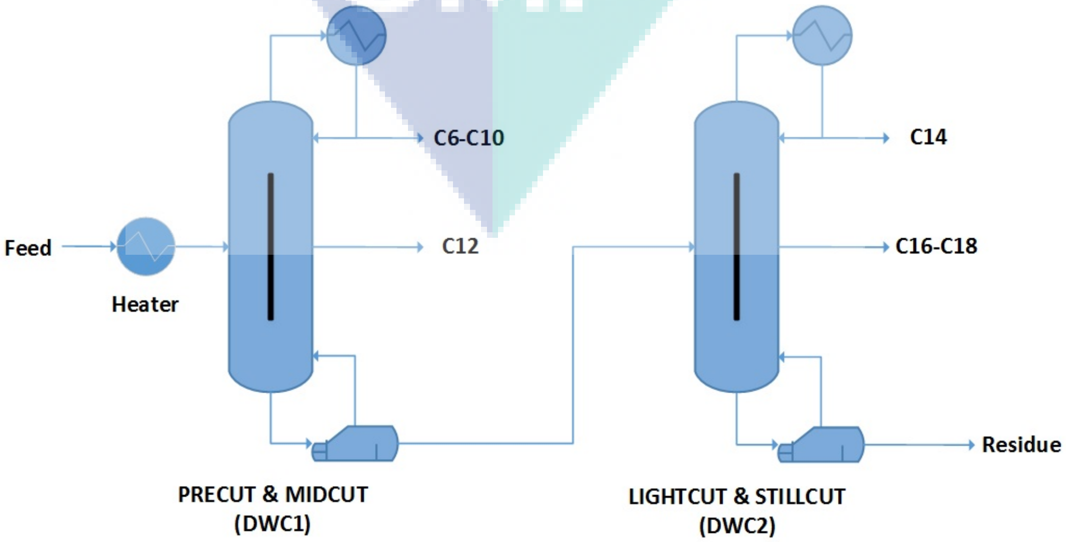


Figure 2.2. Flowsheet configuration for four product fractionation using DWC

Table 2.3

Fatty acids feed information.

Feed flowrate; kg/h	9167	
Feed temperature, C	30	
Feed pressure, bar	1	
Component name	Alias	Mole fraction
Water	H_2O	0.00035
Caproic acid	$C_6H_{12}O_2$	0.0012
Caprylic acid	$C_8H_{16}O_2$	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
Triglycerides (TAG-POLN)	$C_{55}H_{98}O_6$	0.0099

CHAPTER 3

MODELING AND SIMULATION OF A DIVIDING WALL COLUMN FOR FRACTIONATION OF FATTY ACID IN OLEOCHEMICAL INDUSTRIES

This chapter is dedicated to the effort of modelling and simulation of dividing wall column. The research paper have been published in PERINTIS E-Journal in 2015. The paper was authored by an internship student from Technical University of Berlin, Germany named Markus Illner. Below are the authors:-

- Markus Illner, Chair of Process Dynamics & Operation, Technische Universitt Berlin, 10623 Berlin, Germany
- Mohamad Rizza bin Othman, Process Systems Engineering Research Group, Universiti Malaysia Pahang, 26300 Gambang, Malaysia

3.1 Abstract

The dividing wall column (DWC) is particularly useful for separating multicomponent mixtures into their pure fractions within one column shell and has attracted many researchers and companies for various applications. In the oleochemical industries, it is observed, that the fractionation of fatty acid commonly uses conventional two stage distillation columns. In this research, DWC was studied for

a more efficient fractionation of fatty acid mixtures. 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 was 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. In addition, some technical aspects were also highlighted to comprehend the design and operational configuration of DWC.

3.2 Introduction

Distillation is one of the most common separation methods, as it is widely understood and used to a great extent in mixture separation techniques. Although the thermodynamic efficiency of distillation processes is low, the ease and confidence in operation makes it one of the most preferred separation methods [1]. However, Sangal et al. [1] argue that distillation columns consume a substantial part of the entire energy requirement for chemical industry. This motivates researchers to focus on developing and improving the efficiency of distillation processes. The separation of multicomponent mixture has typically been done by sequential distillation columns [2]. This configuration has basic drawbacks related to operation and capital cost. Over the years, researchers proposed several designs to improve the efficiency of distillation processes such as the Petlyuk column and divided wall column (DWC).

DWC are especially advantageous for separating ternary mixtures [3] into pure fractions, especially for medium boiling components. The column configuration is thermodynamically equivalent to the Petyluk column configuration, consisting of a prefractionator and a main distillation column. Instead of an external prefractionator in the Petyluk design, a vertical wall is introduced in the middle part of the main distillation column of the DWC. This creates a feed pre-heater and draw off section in the middle section. When multicomponent mixtures are introduced into the column, the low boiling component goes overhead as distillate and the high boiling components goes out as bottom product, while the intermediate boiling component draws off in the side stream. The reflux is split between the two wall of the divided sections in the column and the vapour flow is split according to the pressure drop in both section [4]. Such configurations save a second column, where compartments like the column shell, internals, evaporator, condenser, control and maintenance are significantly reduced. Julius Montz GmbH claimed that by using DWC, the investment costs can be cut by 20% - 30% and operating costs by around 25%. In detail, the energy consumption can be reduced up to 50% [5]. This leads to lower carbon dioxide emission and smaller column diameter due to reduction in internal flows [5]. Because of these advantages, DWCs have attracted many researchers and companies for various applications.

It is observed that the most common approach to fractionate fatty acid mixtures in oleochemical industry is through sequential separation using two distillation columns to separate the light cut (C8-C10), middle cut (C12-C14) and heavy cut (C16-C18) components. Because of distinctive boiling point differences, it is interesting to analyse the possibility of fractionating the three cuts using DWC. In doing this, we proposed a four column configuration model, representative of the DWC in which Aspen Plus is used as computer aided process engineering tools for rigorous steady state simulation of the model. In addition to that, a review of some technical aspects will also be highlighted to comprehend the design and operation configuration of DWC for fatty acid fractionation and significantly open the possibility for industrial implementation.

3.3 Modelling and simulation approach

Modelling and simulation of DWC has been studied in the open literature using commercial simulators such as Aspen HYSYS and Aspen Plus. However, most often two [4, 6] or three columns [7] configurations were investigated. Due to simulation convergence issues and resulting simplified models most researchers avoid using four column configurations. As illustrated in Fig. 3.1(left), typical DWC configuration can be divided into four sections. Therefore, attempting to represent the real process it is logical to use a four column configuration as represented in Fig. 3.1(right). Such an arrangement could describe the DWC operation in a more realistic and logical approach for the modeling work. Moreover, all main streams and straws could be taken into account even under rising the degree of freedoms, as to be found for a real plant [8].

In this representation (see Figure 3.1(right)), the feed stream is introduced to a pre-heat section (section B) which is equivalent to the pre-fractionator of the Petyluk distillation column. It has two output streams. The vapor output stream is introduced to the rectifying column (section A), while the liquid output stream is introduced to the stripping column (section D). The rectifying section (section A) has two input and output streams. The input streams are the vapor streams from the pre-heat (section B) and middle column (section C). While the output streams are the vapor distillate stream and liquid bottom stream. The bottom stream is then split into reflux streams for section B and section C. A split factor could be applied here to control the reflux and rectification behavior of the regarded sections. Section C has two input and three output streams. The input streams are the liquid reflux from section A and vapor boil up from section D. The output streams are the distillate vapor stream, middle product stream and bottom liquid stream. Section D has two input stream and output stream. Input to this column are the liquid refluxes from section B and C. Whereas the output stream is the bottom liquid stream and the vapor boil up, which then split to section B and C according to the fluid dynamic conditions in the sections, mainly pressure drop.

3.3.1 Modelling set-up

Basic steps to process modeling and simulations, using process simulators, include defining chemical components, selecting thermodynamic models and methods, designing the process flowsheet by choosing proper operating units, determining

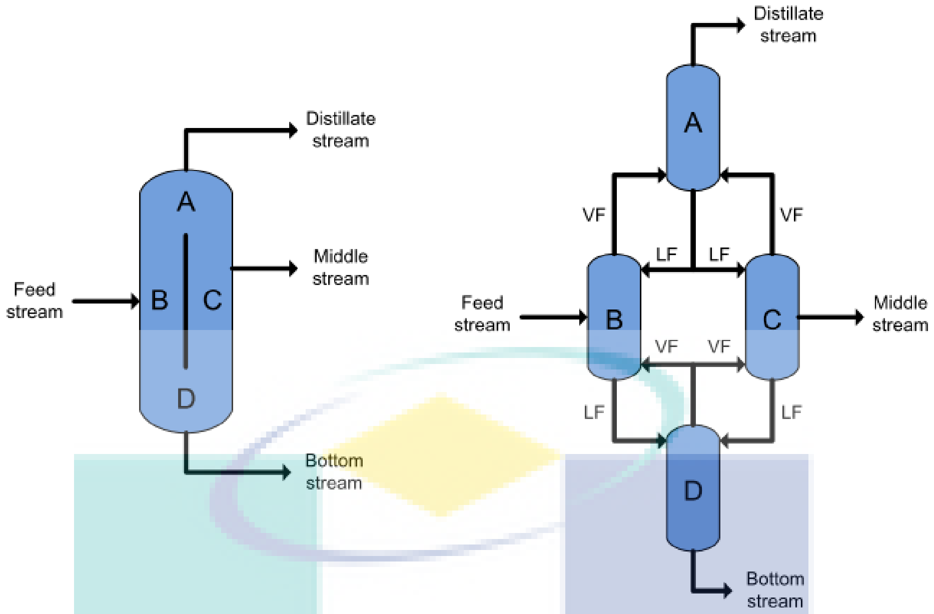


Figure 3.1. (left) Typical DWC column configuration (right) Equivalent 4 column DWC configuration used for the modelling work

plant capacity and setting up input parameters. Based on the information from our industrial partner, the feed information for fatty acid fractionation based on palm kernel oil (PKO) is shown in Table 3.1. Note that there are residues of triglyceride and unsaponifiables in the feed, which are represented by pseudocomponents methyl oleate and n-hentriacontane respectively. For the determination of the thermodynamic and hydrodynamic properties, different property packages can be used to estimate the required parameters, according to the case under study. With PKO based fatty acids being non-polar components and few experimental data, as well as a variety of properties to be estimated, equations of state have the priority. Therefore, the thermodynamic model SRK (Soave-Redlich-Kwong) is used for the modelling work [9].

The simulated capacity of the column to handle is 9000 kg/hr of PKO-based fatty acids. The DWC column configuration uses four rigorous RADFRAC units with splitters to manage the vapour and liquid load from and towards the two areas of the dividing wall section as shown in Fig. 3.2. Section A (rectifying column) and section D (stripping column) are set to only have a condenser and reboiler respectively. Whereas, both section B (pre-heat column) and section C (middle column) have no condenser or reboiler. By using this set-up, the DOF is reduced compared to a typical column compartment, which usually has a degree of freedom (DOF) of 2. Section A has only 1 DOF, where the distillate rate is then selected and specified. Section B and C have no DOF and so no specification to be given. Section D has 1 DOF, where the reboiler duty is selected and specified. In addition to that, the DWC has two additional DOF which are the liquid (RECT-SPL) and vapour (STR-SPL) load split [10]. Since vapour split is difficult to control, an equal split ratio is specified, whereas liquid split can be manipulated.

Table 3.1
PKO-based fatty acid compounds and its mole fraction defined in Aspen Plus.

Feed flowrate; kg/h	9167	
Feed temperature, C	30	
Feed pressure, bar	1	
Component name	Alias	Mole fraction
Water	H_2O	0.00035
Caproic acid	$C_6H_{12}O_2$	0.0012
Caprylic acid	$C_8H_{16}O_2$	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
Triglycerides (TAG-POLN)	$C_{55}H_{98}O_6$	0.0099

The approach for rigorous simulation is based on equilibrium-stage models, taking into account the packing rating. Using packing rating we were able to specify the column section diameter and packing details. Accordingly, the model calculates performance and hydraulic information such as flooding, downcomer backup, and pressure drop. In addition, using packing rating the dimension of the DWC column diameter can be specified and thus gives a more accurate behaviour of the column. Section A has 21 stages with a diameter of 2 meter. Both section B and C have 30 stages with 2 meter in diameter each. Section D has 35 stages with a diameter of 2 meter. The column is design for a packed column with packing factor of 72 m-1. For

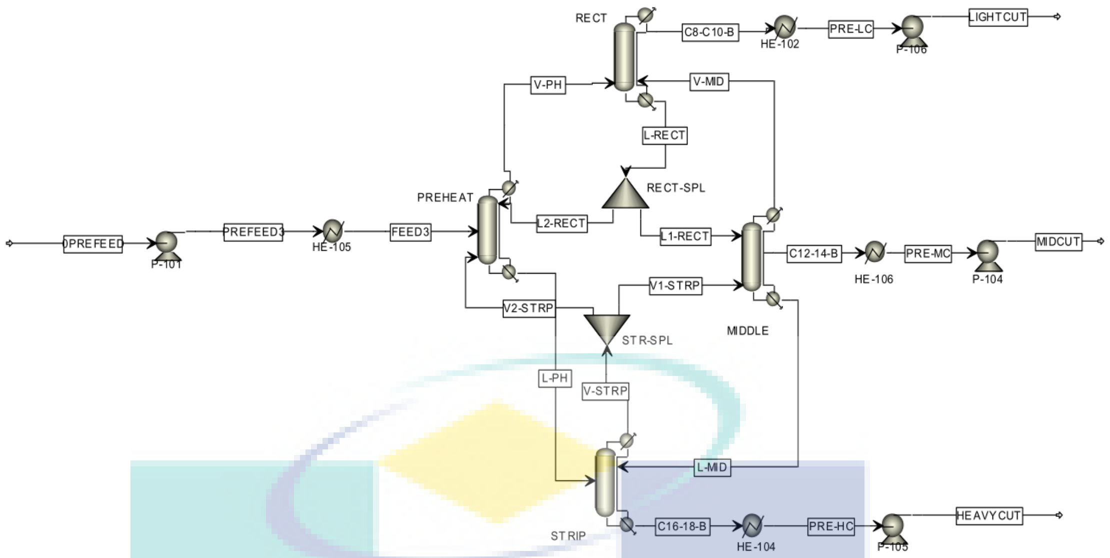


Figure 3.2. DWC four column configuration in Aspen Plus.

all stages a HETP of 0.3 meter is assumed. The feed stream is introduced at stage 10 of the pre-heat column, whereas the middle product stream draws off at stage 3 of the middle column. To prevent product degradation, the column the reboiler temperature must be below 240 C. Therefore, the top column pressure is set to 20 mbar with total column pressure drop of 12.25 mbar. The column is design to have ¿99.0 mole% product purity for each cut.

3.4 Simulation results

The simulation is done in Aspen Plus Ver. 8.3. We do encounter convergence problems. These issues result out of the lack of proper initial values and mainly the multiple internal streams, which need to be initiated, calculated for one section and concurrently serve as feed for another section i.e. simulated unit. However, convergence issues can be minimized by changing the convergence method in the RADFRAC specification tab to other than standard such as strongly non ideal liquid. Other than that the minimum convergence iteration can be increase appropriately, besides the choice of an appropriate solver method (Newton instead of Wegstein e.g.). Note that the model is not in optimal condition. Further sensitivity

Table 3.2
Result summary of the simulated DWC.

Parameters	Value	Unit
Flow rate of feed stream	9000	kg/hr
Flow rate of distillate stream	430.3	kg/hr
Flow rate of middle stream	5276.3	kg/hr
Flow rate of bottom stream	3293.5	kg/hr
Liquid split ratio	0.756	-
Vapour split ratio	0.5	-
Light cut purity (C6-C10)	99.02 / 99.34	%mol / %wt
Middle cut purity (C12-C14)	99.94 / 99.95	%mol / %wt
Heavy cut purity (C16-C18, TG, wax)	99.99 / 99.99	%mol / %wt
Reboiler duty	2300	kW
Condenser duty	-2130	kW

analysis need to be done and will be the scope of our future work. Fig. 3.3 shows the temperature and composition profile of the column. The temperature difference between the two sides is around 20 C. Such conditions seem to be easily achievable in the practical application, as little heat transfer and negligible effect on the column performance is expected [4]. Table 4.1 shows summary of the simulation results.

3.5 Technical insights

The successful implementation of unfixed wall technology by Julius Montz GmbH has increased the acceptance and implementation of DWC in industries. The application ranges from chemical to petrochemical industries. However, in the literature, Schultz et al. [11] and Kaibel et al. [12] indicate that, while theoretical researches have shown the economic advantages of DWCs, industry has been avoiding investments in these columns. One reason to avoid this type of application is a lack of understanding of its design and control.

Unlike DWC, common distillation columns are well investigated and, for moderate separation tasks, could be designed and planned via models and simulations only. For dividing wall columns however, the design process and technical aspects are more tedious. Detailed simulations and mini-plant scale experiments are necessary for model validation and general investigation of controllability [13].

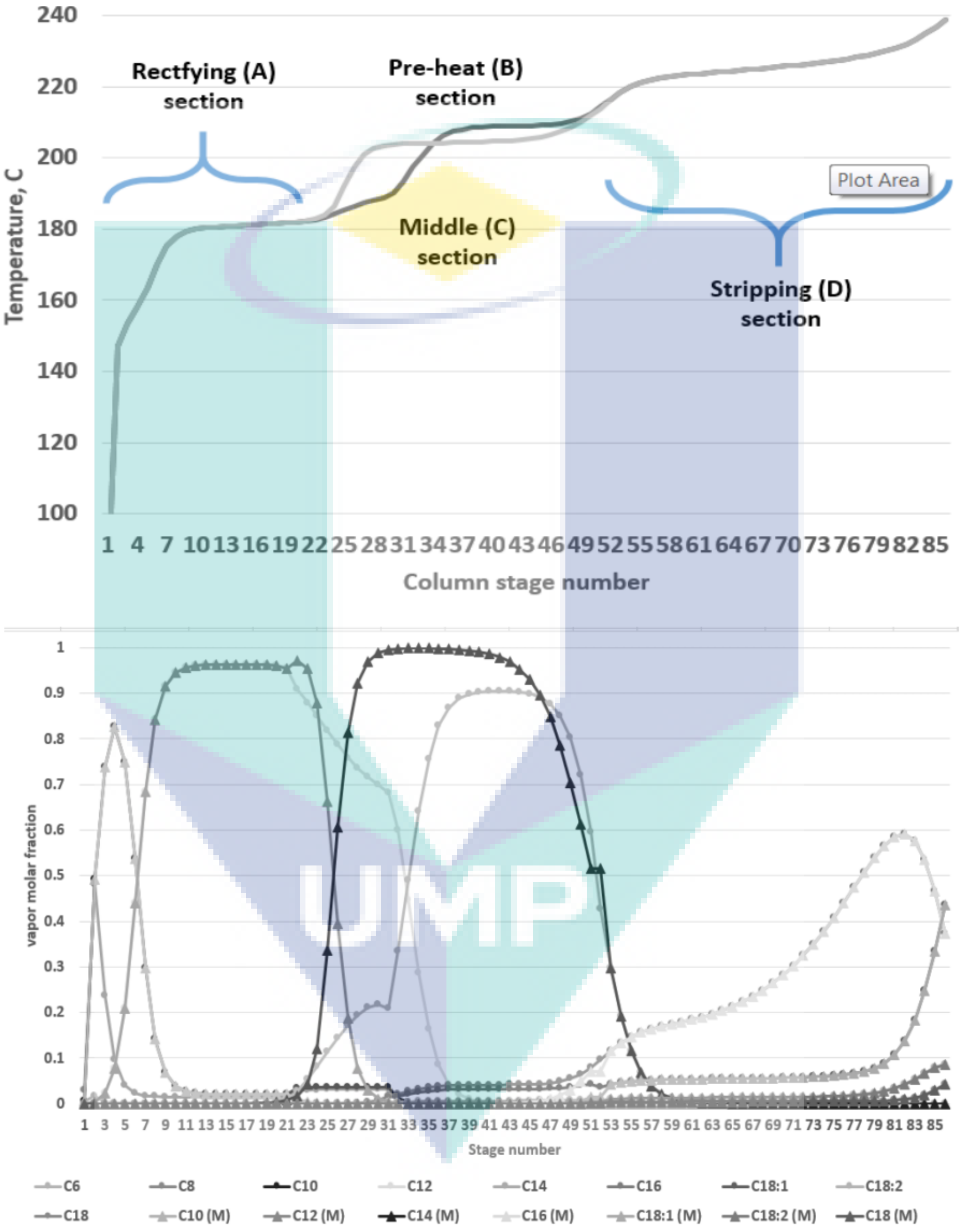


Figure 3.3. Column (top) temperature and (bottom) composition profile

The general structure and necessary utilities are similar to common distillation columns. Therefore, the set-up of general units, like reboilers, the column shell and sensors underlies a large variety of available products and expertise in establishing. But the aspect of the dividing wall leads the focus on optimal fixation of the wall, mostly within structured packages, to avoid short cut flows to gain an optimal surface area over the column diameter [8]. Therefore, it is important to understand the hydrodynamics behaviour of the column i.e. pressure drops, vapour and liquid flows, which depends on column internals such as packing selection.

Start-Up and controllability are far more complex for DWCs, due to more complex fluid dynamic relations. One issue is the above mentioned vapour split, which cannot be controlled properly. Design and simulations refer to a fixed value, which must be maintained while operation and under the influence of disturbances [10]. Here, it is possible to maintain steady state in the desired operation point via plant control or a rigorous modelling of the fluid dynamic behaviour, to determine the dependant vapour split and resulting product purifications. This behaviour could be shown via simulations, as the product specifications are sensitive regarding the vapour split. The different rectifying zones inside the DWC result in a higher necessary amount of sensors to observe vital measurements to enable sufficient plant control, as well as well-trained plant operators.

Thus the higher effort in simulation, control and construction of a DWC limits applications to more complex separation processes, like close boiling multi-component system, where no simple side straw column could be used. One asset is the higher degree of freedom of DWCs, as it enables multi-purpose plants for a variety of separation tasks for a fixed design of column and utilities [14]. For suitable applications in multi component systems DWC could be a unit of choice, as it reduces installation costs and offers the vital advantage of an internal heat integration concept compared to common applications.

3.6 Conclusion and Outlook

This work outlines the modelling and simulation work of a DWC using four column configuration model for fatty acid separation. In addition, an insight to the technical aspects is also highlighted. Our steady state rigorous simulations show the feasibility of the DWC under satisfying purification specifications. However, the overall technical process is more complex in terms of controllability and set up. Especially the fluid dynamic behaviour has to be understood to carry out proper rigorous modelling and simulation and thus be able to set up an appropriate plant control. In conclusion, it is feasible to fractionate fatty acid using DWC instead of the conventional two column separation. It is interesting however to compare both option and will be our future works. Other than that, our future work will also focus on the installation of a mini-plant, based on first simulations and design. Hence, existing models could be validated, extended or parameterized. Furthermore, start-up and dynamic behaviour will be investigated, as well as control and optimization strategies.

3.7 Acknowledgement

We gratefully acknowledge the financial support from the Malaysia Ministry of Education through ERGS (RDU130601) and FRGS (RDU140105) grant. Special thanks to Prof Dr.-Ing. Gnter Wozny (TU Berlin) and Julius Montz GmbH for their kind assistance and advice.

3.8 References

1. V.K. Sangal, V. Kumar, I.M. Mishra, Optimization of a divided wall column for the separation C4-C6 normal paraffin mixture using Box-Behnken Design, Chemical Industry & Chemical Engineering Quarterly. Volume 19(1) (2013) 107119

2. E.A. Wolff, S. Skogestad, K. Havre, Dynamics and Control of Integrated ThreeProduct (Petlyuk) Distillation Columns. AIChE Annual Meeting, (1993) Paper 195a.

3. R.K. Dohare, K. Singh, R. Kumar, S.G. Upadhyaya, Dynamic Model of Dividing Wall Column for Separation of Ternary System, Malaviya National Institute of Technology Jaipur, India. 2011

4. A.A. Kiss, R.M. Ignat, Enhanced methanol recovery and glycerol separation in biodiesel production DWC makes it happen, Applied Energy, Volume 99 (2012) 146-153.

5. F.O. BarrosoMuoz, S. Hernandez, J.G. SegoviaHernndez, H. HernndezEscoto, V. RicoRamrez, R.H. Chvez, Implementation and Operation of a DividingWall Distillation Column. Chemical Engineering & Technology, 34(5) (2011) 746-750.

6. R. Delgado-Delgado, S. Hernndez, F.O. Barroso-Muoz, J.G. Segovia-Hernndez, A.J. Castro-Montoya, From simulation studies to experimental tests in a reactive dividing wall distillation column. Chemical Engineering Research and Design, 90(7) (2012) 855-862.

7. R. Premkumar, G.P. Rangaiah, Retrofitting conventional column systems to dividing wall columns. Chemical Engineering Research and Design, 87(1) (2009) 47-60.

8. Z. Oluji, L. Matijaevi, I. Dejanovi, Dividing wall column - A breakthrough towards sustainable distilling. Chemical Engineering and Processing:Process Intensification, 49 (2010) 559580.

CHAPTER 4

PROCESS OPTIMIZATION OF DWC FOR FATTY ACID FRACTIONATION USING TAGUCHI METHODS OF EXPERIMENTAL DESIGN

The research paper have been published in Chemical Engineering Transactions Vol 45 (2015). It has an impact factor of 1.05. The paper was co-authored with Prof Gade P. Rangaiah from National University of Singapore. Below are the authors:-

- Mohamad Rizza bin Othman, Process Systems Engineering Research Group, Universiti Malaysia Pahang, 26300 Gambang, Malaysia
- Gade P. Rangaiah, Dep. of Chem. & Biomolecular Eng., National University of Singapore, Engineering Drive 4, 117585, Singapore
- Umarul I. Amran, Process Systems Engineering Research Group, Universiti Malaysia Pahang, 26300 Gambang, Malaysia

4.1 Abstract

Experimental design (ED) is a powerful technique for optimizing the performance of a process. Compared to a model-based optimization technique, ED does not require the development of mathematical formulation but nevertheless needs a good model or experimental setup and statistical analysis. This paper presents an ED based optimization approach of a dividing wall column (DWC) model for industrial oleochemical fatty acid fractionation. Taguchi method of ED will be used for the optimization of the control variables for efficient fractionation of fatty acid cuts, namely light-cut (LC), middle-cut (MC) and heavy-cut (HC). The DWC model was developed in Aspen Plus software using a rigorous four-column configuration. It is used to identify sensitive parameters, simulate experimental layout trials and results validation. The process is designed to achieve product purity of ¿99 mol.% LC and MC, and ¿90 mol.% for HC. A step-bystep approach to process optimization using Taguchi method is presented along with the statistical analysis results and their interpretation. The ED output will help in understanding the interaction between variables and their effects. With its simple, fast and non-tedious approach, ED using Taguchi method could prove its significance in improving the performance of fatty acid fractionation using DWC for possible industrial application.

4.2 Introduction

Optimization has been employed in various chemical engineering problems to find the best solution to a process within given bounds and constraints. One approach of optimization is by formulating the problem using model equations. This task requires the elements such as predictive model, objective function, constraints and control variables. Such an approach, however, requires extensive mathematical model development thus prone to modelling error, ill-defined problem, convergence issues and computational complexity. Another approach with less mathematical effort is through the use of a modular based process simulator which helps especially in developing highly interactive and complex processes. However, it still demands robust computational algorithms and prone to convergence problems. ED is an interesting alternative to model or equation based optimization. ED does not require extensive mathematical development; nevertheless, it needs a good model or exper-

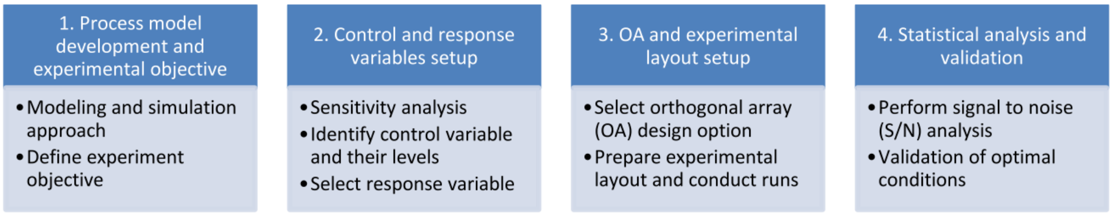


Figure 4.1. General steps for optimization using experimental design

imental setup and statistical analysis. It is useful to study and understand process parameters interaction and then optimise the process performance using limited budget and resources. Taguchi method is a powerful ED technique (Antony et al., 2001), and has been applied successfully in many applications that involve complex process interactions. This paper discusses the application of Taguchi method for optimising a rigorous four column DWC model developed in Aspen Plus. A four column DWC configuration is a non-standard model in Aspen Plus and not easily converged. Carrying such a simulation requires experience and is computationally demanding. To achieve optimal design a lot of tuning is needed especially on sensitive parameters; for this, ED comes in handy. By implementing ED based optimization it is expected to minimize the computational complexity compared to model or equation based optimization i.e. Aspen optimization tool. This way, the complex model development using process simulators could be utilised along with a simpler approach for optimization; hence, it provides a simpler and practical approach for optimizing the performance of the process. The general steps for optimization using Taguchi method is illustrated in Figure 4.1.

4.3 DWC model development and experimental objective

DWCs are advantageous for separating ternary mixtures into pure fractions, especially medium boiling component. Its unique internal configuration reduces the investment and operating costs, which leads to lower carbon dioxide emission (BarrosoMuoz et al., 2011). Because of these advantages, DWC has attracted many researchers and companies for various applications. To name a few biodiesel fractionation (Cho et al., 2015), azeotropic and extractive distillation process (Kiss et al., 2013), pressure swing absorption (Loy et al., 2015) and methyl acetate production (An et al., 2015). In 2013, crude palm oil (CPO) production in Malaysia recorded an increase of 2.3% to 19.22 Mt against 18.79 Mt recorded in 2012 (AOTB, 2014). The oleochemical industry in Malaysia is now one of the largest in the world, which currently accounts for 39% of world palm oil production and 44% of world exports (MPOC, 2014). There are 18 oleochemical companies in operation in Malaysia; our survey shows that none of them apply DWC for their fractionation process. To show the applicability of DWC in oleochemical industry, an industrial DWC column for fatty acid fractionation is modelled using rigorous four RADFRAC configuration in Aspen Plus. The model is based on the modelling work of Othman and Illner (2014), which will be used to identify sensitive parameters, simulate experimental trials and results validation. The experimental objective is to have the response variable or product purity of ¿99 mol.% for LC and MC and ¿90 mol.% for HC with optimal setting of the control variables. Insights on the modelling approach is presented next.

4.3.1 Modelling and simulation approach

Sequential-modular (SM) based modelling of DWC using commercial simulators typically involves rigorous simulation of two columns (Kiss and Ignat, 2012); prior to rigorous simulation, Premkumar and Rangaiah (2009) employed three short-cut columns for estimating number of stages for rigorous simulation. A four column configuration has been employed by Dejanovic et al., (2011) for an aromatics processing plant. Recently, Othman and Illner (2014) employed four column configuration for fatty acid fractionation as shown in Figure 4.2. A four column configuration offers few advantages including flexibility in dimensioning the column sections and suitable for control system study in dynamic simulation (Dejanovic et al., 2011). Conversely, it requires extensive computational effort because of the complex interactions between different blocks that involves several recycle loops and more interconnected

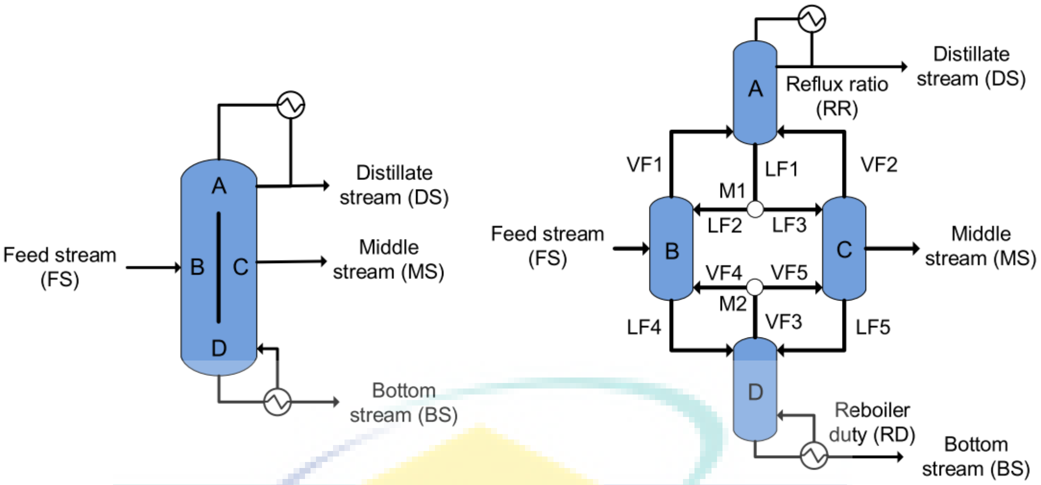


Figure 4.2. (Left) Typical DWC configuration (Right) Equivalent 4-column DWC configuration

streams. This lead to lack of proper initialization and therefore prone to convergence error. Simulating the model is not straightforward compared to a standard distillation block in Aspen Plus. A lot of tuning need to be done for the model to converge and modelling experience could help a lot.

4.4 Control and response variables setup

The next step in ED procedure is to define the control and response variables. In this work, several control variables were selected based on work by Dohare et al. (2011), they include reflux ratio (RR), liquid split to section C (LF3), vapour split to section B (VF4), and reboiler duty (RD) as shown in Figure 2. These control variables were varied to investigate their effect on the response variables, which are the product compositions, namely, LC at the distillate stream, MC at the middle stream and HC at the bottom stream. These effects can be investigated by applying sensitivity analysis approach. In Aspen Plus, sensitivity analysis can be done either in sequential-modular (SM) mode or equation-oriented (EO) mode. In this work, sensitivity analysis based on SM mode was employed using the model analysis tool in Aspen Plus. During sensitivity analysis, all the variables were kept constant except the control variable under study. Apart from observing the variables interactions,

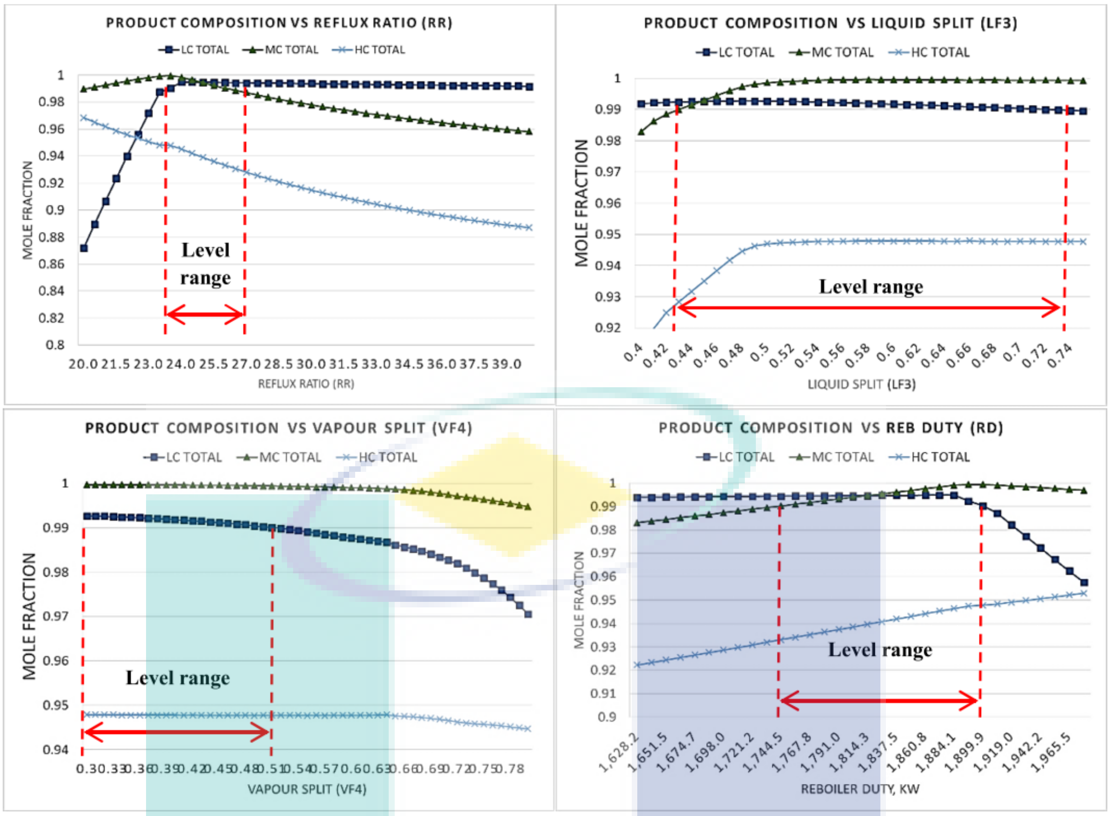


Figure 4.3. Effect of different control variables on product composition

Table 4.1
Control variable and corresponding levels

Control variable	Level				
	1	2	3	4	5
Reflux ratio (RR)	23.6	24.3	25.1	25.8	26.5
Liquid split (LF3)	0.43	0.503	0.575	0.648	0.72
Vapour split (VF4)	0.30	0.353	0.405	0.458	0.51
Reboiler duty (RD)	1733 kW	1775 kW	1817 kW	1858 kW	1900 kW

sensitivity analysis is also used to identify possible range for the control variable levels. It is recommended to choose the range, which is able to meet the desired design value. The sensitivity results and its corresponding control variable level range are shown in Figure 4.3. The control variables and its corresponding levels are then given in Table 4.1.

4.5 Orthogonal array and experimental layout design and run

Orthogonal array (OA) is a simple and useful tool to design an experiment that helps the designer to study the influence of multiple controllable factors on the average of quality characteristics and the variations in a fast and economic way (Panda and Singh, 2013). The selection of OA depends on several factors including number of interactions, number of factors, time and cost (Antony et. al, 2001). For the present study, the experimental design was based on the L25 orthogonal array of Taguchi method. The OA layout is shown in Table 4.2. The simulation was conducted according to the experimental layout. For physical experiment, it is necessary to repeat the experimental run several times in order to have adequate degrees of freedom for the error term (Antony et al., 2001). However, in the present work, the case is modelled and simulated using process simulators. Therefore, a single unrepeated run is sufficient since the initialization value is the same. The product composition for each cut corresponding to each run is also shown in Table 2. It can be seen from this table that not all runs produce the desired product specification. This is due to the variation of control variables setting in the OA matrix that significantly affects the response variable unlike sensitivity analysis approach whereby only one control variable was varied while the rest were kept constant.

4.6 Statistical analysis and validation

The simulation data were analysed using signal to noise (S/N), which measures the functional robustness of product or process performance in the presence of undesirable external disturbances (Kapur and Chen, 1988). The larger-the-better (LB) response was applied to ensure adequate product purity. Using this response, the S/N ratio was calculated using the following equation:

$$\frac{S}{N} = -10 \log \frac{1}{n} \sum_{k=1}^{n} \frac{1}{y^2} \tag{4.1}$$

Table 4.2

L25 orthogonal array of Taguchi experimental layout, its response and S/N results

Run	RR	LF3	VF4	RD	Mole %			S/N ratio			
					LC	MC	HC	LC	MC	HC	Overall
1	23.6	0.430	0.300	1733	99.4369	95.9924	86.5863	39.9509	39.6447	38.7490	39.4177
2	23.6	0.503	0.353	1775	99.4499	98.4151	92.0833	39.9521	39.8612	39.2836	39.6887
3	23.6	0.575	0.405	1817	99.4637	99.4209	93.9559	39.9533	39.9496	39.4585	39.7808
4	23.6	0.648	0.458	1858	99.4769	99.7414	94.3889	39.9544	39.9775	39.4984	39.8045
5	23.6	0.720	0.510	1900	98.9761	99.9332	94.7764	39.9106	39.9942	39.5340	39.8083
6	24.3	0.430	0.353	1817	99.4496	97.1675	89.1709	39.9521	39.7504	39.0045	39.5495
7	24.3	0.503	0.405	1858	99.4619	99.1113	93.4453	39.9531	39.9225	39.4111	39.7550
8	24.3	0.575	0.458	1900	99.4751	99.6907	94.3239	39.9543	39.9731	39.4924	39.8008
9	24.3	0.648	0.510	1733	99.4181	98.6817	92.7931	39.9493	39.8847	39.3503	39.7197
10	24.3	0.720	0.300	1775	99.4335	98.9367	93.1727	39.9507	39.9072	39.3858	39.7402
11	25.1	0.430	0.405	1900	99.4589	98.1407	91.3360	39.9529	39.8370	39.2128	39.6552
12	25.1	0.503	0.458	1733	99.4001	97.8118	91.3348	39.9477	39.8078	39.2127	39.6442
13	25.1	0.575	0.510	1775	99.4154	98.6137	92.7139	39.9491	39.8787	39.3429	39.7150
14	25.1	0.648	0.300	1817	99.4304	98.8739	93.0890	39.9504	39.9016	39.3780	39.7355
15	25.1	0.720	0.353	1858	99.4444	99.1282	93.4587	39.9516	39.9239	39.4124	39.7555
16	25.8	0.430	0.458	1775	99.4011	96.7046	88.8041	39.9478	39.7089	38.9687	39.5215
17	25.8	0.503	0.510	1817	99.4151	98.4683	92.5614	39.9491	39.8659	39.3286	39.7057
18	25.8	0.575	0.300	1858	99.4294	98.7795	93.0151	39.9503	39.8933	39.3711	39.7303
19	25.8	0.648	0.353	1900	99.4433	99.1072	93.4291	39.9515	39.9221	39.4096	39.7539
20	25.8	0.720	0.405	1733	99.3836	98.1580	92.0198	39.9463	39.8385	39.2776	39.6774
21	26.5	0.430	0.510	1858	99.4151	97.6218	90.7370	39.9490	39.7909	39.1557	39.6182
22	26.5	0.503	0.300	1900	99.4290	98.3893	92.2907	39.9503	39.8590	39.3032	39.6946
23	26.5	0.575	0.353	1733	99.3678	97.8144	91.6163	39.9449	39.8081	39.2395	39.6533
24	26.5	0.648	0.405	1775	99.3841	98.1608	92.0279	39.9463	39.8388	39.2784	39.6778
25	26.5	0.720	0.458	1817	99.3997	98.3953	92.3665	39.9477	39.8595	39.3103	39.6965

Here, n is the number of repeated experiments and y is the response of the experiment. Note that n is considered 1 for computational modelling. Calculation of S/N ratio was done in Minitab Ver. 17. The calculated S/N ratio is shown in Table 4.2, and its main effect plot is illustrated in Figure 4.4. The main effect plot elucidate the interaction of different control factor levels to the responses output. Generally, the highest peak of S/N ratio in the graph is the optimum condition for robust process performance. In Figure 4a, RR and RD have major contributions to LC purity, and LF3 and VF4 have minor effect except at the end of the levels range. The optimum design variables for LC was found to be 24.3 for reflux ratio, 0.43 for liquid split, 0.30 for vapour split and 1,858 kW for reboiler duty. With this setting, the purity of LC is 99.46%. Interestingly, it is found that the main effect plots for MC and HC (in Figure 4.4b and 4.4c) have the same trend. Whereby, LF3 and RD contributes the most in controlling the product purity of both streams. RR and VF4 contribute moderately. Figures 4.4b and 4.4c show the main effects plot for MC and HC. Both figures show almost identical effect. For MC the optimal setting was 24.3 for reflux ratio, liquid split and vapour split were 0.648 and 0.51, the reboiler duty was 1,900

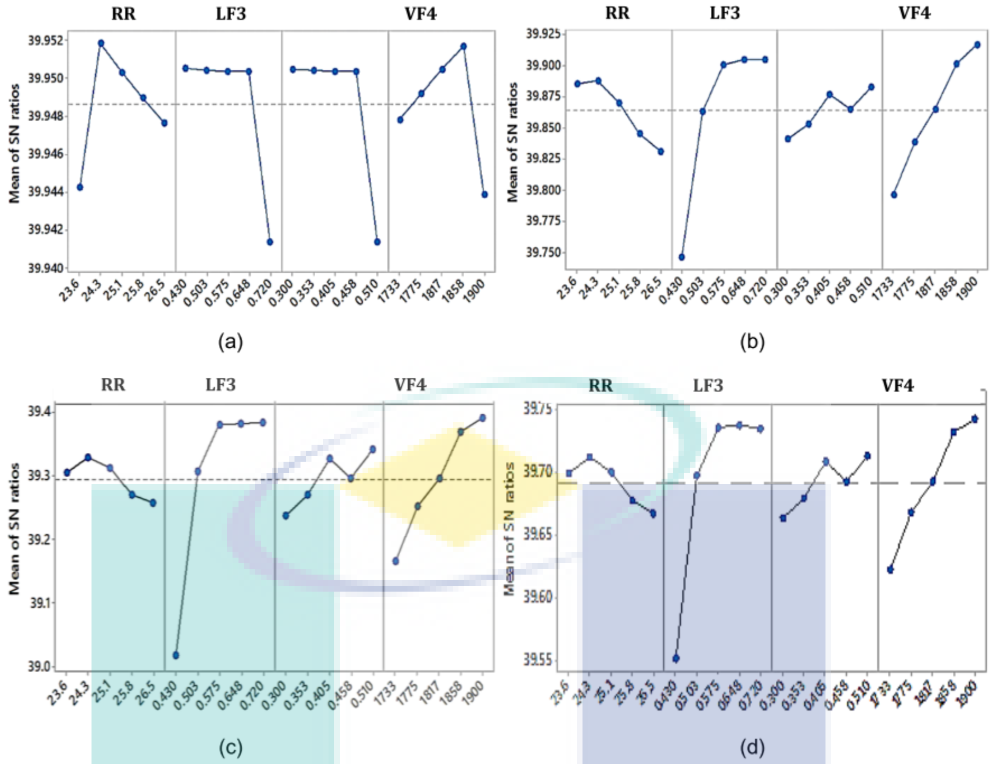


Figure 4.4. Main effect response plot for S/N ratio (a) Light-cut (b) Middle-cut (c) Heavy-cut (d) Overall

kW. This setting achieves MC purity of 99.70%. HC purity achieved is 94.33% with optimum setting of 24.3 for reflux ratio, 0.72 and 0.51 for liquid and vapour split and 1,900 kW for reboiler duty. For overall response, the analysis takes into account the multiple response parameters, namely LC, MC and HC purity. Such analysis is possible due the same unit of measurement. Overall response result in Figure 4d shows that the most contributing variables out of the selected control variables were LF3 and RD whereas RR and VF4 contribute moderately. It is also found that increase of RR reduces the product purity especially MC and HC whereas increase of LF3, VF4 and RD increases the product purity especially the MC and HC. The overall optimum setting was 24.3 for reflux ratio, liquid split and vapour split were at 0.648 and 0.510, and reboiler duty was 1,900 kW. With this setting, the process able to achieve the desired purity: 99.98 mol.% for LC, 99.70 mol.% for MC and 94.33 mol.% for HC.

4.7 Conclusions

In this paper, an experimental design based on Taguchi method was applied to optimize the control variables of a complex four column DWC model for fatty acid fractionation. Analogous to physical experiment, the model was used to identify sensitive parameters, simulate experimental trials and validate results. The paper also illustrated sensitivity analysis approach to identify suitable range for selection of the control variable levels. It is suggested to select control variable range that lies within the desired conducted using S/N ratio. Overall it is found that the most contributing variables that affect the product purity were LF3 and RD. The selected optimal design variable values were found to be reflux ratio of 24.3, liquid split and vapour split were 0.648 and 0.510, and reboiler duty of 1,900 kW. With this setting, the experimental design was able to obtain the desired purity for all products. It will be interesting to apply the approach for optimizing the operation and installation cost of DWC, which will be our future work. Use of ED significantly reduces the extensive effort of mathematical development especially for complex processes. Other than that, fewer runs and iteration were needed, thus avoiding computational complexity. However, ED might not be as accurate, precise and extensive as mathematical model based optimization since the latter accounts for process phenomenon through mass, equilibrium, summation and enthalpy (MESH) equations. Overall, our study demonstrates the successful integration of Taguchi method to a complex model developed using process simulators, for simple, fast and non-tedious approach of process optimization.

4.8 Acknowledgement

We gratefully acknowledge the financial support from the Malaysia Ministry of Education through ERGS (RDU130601) and FRGS (RDU140105) grant.

4.9 References

1. An D., Cai W., Xia M., Zhang X., Wang F., 2015, Design and control of reactive dividing-wall column for the production of methyl acetate, Chemical Engineering and Processing: Process Intensification, 92,45-60
2. Antony J., Warwood S., Fernandes K., Rowlands H., 2001, Process optimisation using Taguchi methods of experimental design, Work Study, 50(2), 51-58.
3. AOTB (Advanced Oleochemical Technology Division), 2015. Oleochemical Industry in Malaysia ¡portal.mpob.gov.my/aotd/industry.htm¿, accessed 06.08.2015.
4. BarrosoMuoz F.O., Hernandez S., SegoviaHernndez J.G., HernndezEscoto H., RicoRamrez V., Chvez R.H., 2011, Implementation and operation of a dividingwall distillation column, Chemical Engineering & Technology, 34(5), 746-750.
5. Cho H.J., Choi S.H., Kim T.Y., Kim J.K., Yeo Y.K., 2015, Design of a dividing wall column for fractionation of biodiesel, Korean Journal of Chemical Engineering, 32(7), 1-14.
6. Dejanovic I., Matijasevic L., Jansen H., Olujic Z., 2011, Designing a packed dividing wall column for an aromatics processing plant, Industrial and Engineering Chemistry Research, 50, 5680-5692.
7. Dohare R.K., Singh K., Kumar R., Upadhyaya S., Gupta S., 2011, Dynamic model of dividing wall column for separation of a ternary system, In CHEMECA -2011, September 2011. New South Wales, Australia.
8. Kapur K.C., Chen G., 1988, Signal-to-noise ratio development for quality engineering, Quality and Reliability Engineering International, 4, 133-41.
9. Kiss A. A., Ignat R. M., 2012, Enhanced methanol recovery and glycerol separation in biodiesel production DWC makes it happen, Applied Energy, 99, 146-153.

10. Kiss A.A., Suszwalak D.J.C., Ignat R.M., 2013, Breaking azeotropes by azeotropic and extractive distillation in a dividing-wall column, Chemical Engineering Transactions, 35, 1279-1284.

11. Loy Y.Y., Lee X.L., Rangaiah G.P., 2015, Bioethanol recovery and purification using extractive dividing wall column and pressure swing adsorption: An economic comparison after heat integration and optimization, Separation and Purification Technology, 149, 413-427.

12. Othman M.R., Illner M., 2014, Modelling and simulation of a dividing wall column for separation of fatty acid in oleochemical industries, In SOMChE & RSCE 2014, 29-30 October 2014. Kuala Lumpur, Malaysia.

13. Panda A.K., Singh R.K., 2013, Optimization of process parameters by Taguchi method: Catalytic degradation of polypropylene to liquid fuel, Int. J. of Multidisciplinary and Current research, Sept/Oct 2013, 50-54.

14. Premkumar R., Rangaiah G.P., 2009, Retrofitting conventional column systems to dividing-wall columns, Chemical Engineering Research and Design, 87(1), 47-60.

CHAPTER 5

PRIORITIZING HAZOP ANALYSIS USING ANALYTIC HIERARCHY PROCESS (AHP)

The research paper have been published in Clean Technology and Environment Policy. It has an impact factor of 1.05. Below are the authors:-

- Mohamad Rizza bin Othman, Process Systems Engineering Research Group, Universiti Malaysia Pahang, 26300 Gambang, Malaysia
- Rosshila Idris, Process Systems Engineering Research Group, Universiti Malaysia Pahang, 26300 Gambang, Malaysia
- Mimi Haryani Hassim, Department of chemical Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
- Umarul I. Amran, Process Systems Engineering Research Group, Universiti Malaysia Pahang, 26300 Gambang, Malaysia

5.1 Abstract

Hazard and operability (HAZOP) analysis is one of the most widely used methods for process hazard analysis (PHA). However, the outcome of HAZOP analysis could results in identifying large number of hazards thus poses a challenge for assessors to take actions in dealing with all the hazards. The common practice in prioritizing the critical hazards is based on assessors experience through deductive judgment using rating scale taking into consideration safety and the associated costs. Although simple and straight forward it lack the systematic approach to elucidate different conclusions into an integrated outcome thus susceptible to inaccurate and unjustified decisions. In this paper, we present a methodology for incorporating prioritization in HAZOP analysis using analytic hierarchy process (AHP). Through this approach the identified hazards from a process will be quantitatively weighted and ranked based on their priority along with the appropriate counter measures to be taken. The proposed methodology is a thorough decision making tool as it does not only prioritized the hazards identified from HAZOP assessment, but also provides medium for the assessors to quantitatively analyze the hazards. To show its efficacy the approach will be applied to a simple reactor unit and a more complex system of dividing wall column (DWC) pilot plant as case studies. The result shows that the proposed methodology is able to identify and rank the most significant hazards in a process following HAZOP analysis. This is particularly useful, especially to process designers/engineers in prioritizing their efforts and resources on more significant hazards, hence aiding towards achieving an inherently safer chemical process.

5.2 Introduction

Over the past 20 years, the concept of occupational health and safety in management system (OHSMS) has become common in industry. There are various OHSMS-based standards, guidelines, and inspections system have been developed (Robson et al., 2007) and applied within the public and private sector worldwide. In spite of stricter and more stringent execution management of projects throughout the project lifecycle in particularly from the occupational safety and health (OSH) aspect, there are still accidents take place from time to time in construction as well as the industries. Industrial accidents continue to cause human suffering, capital losses, environmental destruction and social problem (Badri et al., 2012). Therefore it is very important to evaluate the potential risk of a project before it embarks, which is typically evaluated in terms of its consequences with respect to project performance. In risk assessment, quality, schedule and costs are the most important parameters that need to be considered.

Process hazard analysis (PHA) is imperative for inherently safer design and operation of chemical processes. Many methods and tools are available for performing PHA either quantitatively and/or qualitatively. One of the most widely used methods is the hazard and operability (HAZOP) study. HAZOP is considered as a formal procedure to identify hazards in a chemical process facility. Conducting HAZOP however is demanding and exhaustive. Due to its let the mind go free approach, HAZOP analysis could result in a vast number of hazards being identified. These situations form a complex decision making process with interrelated components. This consequently led to poor hazards prioritization and difficulty in selecting actions that address the most substantial hazards especially when safety and cost criteria are involved. Presumably, while interacting with such complex scenarios, the better the decision makers understand this complexity, the better the decision will be.

When facing a multi criteria problem, generally, there are two known ways to derive an answer (Saaty, 2001). First is by using deductive logic with assumptions and carefully deducing an outcome from them using scale such as Likert scale. This method is commonly used in rating HAZOP analysis results, but it has its drawbacks whereby the lack of information on how to bring the different conclusions into an integrated outcome can elucidate inaccurate and unjustified conclusions. The second approach is done by laying out all possible factors in a hierarchy or in a network system and deriving answers from all possible relative influences. While both approaches offer simplicity in deducing answers, the drawbacks of the first approach lies on its inability to consider the assessor's preferability towards certain criteria or indicators. Often the importance of the elements is neglected. Analytic hierarchy process (AHP) is a multi-criteria decision making (MCDM) methodology based on hierarchical structure and most suitable for MCDM problems (Narayanan et al., 2007). Its hierarchical and systematic method makes it a popular technique to solve MCDM problems and have been successfully implemented in various fields from education (Othman et al., 2012), chemical process assessment (Othman et al., 2010; Tan et al., 2014), business, sports and even military purposes. In safety related fields, AHP has been applied in several applications such as selection of contractors for safer turnaround maintenance (Hadidi & Khater, 2015), selection of safety devices (Caputo et al., 2013) and safety risk assessment of constructions projects (Aminbakhsh et al., 2013; Taylan et al., 2014). However, application of AHP to HAZOP analysis were scarce. Therefore, in this paper, we present and highlighted a systematic methodology that embed hazard prioritization in HAZOP analysis procedure using analytic hierarchy process (AHP), called as the HAZOP-AHP. The proposed approach will be demonstrated to a simple reactor and dividing wall column pilot plant for fatty acid fractionation as case studies.

5.3 HAZOP-AHP Methodology

HAZOP-AHP is developed as a methodology which incorporates a multi-criteria decision problem making approach to prioritize the hazards that may contribute to the undesirable events identified from the HAZOP analysis. The general steps to the methodology is depicted in Figure 5.1.

5.3.1 HAZOP Analysis

Generally, HAZOP analysis is used to identify how a process may swerve from its design. It is considered as an engineering intention of new facilities to judge the potential for malfunction of individual equipment, the consequences and the actions that need to be taken. Some questions can be asked during HAZOP implementation, such as What deviations could occur, What are the relevant guide words and process parameters, Why do they occur? (implying the causes) and How

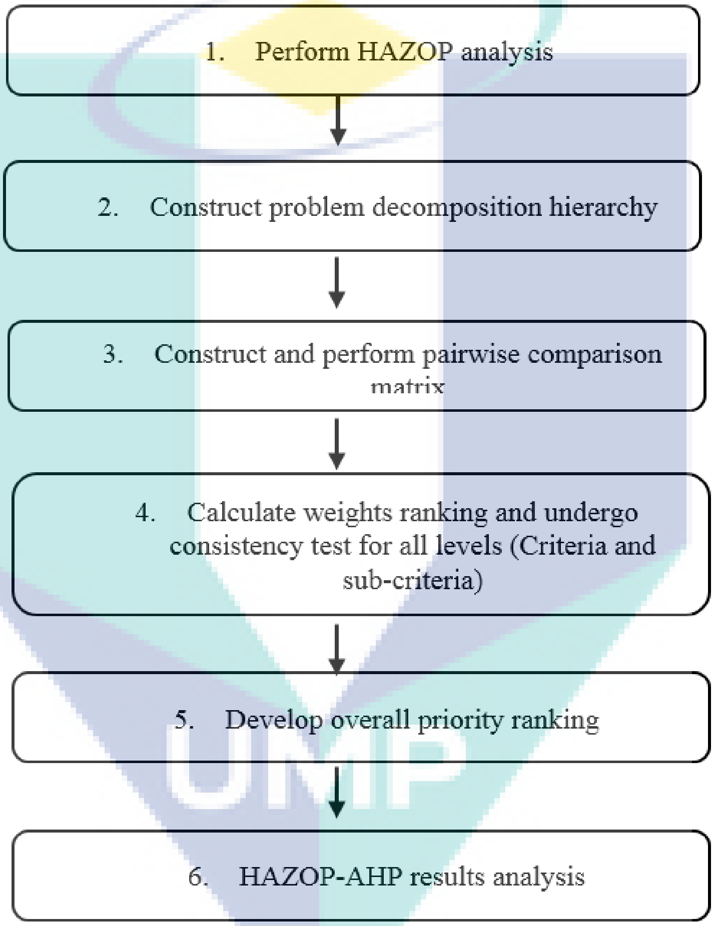


Figure 5.1. The procedure steps in conducting the HAZOP-AHP

are they revealed (indicating the consequences) (Rossing et al., 2010). In HAZOP, there are a set of guide words that need to be applied, namely NONE, LESS OF, MORE OF, PART OF, REVERSE, AS WELL AS, and OTHER THAN, which correspond to qualitative deviations of process variable (Venkatasubramanian et al., 2000). HAZOP is preferable to be carried out as early as possible in the design phase to have significant influence on the design. However, to perform HAZOP, a complete and detailed design of a process is needed. Therefore, HAZOP is often carried out as a final check when the detailed design has been completed (Rausand, 2005). Despite of requiring such detailed information on process, the resulting HAZOP analysis output however provides limited data only (plus qualitative), thus many of those hazards identified may have low probability or consequences (Crowl & Louvar, 1990). As an alternative to address this issue, AHP, which is a widely used decission making tool, can be incorporated into the typical HAZOP procedure to provide a mean for prioritization of the risks and consequences. This is to ensure that the most significant hazard(s) is being addressed first properly within the available resources.

5.3.2 AHP Methodology

Analytic hierarchy process (AHP) is a multi-criteria decision making (MCDM) methodology based on hierarchical structure which performs decision trade-off between multiple objectives in a hierarchical structure. It provides the objective mathematics needed to process the inescapably subjective and personal preferences of individuals or groups in making a decision and well suited to decisions in which the criteria are qualitative and have a large subjective component, thus requiring judgments (Bahurmoz, 2003). It accepts any particular constitutive criterion for inclusion and allows individual decisions to be aggregated into overall criteria, which allows other members to review and participate in that aspect of the decision making process at an appropriate level of detail. AHP was introduced in 1980 by Thomas L. Saaty and makes it a popular technique for solving multi criteria decision making (MCDM) problem. Some of the advantages of AHP are that it:-

- Provides a systematic and simple approach
- Hierarchy-based
- Offers multiple and specific criteria for decision inclusion
- Accepts team work participation (Dyer & Forman, 1992)

The development of AHP for decision making requires four steps, namely, problem decomposition, weighting, ranking and evaluation. Apart from decision making, AHP can be used to derive scales of measurements such as priority or weights setting for tangible and intangible elements by simple pairwise comparisons. It uses an absolute scale (see Table 1) to express how much one element dominates another with respect to a control criterion. A value greater than 1 indicates that the base criterion is relatively more important that the paired criterion. A reciprocal value is assigned to the inverse comparison. The comparison proses can be aided using a series of questions that relates the compared elements to the control criteria. An example, one can ask How much important A1 compared to A2 when assessing process and plant design project? Based on the scale in Table 5.1, if value of three is taken, it simply means that, A1 is moderately more important that A2 when assessing process and plant design project. The scale or priority value derived from this comparison matrix is a ratio scale. It is important to note that assigning scale to the elements is subjective thus the assessors knowledge, experience and judgement is crucial.

The mathematics used to derive the priority value is based on reciprocal matrices and eigenvector theory. In general, the priority value is calculated by solving the following equation;

$$\mathbf{A} \cdot \omega = \gamma \cdot \omega \tag{5.1}$$

where **A** is the pairwise comparison matrix, ω is the eigenvector and is the largest eigenvalue of **A**. The mathematical theorem is rather complicated and any

Table 5.1

The fundamental scale of absolute numbers

Intensity of	Importance	Definition Explanation
1	Equal Importance	Two activities contribute equally to the objective
2	Weak or slight	
3	Moderate importance	Experience and judgement slightly favour one activity over another
4	Moderate plus	
6	Strong plus	
7	Very strong or demonstrated importance	An activity is favoured very strongly over another; its dominance demonstrated in practice
8	Very, very strong	
9	Extreme importance	The evidence favouring one activity over another is of the highest possible order of affirmation
Reciprocals of above	If activity i has one of the above non-zero numbers assigned to it when compared with activity j, then j has the reciprocal value when compared with i.	A reasonable assumption
1.11.9	If the activities are very close	May be difficult to assign the best value but when compared with other contrasting activities the size of the small numbers would not be too noticeable, yet they can still indicate the relative importance of the activities.

interested reader may refer to Saaty (1980) for further reading. There are several algorithms for approximating ω. Chung et al. (2005) explain a three step procedure to synthesize the priority value. The method of calculating priorities reflects the benefits the AHP provides over simply assigning numbers to subcriteria and the alternatives (Bahurmoz, 2003).

The eigenvector value ω can be presented into two modes; distributive and ideal. Distributive mode normalizes the value under each element in such that the summation of all value equal to one. On the other hand, ideal mode divides the value for each element by score of the best element. The best element will always have ideal value of one. To choose which mode to be used, Millet and Saaty (2000) suggest using distributive mode to determine the extent to which each element dominates all other element under the control criterion. Whereas, ideal mode is use to determine how well each element performs relative to a benchmark or best practice element. However, study by Saaty and Vergas (1993) shows that the different of using each mode will only lead to 8%.

5.3.3 Problem decomposition hierarchy

Problem decomposition is very important in decision making. The best and most organized way to decompose a problem is by structuring it into a hierarchical form which starts at the top or first level with a goal or problem statement and ends with the alternatives to be evaluated. Between these two levels are the top down related elements that describe the system. A hierarchy is an abstraction of the structure of a system to study the function interactions of its components and their impacts on the entire system (Saaty, 1980). The abstraction of the problem model can range from simple to complex decision tree depending on the problem complexities. However, it must be well defined for a justifiable and accurate outcome. The interaction at the highest level with the elements at the lower level can be in a of a linear hierarchy or non-linear hierarchy. The former is the simplest form, rising from one level of elements to an adjacent level. The latter involves circular arrangements in which an upper level might be dominated by a lower level as well as being in a dominant position. The advantages of hierarchy modeling include (Saaty, 1980):

- Hierarchical representation of a system can be used to describe how changes in priority at an upper level affect the priority of elements in lower levels.
- They give great detail of information on the structure and function of a system in the lower level and provide an overview of the actors and their purposes in the upper level.
- Natural systems assembled hierarchically, i.e. through modular construction and final assembly of modules, evolve more effiently than those assembled as a whole.
- They are stable and flexible; stable in that small changes have small effect and flexible in that addition to a well-structured hierarchy they do not disrupt the performance.

Figure 5.2 shows the general problem decomposition guideline in HAZOP-AHP with several levels which include the overall goal, criteria, sub criteria, and alternatives to form a linear hierarchy involving all of them in several levels. It starts with the first level indicating the goal of the analysis, which could be identifying the main causes or consequences of a HAZOP analysis. The goal is then expanded into the second level criteria which is the analysis boundary node. The node could be identified as process stream, unit operation etc. Note that for each node has its own unique breakdown. Each of the node will be further broken down to the third level which is the related process parameters i.e. flow, pressure, temperature etc. For each process parameters it will be further broken down to the fourth level which describe the deviation of the parameters, according to the guide words (no, less, more, inverse, high, low). The fifth level is attached to the process parameters level which list the causes that indicate condition that gives rise to the deviation of the

Table 5.2

Example of pairwise comparison matrix

FLOW	No (j=1)	Less (j=2)	More (j=3)	Reverse (j=4)
No (i=1)	1	A12	A13	A14
Less (i=2)	1/A12	1	A23	A24
More (i=3)	1/A13	1/ A23	1	A34
Reverse (i=4)	1/ A14	1/ A24	1/ A34	1

parameters such as clogging in pipeline, pump failure, main valve close, empty tank, etc. Finally, the last level of the hierarchy, are the consequences which are attached to each of the fifth level. To facilitate the event tracking, a unique identification number is used which is based on multilevel numbering system.

5.3.4 Pairwise comparison matrix

In pairwise comparison, two components are compared with respect to the upper level control criteria using scale of relative importance. Identify a value of Aij, which indicates the importance of i-th element (left) compared to the j-th element (top) as shown in Table 5.2 below. The scaling factor is based on the guideline in Table 5.1. In AHP, relative measurements about pairwise comparison ratios with respect to the strength of preference between elements of comparison are based on human intuition (Xia & Wu, 2007). Therefore, the decision makers need to express their opinion regarding the value of single pairwise comparison at a time and need to choose their answer based on the Saaty (1980) evaluation scale.

Where the value ¿1, if i-th element is more important than j-th element. Value ¡1, for the inverse importance.

For the inverse comparison between the elements, a reciprocal value is allocated as Aji = 1/ Aij. Each entry Aij of A is the answer of a typical question, between the two factors Fi and Fj, which one is more dominant (or preferable or important) and what is the degree of this dominance? The answers are usually given verbally, like F1 is weakly (or strongly) more dominant over Fj. Later, these verbal qualitative phrases (weakly or strongly more) are quantified by means of the (1-9)

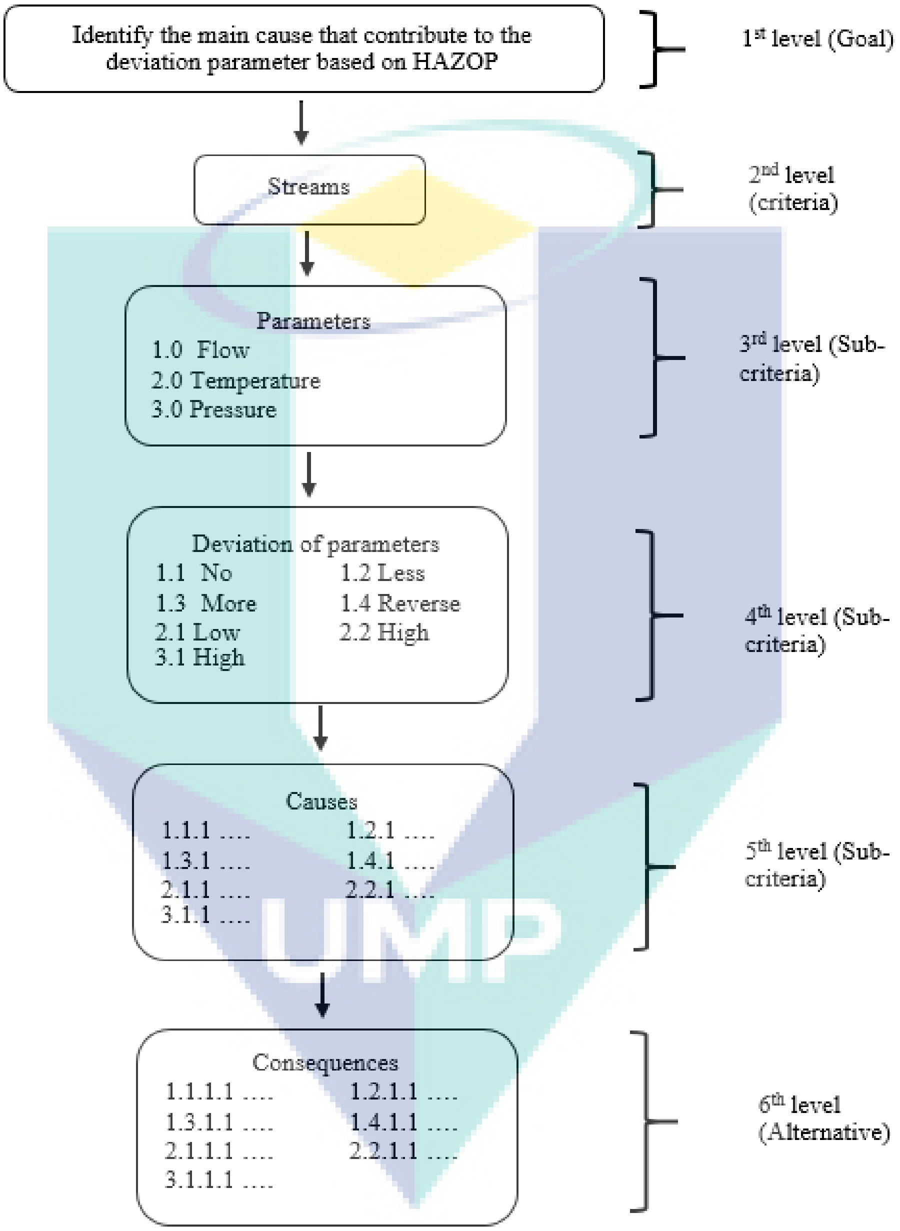


Figure 5.2. General hierarchical problem decomposition guideline in HAZOP-AHP

ratio-scale. For example, if F1 is strongly more dominant over F2, then a12 = 5. The interpretation of all the numerical judgments of the (1-9) scale is given in Table 5.1.

5.3.5 Weight ranking and consistency test

It has been stated that each Aij is the ratio of the two weights wi and wj. Now, if we multiply A by the weight vector w from the right, we get

$$\mathbf{A} \cdot w = n \cdot w \tag{5.2}$$

where n is the order of the matrix, i.e., the number of factors compared. So, we can recover the weight vector w from (2), provided $(\mathbf{A}\text{-}nI)w = 0$ has non-trivial solution, i.e., —A-nI— = 0, i.e., n is the eigenvalue of A. We also note that which is known as cardinal consistency relation. If all the elements of **A** satisfy this relation, then we say that the matrix is consistent, otherwise it is inconsistent. In reality, especially within the framework of the AHP, the matrix **A** is hardly consistent. In the inconsistent case, Equation 5.2 becomes

$$\mathbf{A}' \cdot w' = \lambda_{max} \cdot w' \tag{5.3}$$

where λ_{max} max is the largest eigenvalue of A'. Here the matrix A has been perturbed to A' and the consistency relation is violated at least once. For simplicity, the primes are omitted in the following notations and expression. To find out the weights, at first we determine the largest eigenvalue λ_{max} of A. Then the weights wis are determined by solving the following system of linear simultaneous equations:

$$w_i = \frac{1}{\lambda_{max}} \sum_{j=1}^{n} a_{ij} w_j, i = 1, 2, ..., n \tag{5.4}$$

For uniqueness, we normalize the set of weights such that . In practice, Expert Choice software is used to compute the weights from the pairwise comparison matrices (Islam, 2003).

These final numbers show an approximation of the relative priorities for the elements being compared with respect to its upper level criteria (eigenvector). These calculations can be done easily using spreadsheet either manually or using the eigenvector method. Other tools such as Super Decisions can also be used to calculate the weights. Next, check the consistency of the judgement by using Principle Eigen Value, . Eigen value is obtained from the summation of products between each element of eigenvector and the sum of reciprocal matrix column. The Consistency Index (CI) is defined as (Xia & Wu, 2007):

$$CI = \frac{\lambda max^{-n}}{n-1} \tag{5.5}$$

To overcome the order dependency of CI, the value of CI is then compared with the appropriate CI which is known as Random Consistency Index (RI). The term was defined as the expected value of the CI corresponding to the order of matrices. The average RI is shown as Table 5.3 below.

Table 5.3

Random Consistency Index, RI (Aguaron & Jimenez, 2003).

n	3	4	5	6	7	8	9	10	11	12	13	14	15	16
RI(n)	0.525	0.882	1.115	1.252	1.341	1.404	1.452	1.484	1.513	1.535	1.555	1.570	1.583	1.595
k(n)	3.147	3.526	3.717	3.755	3.755	3.744	3.733	3.709	3.698	3.685	3.674	3.663	3.646	3.646

Then, Consistency Ratio (CR) is proposed to compare between the CI and the RI using the following formula:

$$CI = \frac{CI}{RI} \tag{5.6}$$

If the value of CR is smaller than or equal to 10%, the inconsistency is acceptable. If the CR is greater than 10%, comparison matrix must be repeated (Sumi & Kabir, 2010).

5.3.6 Evaluation of the overall hazards ranking

The selection of best alternatives of the consequences (the overall ranking, OR) relies on the combination of multiplication of each priority vector by the parents priority vector as shown in the formula below (Saaty, 1990):

$$OR = p_A \times p_{S1} \times p_{S2} \times p_{S3} \times p_C \tag{5.7}$$

Where p_A = priority vector of alternative, p_S = priority vector of sub-criteria(s) and p_C = priority vector of criteria.

5.3.7 AHP-HAZOP results analysis

The final step in this methodology is analysis of the outcome from the AHP-HAZOP assessment. Since the analysis include quantitative valuation, the selection of outcomes can be easily ranked and prioritize. For example, ranking the overall hazards or nodes and its corresponding actions. In this way, engineers or assessors could focus their efforts on more important or significant hazards especially when safety aspect and the associated cost are considered.

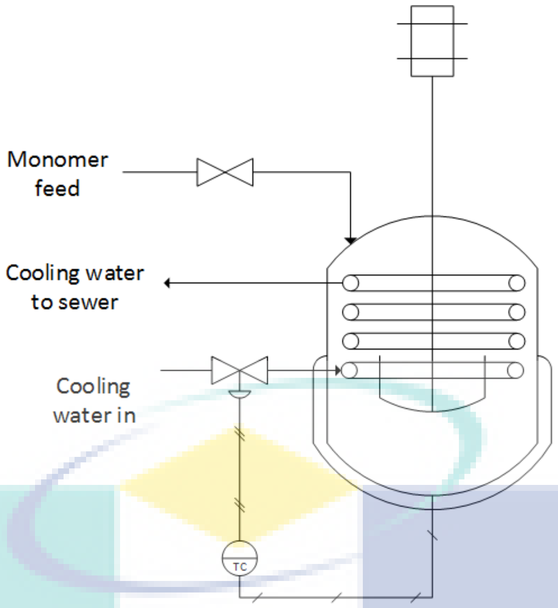


Figure 5.3. Temperature control of an exothermic reactor (Crowl & Louvar, 1990)

5.4 Application to a simple reactor case study

Figure 5.3 shows a classic example of HAZOP analysis in Chemical Process Safety: Fundamentals with Applications by Crowl and Louvar (1990). The reactor system is an exothermic reactor. A cooling system is provided to remove the excess energy of the reaction. In the event when the cooling function fails the temperature of the reactor would increase. This would lead to an increase in the reaction rate leading to additional energy release. The results could be runaway reaction with pressures exceeding the bursting pressure (maximum designated pressure) of the reactor. The corresponding HAZOP analysis for this system is shown in Table 5.4. Note that only LESS FLOW is considered in this example.

Table 5.4

Selected HAZOP study on cooling flow (Crowl & Louvar, 1990)

Guideword	Deviation	Possible causes	Consequences	Action
FLOW	LESS	1. Partially plugged cooling line 2. Partial water source failure 3. Control valve fails to respond	- Diminished cooling - Possible runaway	- Install back-up control valves, or manual bypass valve. - Install back-up controller. - Install control valve that fails open. - Install high temperature alarm to alert operator. - Install filters to prevent debris from entering line. - Install back-up cooling water source. - Install cooling water flow meter and low flow alarm.

After the HAZOP analysis table is constructed, the next step is to decompose the problem into a hierarchical form. Figure 5.4 shows the hierarchical problem decomposition. The analysis goal is to identify the main causes that affects the reactor operation and safety. It is then followed by the second level that represents the boundary analysis node which is in this case, the cooling flow. The third level is the corresponding process parameter which is FLOW. The fourth level is the deviation parameter which is LESS. The fifth level is the causes which indicate condition that gives rise to the deviation of the parameters (e.g. control valve fails to respond). Finally at the last level of the hierarchy, are the consequences anticipated due to the deviation of the parameter (e.g. diminished cooling). Each level is tagged with a unique identification number based on multilevel numbering system to facilitate the activity tracking.

The next step is to construct a pairwise comparison matrix table. Table 5.5 shows the matrix table and pairwise comparison is performed at every level. The comparison process can be aided using series of questions that relate the relationship of the compared elements and the control criterion. For example, the question that may be asked is For the cooling flow, how much important is partially plugged cooling line compared to partial water source failure when investigating the main cause that affects the reactor. In this question, partially plugged cooling line acts as the base criterion while partial water source failure is the paired criteria and investigating the main cause that affects the reactor is the control criterion. The scaling factor is based on the guideline in Table 1 and once the comparison matrix is completed, the priority value can be calculated. Setting the scaling factor during the construction of pairwise comparison matrix table can be done by the HAZOP team members.

The priority value is calculated using Equations 5.2 to 5.5. Using Equation 5.2, the sum of reciprocal of column j (paired criterion) is calculated giving the following result in Table 5.6.

The normalized relative weights are shown below using Equation 5.3 by di-

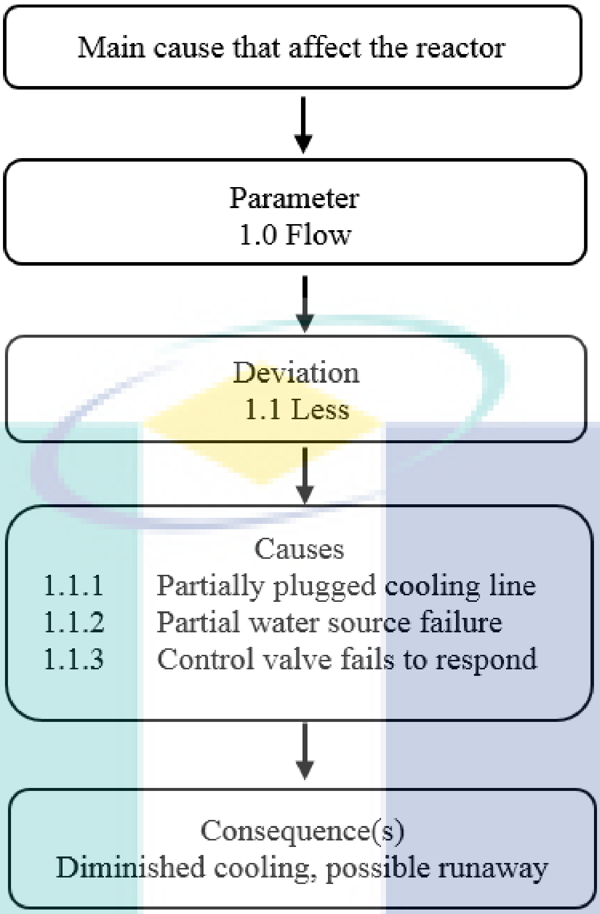


Figure 5.4. Hierarchical problem decomposition for the reactor system

Table 5.5
Pairwise comparison matrix

Causes	1.1.1	1.1.2	1.1.3
1.1.1	1	2	0.25
1.1.2	0.5	1	0.2
1.1.3	4	5	1
Sum of reciprocal column	5.5	8	1.45

Table 5.6
Results

1.1.1	1.1.2	1.1.3	
Sum of reciprocal column	5.5	8	1.45

Table 5.7
Norm. relative weights

Causes	1.1.1	1.1.2	1.1.3
1.1.1	0.182	0.25	0.172
1.1.2	0.091	0.125	0.138
1.1.3	0.727	0.625	0.690

viding each element in a column by the sum of its respective column to give results in Table 5.7.

The priorities are calculated using Equations 5.4 and 5.5 which give the following value.

$$\begin{bmatrix} 0.182 & 0.25 & 0.172 \\ 0.091 & 0.125 & 0.138 \\ 0.727 & 0.625 & 0.690 \end{bmatrix} = \begin{bmatrix} 0.604 \\ 0.354 \\ 2.042 \end{bmatrix}$$

Eigen vector =

$$\begin{bmatrix} 0.2014 \\ 0.1179 \\ 0.6806 \end{bmatrix}$$

The Eigen vector value shows us the prioritization value. The consistency ratio, in this example is 3.6% which is less than 10%. Thus, the comparison is acceptable. The calculation above could be done easily in spreadsheets such as Excel. Table 6 shows the enhanced version of HAZOP analysis table incorporating the AHP analysis for hazard prioritization. From this we can see that activity 1.1.3 (Control valves failed to response) is the most significant causes of hazard in the reactor, this is followed by 1.1.1 (Partially plugged cooling line) and 1.1.2 (Partial water sources failure). By identifying the most significant causes, engineers could take appropriate action associated with the prioritized cause for an inherently safer process design.

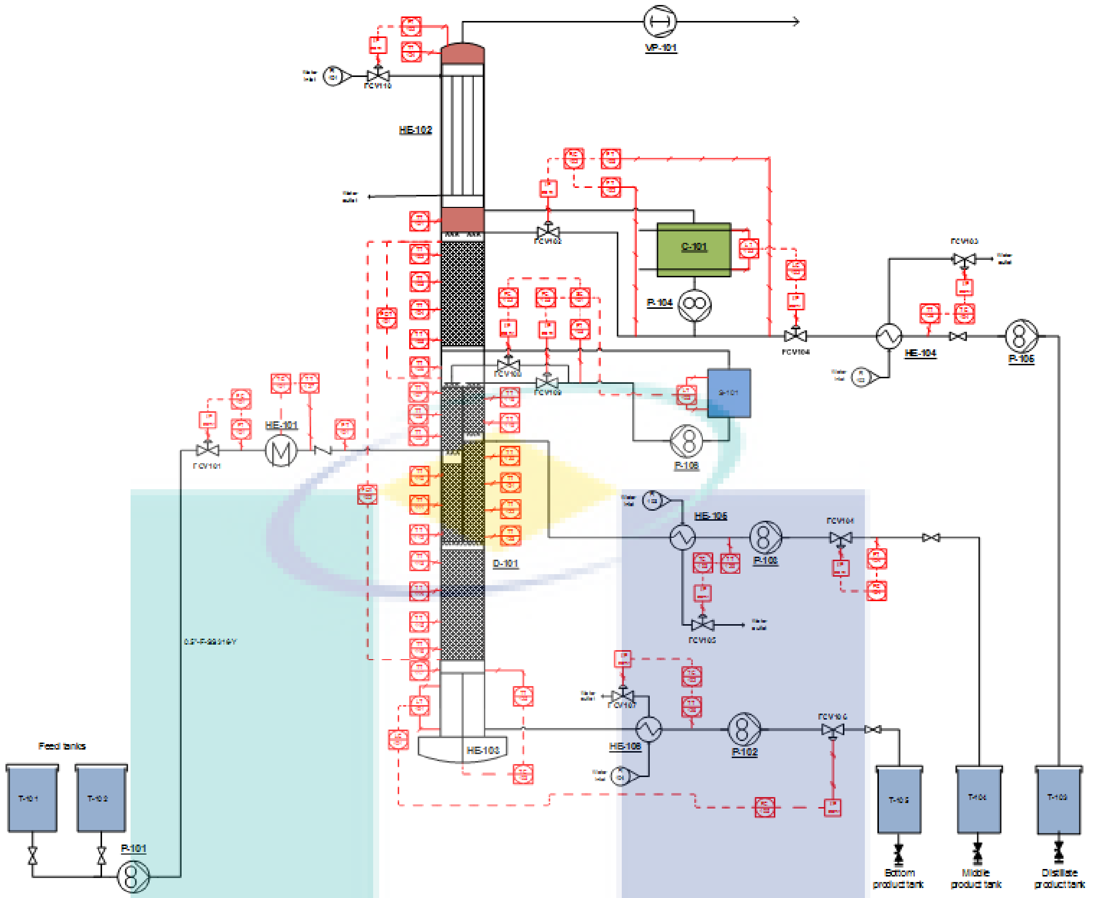


Figure 5.5. P&ID of the DWC pilot plant.

5.5 Application to DWC pilot plant for fatty acid fractionation

DWC is a single shell, fully thermally coupled distillation column which is able to separate mixtures of three or more components into high purity products (Dejanovi et al., 2010). In the Faculty of Chemical and Natural Resources Engineering, Universiti Malaysia Pahang, there is a plan to build DWC pilot plant for fatty acid fractionation. The column is designed with a height of 14 m high and diameter of 15 cm. It is designed with feed flowrate of between 3 to 5 kg/h and operated under vacuum condition between 10-30 mbar. This vacuum condition is needed so that the operating temperature of the column is lower than 270 oC to avoid product degradation. Since fatty acid is highly corrosive, a low carbon stainless steel material e.g. 317SS or 904L is used which able to withstand high corrosive material. Figure 5.5 shows the P&ID of the pilot plant.

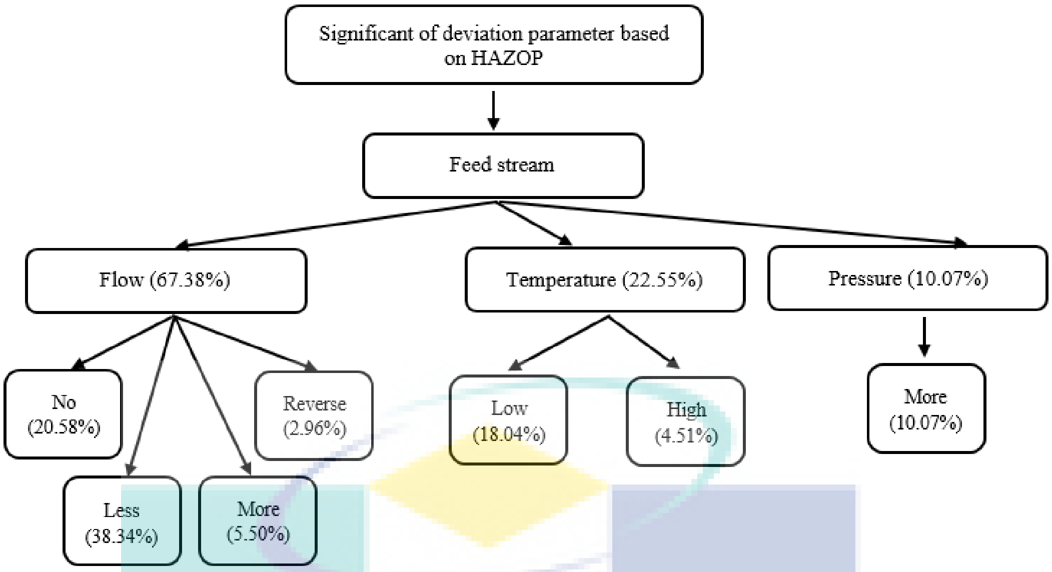


Figure 5.6. Problem hierarchy for the feed stream and its corresponding weights.

Prior to installation, a HAZOP analysis was performed by our HAZOP team members through several meetings. The team members comprises of lecturers, post graduate students and undergraduate students. The main tasks of the team not only to conduct HAZOP analysis but also set the scaling factor for pairwise comparison. In this section, the application of HAZOP-AHP to determine important hazards in the proposed pilot plant is discussed in detail. A Super Decision software (SDS) is used for weights calculation. SDS is one of the AHP tools used due to its powerful and flexible features in making multi-criteria decision (Baby, 2013). Typically SDS consists of four steps: 1) building a hierarchy of the objective or goal, 2) entering the alternatives, 3) comparing the elements and finally, 4) synthesizing the result. Because of space limitation, the case study is discussed on the FEED stream HAZOP analysis only. Figure 5.6 below shows the problem hierarchy for the feed stream.

A pairwise comparison is performed at every level. The comparison process can be aided using series of questions that relates the relationship of the compared elements and the control criterion. For example, in the third level, the question that may be asked is How much important is feed flowrate compared to temperature when performing HAZOP at the feed stream. In this question, feed flowrate acts as the base criterion while temperature is the paired criteria and performing HAZOP

Table 5.8
Priorities result based on parameters

	Parameter	Priorities
1.0	Flow	0.67381
2.0	Temperature	0.22554
3.0	Pressure	0.10065

at the feed stream is the control criterion. For the third level, Table 5.8 shows that flow is identified as the most important parameter in contributing to hazards with relative weight of 0.67381. It is followed by temperature and pressure which have relative weight of 0.22554 and 0.10065, respectively. These values indicate that flow is the most anticipated parameter to be considered in conjunction with HAZOP analysis for the feed stream followed by temperature and pressure. Note that, weights assignment is subjective (Othman et al., 2010) nevertheless to have a meaningful and justifiable comparison, justification and team-work participation among decision makers are very important.

Based on the parameter deviations (fourth level), the results are depicted in Figure 5.9. It shows that for flow parameter, LESS flow (1.2) is anticipated to cause the highest process deviation, with relative weight of 0.38341, followed by NO flow (1.1) which has 0.20578 of relative weight. On the other hand, MORE flow (1.3) and REVERSE flow (1.4) have much lower deviation values of relative weights in the range of 0.029-0.055. For temperature parameter, LOW temperature (2.1) is predicted to cause the highest process deviation with relative weight of 0.18043 and HIGH temperature (2.2) has a lower relative weight of 0.04510. For pressure on the other hand, MORE pressure (3.1) has a relative weight of 0.10065. Overall, based on the seven parameters, deviation of LESS flow is crucial compared to the other deviations. This is followed by NO flow, LOW temperature and MORE pressure. Meanwhile the parameters that may cause the least process deviation are MORE flow, REVERSE flow and HIGH temperature.

Table 5.10 shows the synthesized priorities for the consequences (alternatives) of DWC in feed stream. The Normals column presents the results in the form of the

Table 5.9

Priorities result based on parameter deviations

	Parameter	Priorities
1.1	No	0.20578
1.2	Less	0.38341
1.3	More	0.05502
1.4	Reverse	0.02959
2.1	Low	0.18043
2.2	High	0.04510
3.1	More	0.10065

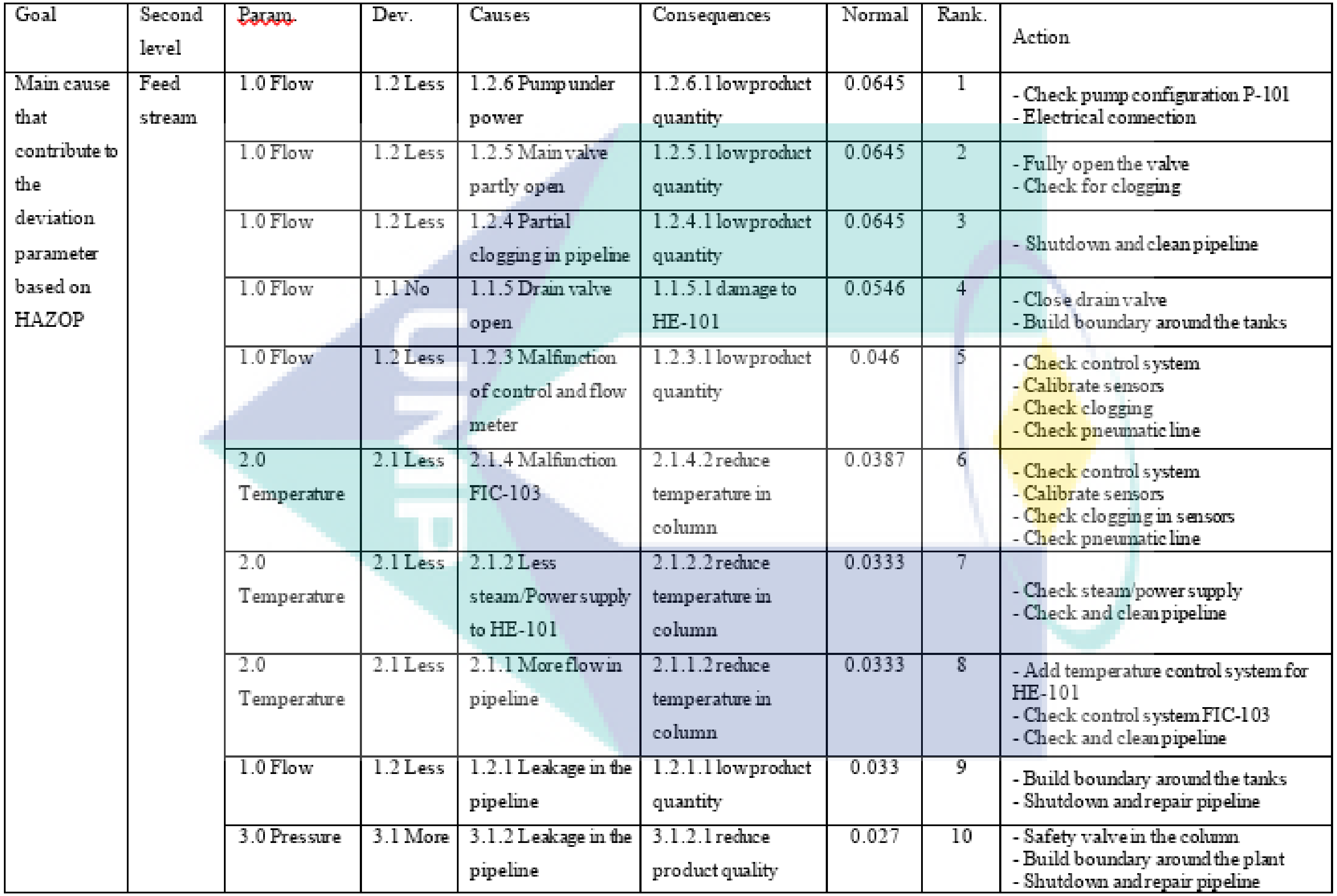
priorities. The Ideals column is obtained from Normals column by dividing each of the value with the largest value in the column, so that the best choice has a priority of 1. Based on the SDS synthesis, the consequence of low product quantity (1.2.6.1) has the highest priority of 0.0645. The lowest priority is referred to damages to pump P-101 (1.1.6.1) with Normals value of 0.0021. Synthesizing for other priorities are the same for distillate and reflux stream, middle stream and bottom stream.

Figure 5.7 up to Figure 5.10 list the top 10 ranking of the consequences (alternatives) which cover all streams in the DWC system. Nevertheless, since the description of the results are the same for all streams, we will be focusing on the feed stream for demonstration in this paper. Table 7 below refers to the overall consequences rankings that probably rising up in DWC system if deviation of parameters occurred. The first three ranking, which are low in product quantity (1.2.6.1, 1.2.5.1 and 1.2.4.1) have the same priorities, indicating that they are equally important with a value of 0.0645 each. This is followed by the fourth and fifth rankings, which are damages to HE-101(1.1.5.1) and low product quantity (1.2.3.1), with the priority value of 0.0546 and 0.046, respectively. The other rankings are in the range of 0.020 to 0.039. All the consistency ratios are below than 10%, thus the pairwise judgments that have been made can be trusted.

Table 5.10

The weight of the consequences from SDS

	Alternatives	Ideals	Normals
1.1.1.1	damage to pump P-101	0.0319	0.0021
1.1.1.2	damage to HE-101	0.1594	0.0103
1.1.2.1	damage to HE-101	0.353	0.0228
1.1.2.2	excessive heat in column	0.0706	0.0046
1.1.2.3	column dry	0.0706	0.0046
1.1.3.1	damage to pump P-101	0.0319	0.0021
1.1.3.2	damage to HE-101	0.1594	0.0103
1.1.4.1	damage to HE-101	0.353	0.0228
1.1.4.2	excessive heat in column	0.0706	0.0046
1.1.4.3	column dry	0.0706	0.0046
1.1.5.1	damage to HE-101	0.8473	0.0546
1.1.5.2	excessive heat in column	0.2708	0.0175
1.1.5.3	column dry	0.2708	0.0175
1.1.5.4	raw material spillage	0.0804	0.0052
1.1.5.5	fire	0.0804	0.0052
1.1.5.6	corrosion	0.0804	0.0052
1.1.6.1	damage to pump P-101	0.0319	0.0021
1.1.6.2	damage to HE-101	0.1594	0.0103
1.2.1.1	low product quantity	0.5124	0.033
1.2.1.2	corrosion	0.0854	0.0055
1.2.1.3	fire	0.0854	0.0055
1.2.1.4	spillage	0.0854	0.0055
1.2.2.1	low product quantity	0.2352	0.0152
1.2.2.2	spillage	0.0392	0.0025
1.2.2.3	corrosion	0.0392	0.0025
1.2.2.4	fire	0.0392	0.0025
1.2.3.1	low product quantity	0.7133	0.046
1.2.3.2	excessive heat in column	0.1783	0.0115
1.2.4.1	low product quantity	1	0.0645
1.2.4.2	excessive heat in column	0.25	0.0161
1.2.5.1	low product quantity	1	0.0645
1.2.5.2	excessive heat in column	0.25	0.0161
1.2.6.1	low product quantity	1	0.0645
1.2.6.2	excessive heat in column	0.25	0.0161
1.2.7.1	reduce pump flow P-101	0.037	0.0024
1.2.7.2	more pump power P-101 consumption	0.037	0.0024
1.2.7.3	back flow in pipeline	0.037	0.0024
1.2.7.4	condenser HE-102 failure	0.037	0.0024
1.2.7.5	clogging in product pipeline	0.037	0.0024
1.3.1.1	reduce product quality	0.1423	0.0092



Goal	Second level	Param.	Dev.	Causes	Consequences	Normal	Rank.	Action
Main cause that contribute to the deviation parameter based on HAZOP	Feed stream	1.0 Flow	1.2 Less	1.2.6 Pump under power	1.2.6.1 low product quantity	0.0645	1	- Check pump configuration P-101 - Electrical connection
		1.0 Flow	1.2 Less	1.2.5 Main valve partly open	1.2.5.1 low product quantity	0.0645	2	- Fully open the valve - Check for clogging
		1.0 Flow	1.2 Less	1.2.4 Partial clogging in pipeline	1.2.4.1 low product quantity	0.0645	3	- Shutdown and clean pipeline
		1.0 Flow	1.1 No	1.1.5 Drain valve open	1.1.5.1 damage to HE-101	0.0546	4	- Close drain valve - Build boundary around the tanks
		1.0 Flow	1.2 Less	1.2.3 Malfunction of control and flow meter	1.2.3.1 low product quantity	0.046	5	- Check control system - Calibrate sensors - Check clogging - Check pneumatic line
		2.0 Temperature	2.1 Less	2.1.4 Malfunction FIC-103	2.1.4.2 reduce temperature in column	0.0387	6	- Check control system - Calibrate sensors - Check clogging in sensors - Check pneumatic line
		2.0 Temperature	2.1 Less	2.1.2 Less steam/Power supply to HE-101	2.1.2.2 reduce temperature in column	0.0333	7	- Check steam/power supply - Check and clean pipeline
		2.0 Temperature	2.1 Less	2.1.1 More flow in pipeline	2.1.1.2 reduce temperature in column	0.0333	8	- Add temperature control system for HE-101 - Check control system FIC-103 - Check and clean pipeline
		1.0 Flow	1.2 Less	1.2.1 Leakage in the pipeline	1.2.1.1 low product quantity	0.033	9	- Build boundary around the tanks - Shutdown and repair pipeline
		3.0 Pressure	3.1 More	3.1.2 Leakage in the pipeline	3.1.2.1 reduce product quality	0.027	10	- Safety valve in the column - Build boundary around the plant - Shutdown and repair pipeline

Figure 5.7. Top 10 ranking of the consequences (alternatives) in feed stream

Goal	Second level	Param.	Dev.	Causes	Consequences	Normal	Rank.	Action
Main cause that contribute to the deviation parameter based on HAZOP	Distillate & Reflux Stream	1.0 Flow	1.2 Less	1.2.2 Drain valve C-101 partly open	1.2.2.1 lowproduct quantity	0.0597	1	- Close the drain valve
		1.0 Flow	1.2 Less	1.2.1 Leakage in the pipeline	1.2.1.1 lowproduct quantity	0.0597	2	- Build boundary around the tanks - Shutdown and repair pipeline
		2.0 Temperature	2.1 Low	2.1.3 Excessive cooling in HE-102&HE-104	2.1.3.5 column flooding	0.0389	3	- Check controller TC-101 & T-102 - Check FI-101 & F-102 - Check and clean pipeline - Clean and repair HE-102 & HE-104
		3.0 Pressure	3.1 More	3.1.2 Leakage in the pipeline	3.1.2.1 reduceproduct quality	0.0251	4	- Safety valve in the column - Build boundary around the plant - Shutdown and repair pipeline
		3.0 Pressure	3.1 More	3.1.4 Column dry due to excessive vapour load	3.1.4.1 reduceproduct quality	0.022	5	- Safety valve in the column - Check reboiler operation (controller LIC-103 and power)
		1.0 Flow	1.1 No	1.1.3 Valve along pipeline close	1.1.3.5 column dry	0.0216	6	- Open the valve - Examine check valve orientation - Install pump interlock
		1.0 Flow	1.1 No	1.1.3 Valve along pipeline close	1.1.3.1 damage to pump P-103,P-102,P-104	0.0216	7	- Open the valve - Examine check valve orientation - Install pump interlock
		1.0 Flow	1.1 No	1.1.3 Valve along pipeline close	1.1.3.4 excessive heat in column	0.0216	8	- Open the valve - Examine check valve orientation - Install pump interlock
		1.0 Flow	1.1 No	1.1.3 Valve along pipeline close	1.1.3.3 no reflux to column	0.0216	9	- Open the valve - Examine check valve orientation - Install pump interlock
		2.0 Temperature	2.1 Low	2.1.3 Excessive cooling in HE-102&HE-104	2.1.3.3 clogging in pipeline	0.0216	10	- Check controller TC-101 & T-102 - Check FI-101 & F-102 - Check and clean pipeline - Clean and repair HE-102 & HE-104

Figure 5.8. Top 10 ranking of the consequences (alternatives) in distillate and reflux stream

Goal	Second level	Param.	Dev.	Causes	Consequences	Normal	Rank.	Action
Main cause that contribute to the deviation parameter based on HAZOP	Middle stream	1.0 Flow	1.2 Less	1.2.1 Leakage in the pipeline	1.2.1.1 lowproduct quantity	0.0842	1	- Build boundary around the tanks - Shutdown and repair pipeline
		1.0 Flow	1.2 Less	1.2.2 Sample valve partly open	1.2.2.1 lowproduct quantity	0.0840	2	- Close the drain valve
		2.0 Temperature	2.1 Low	2.1.2 Raining inside column	2.1.2.2 clogging in pipeline	0.0271	3	- Check temperature control system for TC-101** - Check and clean pipeline - Check reboiler - Check reflux flow
		2.0 Temperature	2.1 Low	2.1.1 Low vapor load	2.1.1.2 clogging in pipeline	0.0271	4	- Introduce temperature control system for TC-101**(at reboiler) - Check and clean pipeline - Check reboiler
		1.0 Flow	1.2 Less	1.2.6 Pump under power	1.2.6.1 lowproduct quantity	0.0253	5	- Check pump configuration P-105 - Check electrical connection
		1.0 Flow	1.2 Less	1.2.5 Valve along pipeline partly open	1.2.5.1 lowproduct quantity	0.0253	6	- Fully open the valve - Check for clogging
		1.0 Flow	1.2 Less	1.2.4 Partial clogging in pipeline	1.2.4.1 lowproduct quantity	0.0253	7	- Shutdown and clean pipeline
		1.0 Flow	1.2 Less	1.2.3 Malfunction of control FIC-101	1.2.3.1 lowproduct quantity	0.0253	8	- Check control system - Calibrate sensors - Check clogging - Check pneumatic line
		1.0 Flow	1.1 No	1.1.2 Pump P-105 failure	1.1.2.1 reduce product quality & quantity	0.0213	9	- Introduce valve between pump and introduce bypass - Isolate and repair the pump - Check electric connection and configuration
		3.0 Pressure	3.1 High	3.1.4 Column dry due to excessive vapor load	3.1.4.1 reduce product quality	0.0201	10	- Safety valve in the column - Check reboiler operation (controller LIC-103 and power)

Figure 5.9. Top 10 ranking of the consequences (alternatives) in middle stream

Goal	Second level	Param.	Dev.	Causes	Consequences	Normal	Rank.	Action
Main cause that contribute to the deviation parameter based on HAZOP	Bottom Stream	1.0 Flow	1.2 Less	1.2.2 Sample valve partly open	1.2.2.1 lowproduct quantity	0.0643	1	- Close the drain valve
		1.0 Flow	1.2 Less	1.2.1 Leakage in the pipeline	1.2.1.1 lowproduct quantity	0.0643	2	- Build boundary around the tanks - Shutdown and repair pipeline
		1.0 Flow	1.2 Less	1.2.6 Excessive heating in reboiler	1.2.6.2 build-up pressure along pipeline	0.0482	3	- Install check valve - Check temperature controller for HE-103 - Check utility
		2.0 Temperature	2.1 Low	2.1.2 Excessive cooling in HE-102	2.1.2.2 clogging in pipeline	0.0331	4	- Check temperature control system for TC-102 - Check and clean pipeline - Check HE-102 - Check reflux flow
		1.0 Flow	1.1 No	1.1.2 Pump P-106 failure	1.1.2.1 reduce product quality & quantity	0.0305	5	- Introduce valve between pump and introduce bypass - Isolate and repair the pump - Check electric connection and configuration
		1.0 Flow	1.1 No	1.1.3 Valve along pipeline close	1.1.3.3 reduce product quality & quantity	0.0264	6	- Open the valve - Examine check valve orientation - Install pump interlock
		1.0 Flow	1.1 No	1.1.3 Valve along pipeline close	1.1.3.1 damage to pump P-106	0.0264	7	- Open the valve - Examine check valve orientation - Install pump interlock
		1.0 Flow	1.2 Less	1.2.5 Valve along pipeline partly open	1.2.5.1 lowproduct quantity	0.0229	8	- Fully open the valve - Check for clogging
		1.0 Flow	1.2 Less	1.2.4 Partial clogging in pipeline	1.2.4.1 lowproduct quantity	0.0229	9	- Shutdown and clean pipeline
		1.0 Flow	1.2 Less	1.2.3 Malfunction of control FCV-103	1.2.3.1 lowproduct quantity	0.0229	10	- Check control system - Calibrate sensors - Check clogging - Check pneumatic line

Figure 5.10. Top 10 ranking of the consequences (alternatives) in bottom stream

Based on the first three rankings, all the consequences are ranked with respect to the upper control criteria. It shows that less flow of raw material inside the feed stream might lower the quantity of fatty acid production, due to the pump operation is under power, main valve is partly open and there is a partial clogging inside the pipeline. By referring to the fourth level of ranking, it is stated that if no flow is occurred, there is a higher probability that HE-101 will damage due to the opening of drain valve in the feed stream. While in the fifth ranking, the quantity of fatty acid will be smaller, due to less flow inside the stream caused from the malfunction of control and flow meter.

Based on the priorities ranking that have been listed above, it can be concluded that pump under power, main valve partly open, and partial clogging in the pipeline are the main causes that lead to deviation of less flow, compared to other causes. These rankings allow the project team to identify the crucial causes that need to be monitored before DWC is fully operated. Thus, early precaution steps to control the risk can be taken in a stepwise manner.

5.6 Conclusion

In this paper a novel approach in prioritizing hazards identified in HAZOP using AHP is introduced. The method has been applied to a simple reactor and DWC pilot plant. The results show that, the proposed method is able to identify and rank the most significant hazards among the identified long list of hazards. However, weights assignment during the pairwise comparison step is subjected to individual preference (assessor) and thus, should be bound by a good team-work participation. In addition, rank reversal phenomenon could also perturb the ranking which usually caused by the addition or deletion of an alternative. An application to final HAZOP results could minimize the rank reversal effect. Nevertheless, by using this approach as a decision making tool, project team will be able to prioritize any action to plant modification, retrofitting or construction within the available resources constraints.

Thus, early precaution steps to control the risk can be taken in a stepwise manner hence aiding towards achieving an inherently safer chemical process.

5.7 Acknowledgements

We gratefully acknowledge the financial support from the Malaysia Ministry of Education through ERGS (RDU130601) and FRGS (RDU140105) grants.

5.8 References

1. Aguaron, J., & Moreno-Jimnez, J. M. (2003). The geometric consistency index: Approximated thresholds. European Journal of Operational Research, 147(1), 137-145.

2. Aminbakhsh, S., Gunduz, M., & Sonmez, R. (2013). Safety risk assessment using analytic hierarchy process (AHP) during planning and budgeting of construction projects. Journal of safety research, 46, 99-105.

3. Baby, S. (2013). AHP Modeling for Multicriteria Decision-Making and to Optimise Strategies for Protecting Coastal Landscape Resources. International Journal of Innovation, Management and Technology, 4(2), 218-227.

4. Badri, A., Nadeau, S., & Gbodossou, A. (2012). Proposal of a risk-factor-based analytical approach for integrating occupational health and safety into project risk evaluation. Accident Analysis & Prevention, 48, 223-234.

5. Bahurmoz, A.M.A. (2003). The Analytic Hierarchy Process at Dar Al-Hekma, Saudi Arabia. Interfaces. Vol. 33, No. 4, 70-78.

6. Caputo, A. C., Pelagagge, P. M., & Salini, P. (2013). AHP-based methodology for selecting safety devices of industrial machinery. Safety science, 53, 202-218.

7. Chung, S.H., Lee, A.H.I., & Pearn, W.L. (2005). Analytic network process (ANP) approach for product mix planning in semiconductor fabricator. International Journal of Production Economics, 96:15, 36.

8. Crowl, D. A., & Louvar, J. F. (1990). Chemical process safety: fundamentals with applications. Pearson Education.

9. Dejanovi, I., Matijaevi, L., & Oluji, . (2010). Dividing wall columna breakthrough towards sustainable distilling. Chemical Engineering and Processing: Process Intensification, 49(6), 559-580.

10. Dyer, R. F., & Forman, E. H. (1992). Group decision support with the analytic hierarchy process. Decision support systems, 8(2), 99-124.

11. Hadidi, L. A., & Khater, M. A. (2015). Loss prevention in turnaround maintenance projects by selecting contractors based on safety criteria using the analytic hierarchy process (AHP). Journal of Loss Prevention in the Process Industries, 34, 115-126.

12. Islam, R. (2003). The Analytic Hierarchy Process: N Effective Multi-criteria Decision Making Tool. International Islamic University.

13. Millet, I. & Saaty, T. (2000). On the relativity of relative measures accommodating both rank preservation and rank reversals in the AHP. Eur. Oper. Res., 121(1) 205-212.

14. Narayanan, D., Zhang, Y., & Mannan, M.S. (2007). Engineering for sustainable development in biodiesel production. Process Safety and Environmental Protection, 85:349-359.

15. Othman, M. R., Repke, J. U., Wozny, G., & Huang, Y. (2010). A modular approach to sustainability assessment and decision support in chemical process design. Industrial & Engineering Chemistry Research, 49(17), 7870-7881.

16. Othman, M. R., Hady, L., Repke, J. U., & Wozny, G. (2012). Introducing sustainability assessment and selection (SAS) into chemical engineering education. Education for Chemical Engineers, 7(3), e118-e124.

17. Rausand, M. (2005). Hazard and Operability Study. System Reability Theory, 1-44.

18. Robson, L. S., Clarke, J. A., Cullen, K., Bielecky, A., Severin, C., Bigelow, P. L. & Mahood, Q. (2007). The effectiveness of occupational health and safety management system interventions: a systematic review. Safety Science, 45(3), 329-353.

19. Rossing, N. L., Lind, M., Jensen, N., & Jrgensen, S. B. (2010). A functional HAZOP methodology. Computers & chemical engineering, 34(2), 244-253.

20. Saaty, T. L. (1990). How to make a decision: the analytic hierarchy process. European journal of operational research, 48(1), 9-26.

21. Saaty. T. L. (1980). The Analytic Hierarchy Process. McGraw-Hill, New York.

22. Saaty. T. L. (2001). The Analytic Network Process: Decision Making with Dependence and Feedback. RWS Publications, Pittsburgh, 2nd edition.

23. Saaty, T. & Vergas, L. (1993). Experiments on rank preservation and reversal in relative measurement. Math. Compul. Modeling, 17(4/5), 13-18.

24. Sumi, R. S., & Kabir, G. (2010). Analytical hierarchy process for higher effectiveness of buyer decision process. Global Journal of Management and Business Research, 10(2).

25. Tan, R. R., Aviso, K. B., Huelgas, A. P., & Promentilla, M. A. B. (2014). Fuzzy AHP approach to selection problems in process engineering involving quantitative and qualitative aspects. Process Safety and Environmental Protection, 92(5), 467-475.

26. Taylan, O., Bafail, A. O., Abdulaal, R. M., & Kabli, M. R. (2014). Construction projects selection and risk assessment by fuzzy AHP and fuzzy TOPSIS methodologies. Applied Soft Computing, 17, 105-116.

27. Venkatasubramanian, V., Zhao, J., & Viswanathan, S. (2000). Intelligent systems for HAZOP analysis of complex process plants. Computers & Chemical Engineering, 24(9), 2291-2302.

28. Xia, W., & Wu, Z. (2007). Supplier selection with multiple criteria in volume discount environments. Omega, 35(5), 494-504.

CHAPTER 6

APPLICATION OF DIVIDING WALL COLUMN FOR IMPROVED FRACTIONATION OF OLEOCHEMICAL PRODUCTS FROM MODELLING WORK TO PILOT PLANT

The paper has been selected as an invitation to POMREQ 2017 conference in Kuala Lumpur. Below are the authors:-

- Mohamad Rizza bin Othman, Process Systems Engineering Research Group, Universiti Malaysia Pahang, 26300 Gambang, Malaysia
- Noorlisa Harun, Process Systems Engineering Research Group, Universiti Malaysia Pahang, 26300 Gambang, Malaysia
- Wan Hanisah Wan Ibrahim, Department of chemical Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
- Mohd Yusri Mohd Yunus, Process Systems Engineering Research Group, Universiti Malaysia Pahang, 26300 Gambang, Malaysia

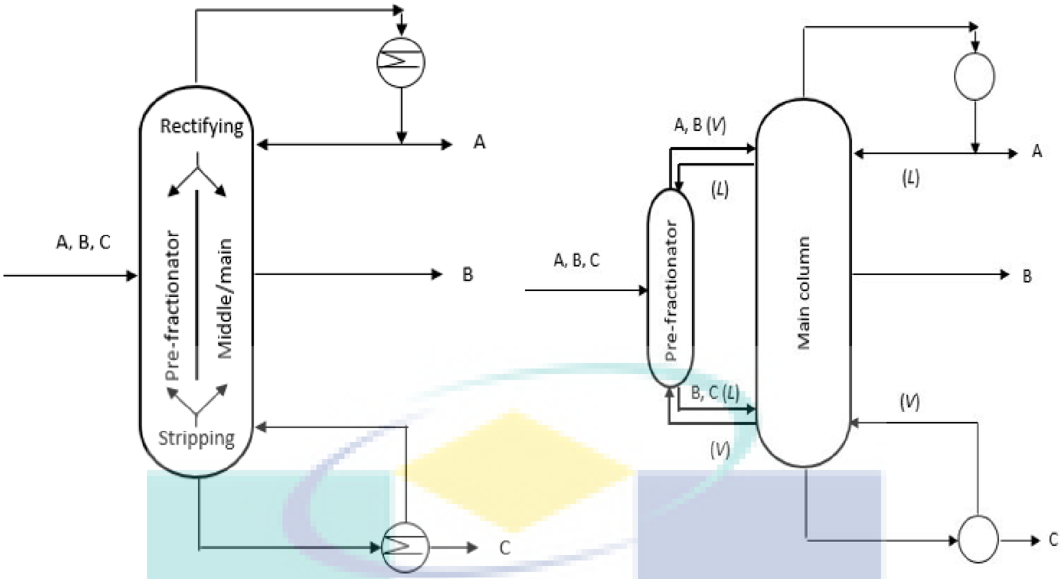
6.1 Abstract

Dividing wall column (DWC) have been successfully applied in various chemical and petrochemical processes with proven cost and energy reduction. Typically, fractionation of oleochemical products in Malaysia used conventional distillation

columns (CDCs). None so far, imply DWC for its fractionation process mainly due to the lack of understanding of its design, control and operation. In this presentation, we share our experience in developing a DWC pilot plant for oleochemical fractionation which is currently being built at Universiti Malaysia Pahang. The pilot plant is design based on borosilicate glass components and operated under vacuum condition with temperature up to 200oC. The feed capacity is in the range of 5 - 7 kg/h for different type of oleochemical products. In this paper we will share our knowledge and experience from concept and feasibility study and pilot plant development. In addition, future work on the operation and controllability will also be highlighted. We believe such study on a pilot plant scale is important to gain the operation experience for future industrial implementation.

6.2 Introduction

Over the years, dividing wall column (DWC) has attracted researchers and industrialists and has been successfully implemented in process industries, especially for separating ternary mixtures. It is a promising energy saving alternative for separating multi-component mixtures such as hydrocarbons, alcohols, aldehydes, ketones, acetals, amines and others. In recent developments, application of DWC has been extended to azeotropic, extractive and reactive distillation (Yildirim et al., 2011). Theoretically, DWC column configuration (see Figure 6.1a) is thermodynamically equivalent to the Petyluk column configuration (see Figure 6.1b). In terms of the stage or tray at which vapour and liquid splitting and mixing taking place, DWC differs from Petlyuk column which instead of an external pre-fractionator, a vertical wall is introduced in the middle part of DWC which create a feed preheater and draw off stream in the middle section. Such existence prevents the lateral mixing of liquid and vapour streams. Moreover, the wall also divides space in the column that prevents contamination of the side stream by the feed stream. Kaibel (1987) shows that for separation of ternary mixtures, DWC shows a good result in



(a) (b)

Figure 6.1. Separation of the ternary mixture via (a) DWC (b) Petlyuk column

terms of products quality achievement, compared to two distillation conventional arrangement method.

The oleochemical industry in Malaysia is now one of the largest in the world, with capacity representing about 20% of the world's capacity. There are currently 18 oleochemical companies in Malaysia and from our survey it is found that all oleochemical plants employ series of conventional distillation columns (CDCs) for fractionating its oleochemical products. Thus, application of DWC to oleochemical industries in Malaysia offers huge opportunity. Yildirim et al. (2011) predicted that future implementations of DWC technology will be in developing countries with emerging markets rather than in countries with established distillation networks. Unlike DWC, CDCs are well investigated and, for moderate separation tasks, could be designed and planned via models and simulations. For DWC however, the design process, modelling and simulation and technical aspects are more tedious. Possible reasons for not utilizing DWC in oleochemical industries are relatively more complex and lack of a systematic design approach. Nevertheless, with the advancement of process simulators and establishment of pilot plant, these gaps can be reduced. Therefore, detailed simulations and pilot plant scale experiments are necessary for model validation and investigation of DWC operability and controllability (Chu et al., 2011). In this paper, an overview look on the concept and feasibility study will be given along with the pilot plant development phases. In addition, several technical issues including operability and controllability will also be highlighted. With the establishment of experimental rig integrated with modelling and simulation works will help to understand the operation of DWC, enhance local experts know-how and significantly accelerate the commissioning of industrial scale application.

6.3 Design and feasibility study

6.3.1 Modelling and simulation work

In our current work, we perform a systematic step by step approach of designing and modelling DWC from simple to rigorous model. A fatty acid fractionation process were selected as a case study. The main fatty acid cuts from refined bleached deodorized (RBD) palm kernel oil (PKO) that need to be fractionated were: C6-C10 (precut, PC), C12 (light cut, LC), C14 (middle cut, MC) and C16-C18 (heavy cut, HC). Three cases were considered in the modelling work namely Case 1, Case 2 and Case 3. Case 1 and Case 2 were based on a typical four column configuration and Case 3 was based on two series of DWC as illustrated in Figure 6.2a and 6.2b respectively.

Case 1 was modelled by shortcut method using four DSTWU blocks in Aspen Plus. The results from Case 1 were then use for initial estimation in modelling of Case 2, which uses four rigorous RADFRAC blocks. The results from Case 2 were then used as initial design parameters for simulating Case 3. Each of the two DWCs in Case 3 were model using four RADFRAC block with splitters to manage the vapour and liquid distribution to both side of the dividing wall. These four-column configuration offer few advantages such as flexibility in dimensioning the column sections and suitability for dynamic simulation and control study (Dejanovi et al.,

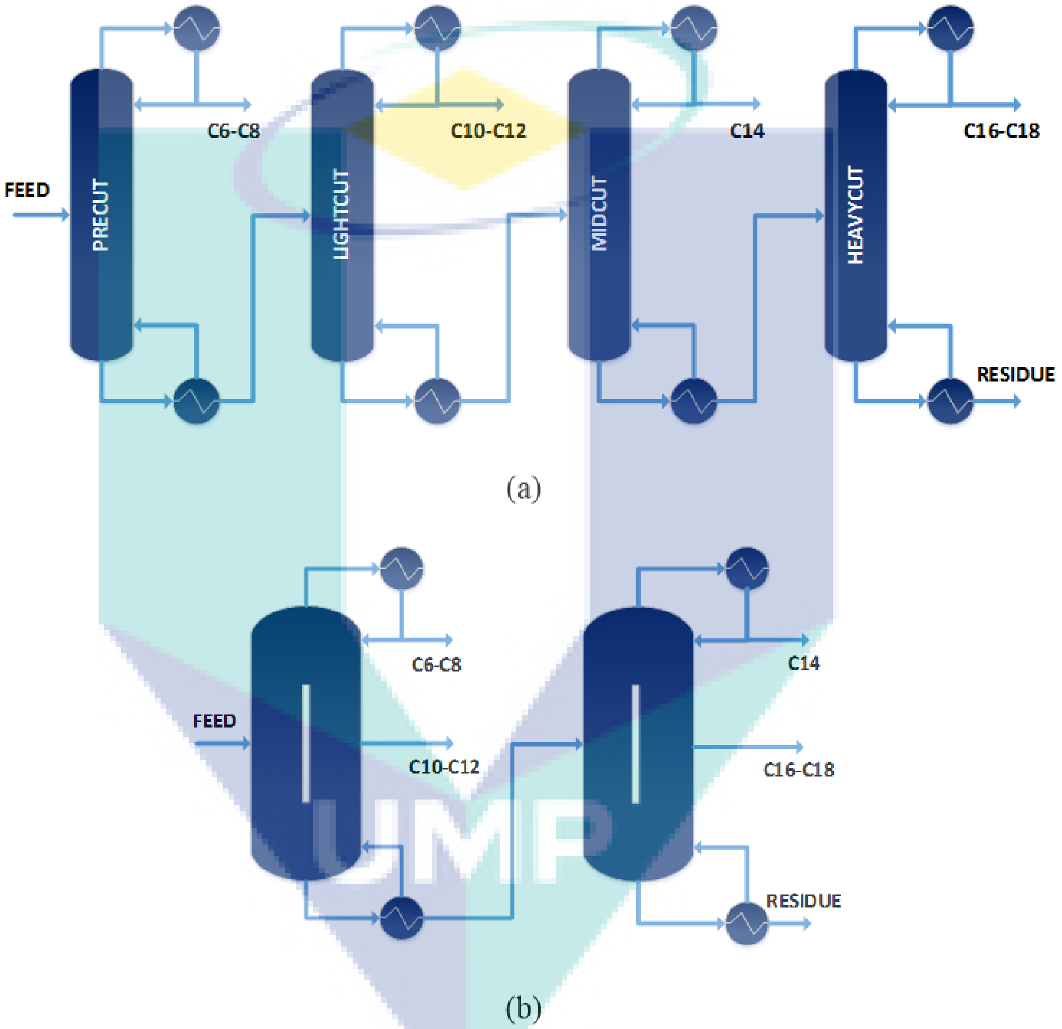


Figure 6.2. Process configuration for (a) CDC for Case 1 and Case 2 and (b) DWC for Case 3

2011). Moreover, since a unique block is used to represent each of the internal sections of DWC, such configuration could offer better insight of the column internal behaviour such as hydraulic and thermal profile as well as relatively more accurate sizing and cost estimation. It is important to note that, DWC block is not readily available in Aspen Plus, and therefore simulating DWC is not straightforward compared to a CDC. Using four-column configuration requires extensive computational effort. A lot of tweaking and tuning is needed, and column simulation experience will help a lot.

6.3.2 Economic and Environmental Comparison

From the simulation work mention in section 6.3.1 an economic and environmental analysis were performed and the results is depicted in Table 6.1. The total bare module cost for column tower in Case 3 is 6% cheaper than that in Case 2. On the other hand, the bare module cost for reboilers and condensers for Case 3 is 12.5% and 7.8% lower than that in Case 2, respectively. In total, the bare module cost of DWC is 8.2% cheaper than CDCs. Nevertheless, major benefits of DWC can be seen in the operating cost. Using DWC can save 36.4% of cooling water cost and 31.1% of high pressure steam cost compared to CDCs. These results are in alignment with DWC advantages for other applications (Premkumar & Rangaiah, 2009). In process plants, this is a huge advantage since distillation consumes almost 40% of total plant energy consumption. This energy reduction leads to similar reduction in CO2 emission.

6.4 Pilot Plant Development

The DWC pilot plant have charging capacity 5-7 kg/h for various oleochemical mixtures. It is very important however when handling fatty acids to consider high corrosive resistance material with low carbon content especially for pipes, sensors and instruments. The pilot plant is design to operate under vacuum condi-

Table 6.1

Summary of economic and environmental analysis

	Case 2 (CDCs)	Case 3 (DWC)	% Savings
Total bare module cost			
- Column towers [$]	934.1k	889.8k	6.1%
- Condensers [$]	630.0k	551,000	12.5%
- Reboilers [$]	1631k	1505k	7.8%
- Total [$]	3195.1k	2932.8k	8.2%
Operating cost			
- Heating [$/hr]	3.47	2.20	31.1%
- Cooling [$/hr]	49.77	34.30	36.4%
Environmental analysis			
- Total CO2 emission [kg/hr]	1309.1	902.1	31.1%

tion with temperature up to 200oC. Figure 6.3 shows the P&ID of the DWC pilot plant. It consists of six main parts namely condenser (A), rectifying section (B), pre-fractionation section (C), middle section (D), stripping section (E) and kettle reboiler (F). These main parts are made from borosilicate glass and sealed together using flanges with inserts and PTFE O ring to maintain vacuum condition inside the column. The main challenge to build the column is in designing and assembling the glass parts. Glass-based design is a good option for visual inspection of phenomenon that occurs within the column and suitable for research and development phase. Since the column is unique, we have to select available glass components in the market to fit the column design. The liquid splitting section is the most intriguing. Typically, the splitter is fabricated using stainless steel. In this work, a special design is employed that suitable for glass column. The splitter is equipped with pneumatic divider and a timer which could control the liquid splitting to the other section. Through this, the control of liquid splitting is possible. The total height of the column is 9.5 meter. To hold the column, it is attached to a metal frame shown in Figure 6.4 and 6.5. The frame material is made of aluminium and manufactured by local company, DSCAFF. The main glass column is packed with structured packings sponsored by Sulzer AG. At the top of each column sections were fitted with PTFE distributor. Meanwhile all column bottoms were fitted with glass support plate.

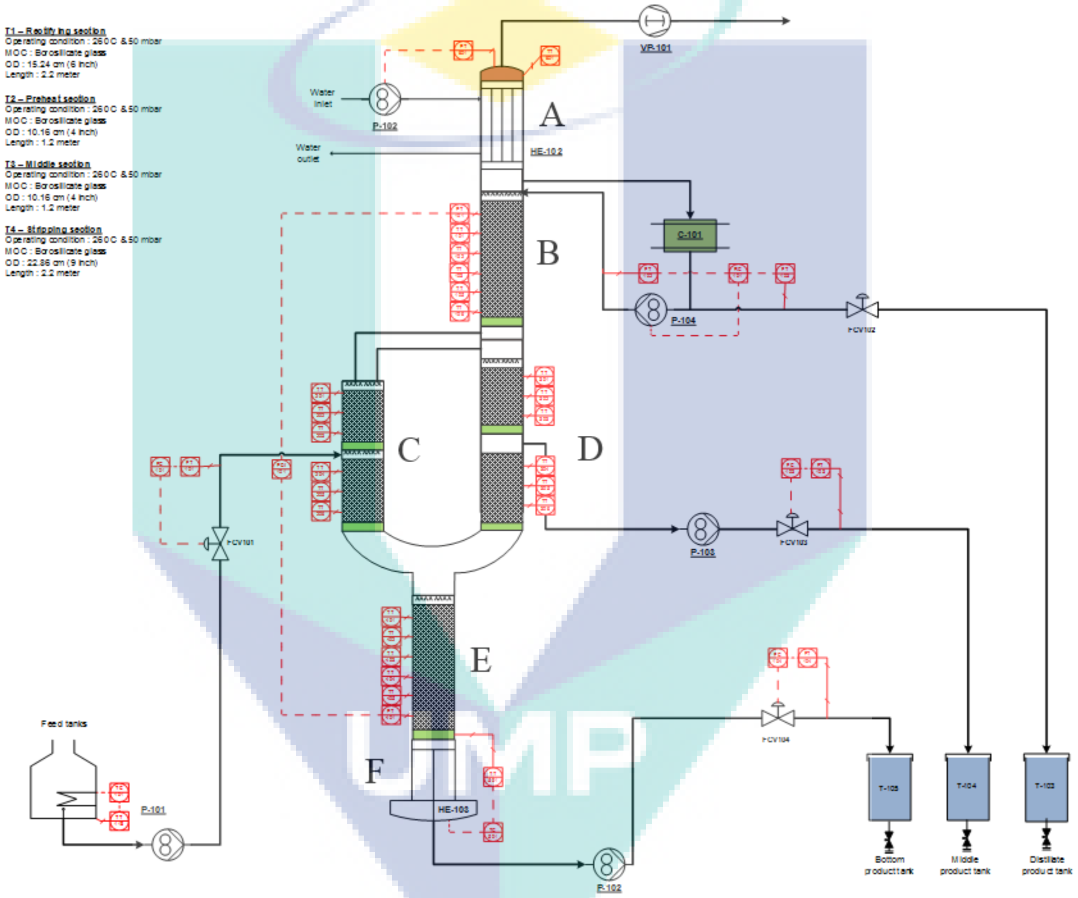


Figure 6.3. Simplified P&ID

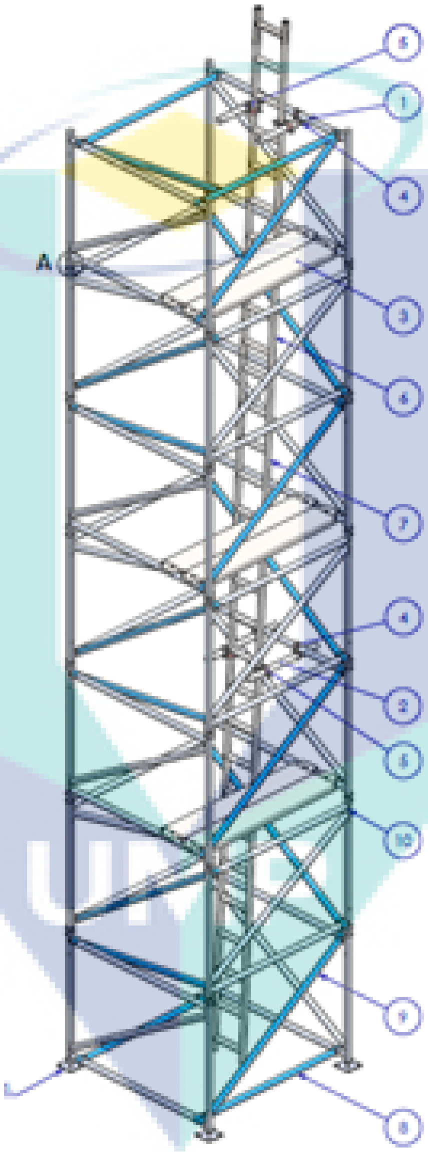


Figure 6.4. Schematic drawing of the metal frame structure

Figure 6.5. Metal frame structure

6.5 Technical Insights

The general structure and necessary utilities of DWC are similar to common distillation columns. Therefore, the set-up of general units, like reboilers, condensers, column internals are very much similar. The fundamental phenomenon occurs during operation is not much different either. But the aspect of the divided wall had very much affect the fluid dynamic relations of the whole column. Therefore, it is important to understand the hydrodynamics behaviour of the column i.e. pressure drops, vapour and liquid flows to the DWC operation and control. One issue is the vapour split. Design and simulations refer to a fixed value, while in operation vapour split must be maintained under the influence of disturbances. Here, it is possible to maintain steady state in the desired operation point via plant control or a rigorous modelling of the fluid dynamic behaviour, to determine the dependant vapour split and resulting product purifications. Having a pilot plant, this behaviour could be model and validated. DWC consist seven degrees of freedom, additional two when comparing to distillation column with side draw, namely condenser duty, distillate rate, side stream rate, bottom rate, reflux rate, reboiler duty, and liquid split. Vapour split is not included since it is impractical to be a manipulated variable since it has been fixed by the position of the wall in the designing state. Control of DWC is more complex since the four sections of the column are coupled and more interactions among controlled and manipulated variables exists. Dynamic analysis is essential because it helps in understanding the column operation and indispensable for successful commercialization of DWC. Besides model-based dynamic analysis, real plant dynamic also helps in understanding the column operation and selection of pre-eminent control structure to stabilize the column, to maintain the composition of the products at the set points, and to remain the minimum energy requirement.

6.6 Concluding remarks

In Malaysia, particularly in the oleochemical industries, our work shows that DWC is feasible to be implemented. However, the plant operation, knowledge and experience is needed before actual industrial scale implementation. In such, the development of this pilot plant enables us to explore the fundamental and advance application of DWC. There are so many areas waiting to be explored such as model identification, model-based and real-time optimization, process safety, hydrodynamic and CFD analysis, advanced process control and process fault detection and diagnosis. Despite for any research work, the pilot plant can also be utilize as training center for plant operators and engineers.

6.7 Acknowledgements

We gratefully acknowledge the financial support from the Malaysia Ministry of Education through ERGS (RDU130601) and FRGS (RDU140105) grant.

6.8 References

1. Chu, K.T., Cadoret, L., Yu, C.C. and Ward, J.D., 2011. A new shortcut design method and economic analysis of divided wall columns. Industrial & Engineering Chemistry Research, 50(15), pp.9221-9235.

2. Dejanovi, I., Matijasevic, L., Jansen, H. and Olujic, Z., 2011. Designing a packed dividing wall column for an aromatics processing plant. Industrial & engineering chemistry research, 50(9), pp.5680-5692.

3. Kaibel, G., 1987. Distillation columns with vertical partitions. Chemical engineering & technology, 10(1), pp.92-98.

4. Yildirim, ., Kiss, A.A. and Kenig, E.Y., 2011. Dividing wall columns in chem-

ical process industry: a review on current activities. Separation and Purification Technology, 80(3), pp.403-417.

5. Premkumar, R. and Rangaiah, G.P., 2009. Retrofitting conventional column systems to dividing-wall columns. Chemical Engineering Research and Design, 87(1), pp.47-60.

CHAPTER 7

CONCLUDING REMARKS

7.1 Summary

In this research work, DWC have been studied for separating multicomponents mixtures in the oleochemical industry. Our survey shows that fractionation of oleochemical products in Malaysia used conventional distillation columns (CDCs) and none so far imply DWC (dividing wall column). This is due to the lack of understanding of its design, control and operation.

A systematic simulation based approach for the design of DWC have been proposed. A four column conguration model were developed to represent DWC internal sections and was succesfully simulated using Aspen Plus. Moreover, sensitivity analysis and optimization work shows that the model were found to be more accurate in representing the behaviour of DWC.

Apart from that, our techno-economic and environmental feasibility study shows that compared to CDC, DWC saves up to 6% in capital cost and more than 30% savings for utility cost. Environmental analysis also shows the efficacy of DWC compared to CDC. We also proposed a novel AHP-HAZOP for process hazard analysis and implement it to the pilot plant.

In addition to the theoritical study, a DWC pilot plant have been built at Universiti Malaysia Pahang. The pilot plant was design on borosilicate glass components and operated under vacuum condition with temperature up to 200oC. The feed capacity is in the range of 5-7 kg/h for different type of oleochemical products.

With the realization of the pilot plant, future exciting work will be explore particularly on the operation and controllability. We believe such study integral work between theoritical and experimental scale is important to gain the operation experience for future industrial implementation.

7.2 Future works

Our research works have open new opportunities for further research. Here are the lists of possible future works to be undertaken:-

- Dynamic modelling and simulation of DWC for understanding the dynamic behaviour of DWC and analysis of different control structure to DWC operation.
- Control and operability study for the mini pilot plant.
- Hydrodynamic study of different packings structure.
- Feasibility study of reactive DWC for advanced application of DWC.

REFERENCES

Barroso-Muñoz, F. O., Hernandez, S., Segovia-Hernández, J. G., Hernández-Escoto, H., Rico-Ramírez, V., Chávez, R.-H., 2011. Implementation and operation of a dividing-wall distillation column. Chemical Engineering & Technology 34 (5), 746–750.

Dejanović, I., Matijašević, L., Olujić, Ž., 2010. Dividing wall columna breakthrough towards sustainable distilling. Chemical Engineering and Processing: Process Intensification 49 (6), 559–580.

Demirel, Y., 2013. Sustainable operations for distillation columns. Chemical Engineering & Process Techniques 1005, 1–15.

Kiss, A. A., Ignat, R. M., 2012. Enhanced methanol recovery and glycerol separation in biodiesel production–dwc makes it happen. Applied Energy 99, 146–153.

Sangal, V. K., Kumar, V., Mishra, M. I., 2013. Optimization of a divided wall column for the separation of c4-c6 normal paraffin mixture using box-behnken design. Chemical Industry and Chemical Engineering Quarterly 19 (1), 107–119.

Serra, M., Espuna, A., Puigjaner, L., 1999. Control and optimization of the divided wall column. Chemical Engineering and Processing: Process Intensification 38 (4), 549–562.

Yildirim, Ö., Kiss, A. A., Kenig, E. Y., 2011. Dividing wall columns in chemical process industry: a review on current activities. Separation and Purification Technology 80 (3), 403–417.