



HAL
open science

Design and simulation of divided wall column: Experimental validation and sensitivity analysis

Trung Dung Nguyen, David Rouzineau, Michel Meyer, Xuan Mi Meyer

► To cite this version:

Trung Dung Nguyen, David Rouzineau, Michel Meyer, Xuan Mi Meyer. Design and simulation of divided wall column: Experimental validation and sensitivity analysis. Chemical Engineering and Processing: Process Intensification, 2016, 104, pp.94-111. 10.1016/j.cep.2016.02.012 . hal-01911995

HAL Id: hal-01911995

<https://hal.science/hal-01911995>

Submitted on 5 Nov 2018

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Open Archive Toulouse Archive Ouverte (OATAO)

OATAO is an open access repository that collects the work of some Toulouse researchers and makes it freely available over the web where possible.

This is an author's version published in: <http://oatao.univ-toulouse.fr/20535>

Official URL: <https://doi.org/10.1016/j.cep.2016.02.012>

To cite this version:

Nguyen, Trung Dung and Rouzineau, David and Meyer, Michel and Meyer, Xuân-Mi
Design and simulation of divided wall column: Experimental validation and sensitivity
analysis. (2016) Chemical Engineering and Processing: Process Intensification, 104.
94-111. ISSN 0255-2701

Any correspondance concerning this service should be sent to the repository administrator:
tech-oatao@listes-diff.inp-toulouse.fr

Design and simulation of divided wall column: Experimental validation and sensitivity analysis

Trung Dung Nguyen^{a,b}, David Rouzineau^{a,*}, Michel Meyer^a, Xuan Meyer^a

^a UNIVERSITE DE TOULOUSE, ENSIACET–INP de Toulouse, Laboratoire de Génie Chimique, UMR CNRS 5503, 4 allée Emile Monso, BP 44362, 31432 Toulouse Cedex 4, France

^b Hanoi University of Science and Technology, 1 Dai Co Viet Street, Hanoi, Vietnam

ABSTRACT

This article deals with design and simulation of divided wall column. Design parameters are provided to the rigorous simulation in the ProSimPlus[®] software. The results show that the procedure can determine parameters quickly in the case studies and can give a good initialization for rigorous simulation. Secondly, a pilot plant has been design, built and operated in our laboratory. The pilot plant will provide necessary experimental evidence to validate the previous procedure. Ternary mixture and four-component mixture of alcohols have been used in our pilot plant in steady state conditions. The results show that the composition of products, composition and temperature profile along the column are in very good agreement with simulation results. Finally, in order to determine the optimal parameters of divided wall columns, the effects of the structural parameters of the divided wall column such as the height of the wall, the vertical position of the wall and number of stages of each section are analyzed. Ternary diagram is used as an indicator both in showing what the most economical configuration is and in showing the distillation boundary.

Keywords:

Process intensification
Divided wall column
Process design
Simulation
Pilot plant
Sensitivity analysis

1. Introduction

The divided wall column (DWC) has many known advantages, and many design methodologies for DWC's have been published over the last years. Almost all papers that have been published were restricted to ternary mixtures with three products, sharp separations, saturated liquid feed, constant flowrate and constant relative volatility. The design of divided wall columns or fully thermally coupled distillations is more complex than traditional distillation because it has more degrees of freedom. A number of papers have been published on the subject which focus on the calculation of the minimum vapor requirement and determined the number of stages in the various column sections. Triantafyllou and Smith [4] published a design oriented shortcut method for three products in a divided wall column based on the Fenske–Underwood–Gilliland–Kirkbridge model (FUGK). In this paper, they presented a method to decompose a divided wall column into a three-traditional-column model. By using the decomposition method, they assume that heat transfer across the column wall can be neglected, hence making the divided wall column equivalent to a fully thermally coupled distillation. The prefractionator is

considered like a traditional column if a partial condenser and a partial reboiler are used. The main column can be represented as two traditional columns if we assume a total reboiler for the upper part of the main column and a total condenser for the lower part of the main column. The interconnecting streams are considered as the feed flowrates with superheated vapor and sub-cooled liquid conditions, respectively. The FUGK method can be applied to determine operational and structural parameters for each column. The minimum number of equilibrium stages can be determined by the Fenske equation, the minimum reflux ratio can be determined by using the Underwood equation, the number of stages can be determined by the Gilliland method when choosing operating reflux ratio, and feed location can be determined by the Kirkbridge method. The reflux ratio of the prefractionator is adjusted until its number of stages equals the number of the side section. The recoveries in the prefractionator column are optimized for the minimum vapor flowrate or the minimum number of stages.

Amminudin et al. [1,2] proposed a semi-rigorous design method based on equilibrium stage composition concept. Certain assumptions are as follows: constant molar overflow, constant relative volatility, and estimation of component distribution at minimum reflux. Their design procedure starts from defining the products composition, and works backward to determine the design parameters required to achieve them. Therefore, firstly, by using

* Corresponding author.

E-mail address: david.rouzineau@ensiacet.fr (D. Rouzineau).

Nomenclature

A, B, C	Ternary mixture (A is the most volatile component and C is the least volatile component)
D	Top product flowrate (kmol h ⁻¹)
F	Feed flowrate (kmol h ⁻¹)
L	Liquid flowrate in the rectifying section (kmol h ⁻¹)
\bar{L}	Liquid flowrate in the stripping section (kmol h ⁻¹)
N	Number of stages
q	Quality of the stream
R	Reflux ratio
R _L	Liquid split between prefractionator and main column
R _V	Vapor split between prefractionator and main column
S	Side product flowrate (kmol h ⁻¹)
V	Vapor flowrate in the rectifying section (kmol h ⁻¹)
\bar{V}	Vapor flow rate in the stripping section (kmol h ⁻¹)
x	Mole fraction at the product stream
W	Bottom product flowrate (kmol h ⁻¹)
z	Mole fraction at the feed stream

Subscripts

1	Column I
2	Column II
3	Column III
b, c, d, e	sections separated by dividing wall
HK	Heaviest key component
LK	Lightest key component
min	minimum value
R	Rectifying section
S	Stripping section

Greek symbols

α	Relative volatility of component
τ	Recovery ratio of the component
$\theta, \theta', \theta''$	Roots of Underwood's equation
ω_G	Gas velocity (m s ⁻¹)
ρ_G	Gas density (kg m ⁻³)

the method of Van Dongen and Doherty [18], a feasible product distribution is estimated for the composition of the top, middle and bottom products, the minimum reflux ratio and the minimum boil-up ratio. Any distillation operation lies between the two limits of total reflux and minimum reflux ratios. At total reflux ratio, the number of stages is minimized and energy consumption is maximized. At the minimum reflux ratio, the number of stages is maximized and energy consumption is minimized. Therefore, a product distribution must be chosen between the two conditions. Secondly, using the equilibrium stage concept the number of stages, flow rates, feed stage and side stream location for the fully thermally coupled distillation are estimated.

An approximate design procedure for fully thermally coupled distillation column is proposed by Kim [11]. The Fenske equation is applied to the main column to determine minimum number of stages. However, the author believed that the design of the prefractionator cannot follow the Fenske equation because the end compositions are unknown. Therefore, a stage-to-stage computation is proposed. Then, the number of stages in the system is taken as twice the minimum number of stages. The minimum vapor flowrate was determined by the Underwood equation. The liquid flowrate of the main column is determined by checking the compositions of the products. Clearly, they take twice the

minimum number of stages as the number of theoretical trays is considered to be equal to two times the minimum number of stages. It is not always true.

Halvorsen and Skogestad [7,10] proposed the V_{\min} diagram method to determine the minimum energy consumption. To use the method, they assume constant molar flowrates, constant relative volatilities, and an infinite number of stages. Firstly, the V_{\min} diagram is drawn based on the Underwood equation. The minimum energy requirement for separation of a feed mixture of n components into n pure products is given by:

$$V_{\min}^{\text{Petlyuk}} = \max \sum_{i=1}^j \frac{\alpha_i z_i F}{\alpha_i - \theta_j}; j \in \{1, n-1\}$$

Here: θ_j are the $n-1$ common Underwood roots found from:

$$1 - q = \sum_{i=1}^n \frac{\alpha_i z_i}{\alpha_i - \theta}$$

Underwood roots obey $\alpha_1 > \theta_1 > \alpha_2 > \theta_2 > \dots > \theta_{n-1} > \alpha_n$

where: q is liquid fraction in the feed (F) and z is the feed composition

Secondly, they choose the actual flowrate around 10% and the minimum number of stages was calculated based on the Underwood equation.

Calzon-McConville et al. [5] presented an energy efficient design procedure for optimization of the thermally coupled distillation sequences with initial designs based on the design of conventional distillation sequences. In the first step, it is assumed that each column performs with specified recoveries of components of 98% (light and heavy key components) and by using the shortcut method (FUG model), the number of stages of conventional distillation schemes are obtained. In the second step, the stage arrangements in the integrated configurations are obtained; finally, an optimization procedure is used to minimize energy consumption. The energy-efficient design procedure for thermally coupled distillation sequences is applied not only for the separation of ternary and quaternary mixtures but also for the separation of five or more component mixtures.

Sotudeh and Shahraki [14,15] proposed a shortcut method for the design of a divided wall column based only on the Underwood equation because authors believe that using the Fenske equation for calculating the minimum number of stages is not adequate for designing divided wall columns. The theoretical number of stages can be calculated by using the basic Underwood equation. In this method, the number of stages in the prefractionator is set to be the same as in the side section. Clearly, we cannot know that the number of stages of prefractionator is correct or not. Moreover, the paper does not carry out simulations to confirm the method.

Ramirez-Corona et al. [13] presented an optimization procedure for the Petlyuk distillation system. The procedure used the FUG model to determine the structural design of the divided wall column as well as the mass and energy balances, the thermodynamic relationships, and cost equations. The objective function was set as the minimization of the total annual cost. In the procedure, they estimated the composition of the interconnection streams between the prefractionator and the main column by solving the feed line and the operating line equations.

$$y_i = \left(\frac{q}{q-1} \right) x_i - \frac{x_{i,D}}{q-1}$$

$$y_i = \left(\frac{R}{R+1} \right) x_i + \frac{x_{i,D}}{R+1}$$

Table 1
Summary of several shortcut methods for design of divided wall columns.

References	Model	Method	Hypothesis	Mixture analysis
Triantafyllou and Smith [4]	Three-column sequence model	FUGK method Minimum cost of system	Constant relative volatilities Constant molar flows	<i>i</i> -Butane/1-butene/ <i>n</i> -butane/ trans-2-butene/ <i>cis</i> -2-butene
Amminudin et al. [2]	Three-column sequence model	Semi-rigorous design method based on the equilibrium stage composition concept	Constant molar overflow Constant relative volatilities Estimate product distribution at minimum reflux	Ethylene/propene/ <i>n</i> -propane/ <i>i</i> -butane/1-butane/ <i>n</i> -butane/ <i>n</i> -pentane/ <i>n</i> -hexane Methanol/ethanol/water Cyclohexane/ <i>n</i> -heptane/ toluene <i>s</i> -Butanol/ <i>i</i> -butanol/ <i>n</i> -butanol
Kim [11]	Two-column sequence model (pre-fractionator and main column)	Fenske equation for the main column and a stage-to-stage computation for the pre-fractionator. Take twice the minimum number of stages as the number of theoretical stages	Ideal equilibrium is assumed between the vapor and liquid of interlinking streams and the shortcut design equations of multi component distillation columns.	
Halvorsen and Skogestad [7,10]	Two-column sequence model (pre-fractionator and main column)	V_{min} diagram method Underwood's equation	Constant molar flow Infinite number of stages Constant relative volatilities	
Sotudeh and Shahraki [15]	Three-column sequence model	Underwood's equation Number of stages in the pre-fractionator is set to be the same as in the side section The compositions of interconnection streams are design variables	Constant relative volatilities	Benzene/toluene/Xylene <i>i</i> -Butane/1-butene/ <i>n</i> -butane/ trans-2-butene/ <i>cis</i> -2-butene
Ramirez-Corona et al. [13]	Three-column sequence model	FUGK method They calculate the composition of interconnection streams Minimization of the total annual cost	Constant relative volatilities Constant molar flowrate The interconnecting streams are saturated	<i>n</i> -Pentane/ <i>n</i> -hexane/ <i>n</i> -heptane <i>n</i> -Butane/ <i>i</i> -pentane/ <i>n</i> -pentane <i>i</i> -Pentane/ <i>n</i> -pentane/ <i>n</i> -hexane
Chu et al. [6]	Six different sections model	Applied the components net flow model FUGK method	Constant relative volatilities Constant molar flowrate The column is symmetric	Ethanol/ <i>n</i> -propanol/ <i>n</i> -butanol Benzene/toluene/ ethylbenzene
Calzon-McConville et al. [5]	Superstructure model	Based on the design of conventional distillation sequences, the stages are rearranged to the integrated configurations Minimize energy consumption		<i>n</i> -Butane/isopentane/ <i>n</i> -Pentane/ <i>n</i> -hexane/ <i>n</i> -heptane

Combining these equations, one obtains:

$$x_i = \frac{z_i(R+1) + x_{i,D}(q-1)}{R+q}$$

$$y_i = \frac{Rz_i + qx_{i,D}}{R+q}$$

Chu et al. [6] presented a new shortcut method based on the efficient net flow model to determine the composition of the key components. They then applied the shortcut method of Fenske, Underwood, Gilliland and Kirkbride to determine the number of stages of each section. Liquid split R_L and vapor split R_V are dependent variables due to the constant molar flow assumption. The values of R_L and R_V are chosen to obtain the same number of trays in the pre-fractionator and in the side section. Table 1 shows the summary of several shortcut methods for design of divided wall columns. Based on the above analysis, we can conclude that a lot of papers focused on the design and simulation for divided wall column. However, these methods still have drawbacks. The method of Triantafyllou and Smith [4] applied the FUGK model that can quickly and easily determine operational and structural parameters of divided wall columns. However the application of the Fenske equation for the estimation of the minimum number of stages of a divided wall column is not correct since the composition of the liquid stream returning from the main column is not equal to the composition of the vapor entering the main column at the connection points. Kim [11] applied a stage-to-stage computation method instead of the Fenske equation for the pre-fractionator.

However the actual number of stages in the system takes twice the minimum number of stages. Sotudeh and Shahraki [14] used only the Underwood equation to determine the number of stages in the main column and they set the number of stages of the pre-fractionator to be the same number of stages as in the side section. Ramirez-Corona et al. [13] also applied the FUGK method and estimated the composition of interconnecting streams. Moreover, all the previous methods are restricted to ternary mixtures with a feed quality (q) equal to 1.

2. Design and simulation procedure for divided wall column

2.1. Model design

This paragraph aims to present, by application of standard shortcut method (FUGK model) and using the component net flow model, a procedure for designing divided wall columns. The approach allows rapid determination of the minimum vapor flow rate, minimum reflux ratio, and number of stages for each section

Table 2
Unknown variables and specifications of DWC system.

	Unknown variables	Specifications
Distillate product	$D_2, x_{A,D_2}, x_{B,D_2}, x_{C,D_2}$	x_{A,D_2} and x_{C,D_2}
Side product	$S, x_{A,S}, x_{B,S}, x_{C,S}$	$x_{B,S}$ and $x_{A,S}$ or $x_{C,S}$ or $\frac{x_{A,S}}{x_{C,S}}$
Bottom product	$W_3, x_{A,W_3}, x_{B,W_3}, x_{C,W_3}$	x_{A,W_3} and x_{C,W_3}

by choosing an operating reflux ratio, liquid and vapor split values, and the possible position and configuration of the dividing wall. Moreover, the compositions of interconnecting streams between the prefractionator and the main column are also estimated and set as the initial conditions for simulation in ProSimPlus® software.

Assumptions and model design

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

- (1) The relative volatility of components is constant;
- (2) The vapor and liquid flows in each section of the divided wall column are constant;
- (3) The pressure of the system is constant;
- (4) The heat transfer across the dividing wall is neglected;
- (5) The heat losses from the column walls are negligible;
- (6) Vapor-liquid equilibrium is achieved on each stage;
- (7) The heavy non-key component is assumed to go completely to the bottom of column II (Fig. 2) and the light non-key component is assumed to go completely to the top of column III (Fig. 2);

Kister [9] defined that key components are the two components in the feed mixture whose separation is specified. They are called light key component (more volatile) and heavy key component (less volatile). Other components are called non-key components. Any components lighter than the light key are called light non-key components, while those heavier than the heavy key are called heavy non-keys components. The components that lie between the light key and the heavy key are called distributed non-key components. The procedure can be applied not only for ternary mixtures but also for four component mixtures. To simplify, we consider separation of a ternary mixture A, B, and C, in which A is the lightest component and C is the heaviest component. The feed flowrate is F (kmol/h), feed composition z_A, z_B , and z_C , and recoveries or purities of component in divided wall column are known. The relative volatilities of each component are constant (assumption 1).

The feed composition is listed in decreasing order of the relative volatility of the component:

$$\alpha_A > \alpha_B > \alpha_C = 1$$

The minimum number of stages at total reflux may be estimated by using the Fenske equation. It is applied with the assumption that all stages reach equilibrium (assumption 6) and requires a constant relative volatility α throughout the column (assumption 1). To determine the minimum reflux ratio, the equations developed by Underwood are based on the assumption (2): constant molar flowrate. Then, the knowledge of minimum stages and minimum reflux ratio in a column can be related to the actual number of stages and the actual reflux required by the Gilliland correlation. Finally, the feed stage can be estimated by using the Kirkbride equation.

Based on the assumption (4), the divided wall column in Fig. 1(a) is equivalent to the fully thermally coupled distillation in Fig. 1(b). Therefore, the prefractionator will be used instead of section 1. The main column will be used instead of sections 2 and 3. The interconnecting streams are added on and connected between the prefractionator and the main column.

Based on the figure of thermally coupled distillation 1 (b), the main column can be represented as two traditional columns shown in Fig. 2 if we assume a total reboiler for column II and a total condenser for column III. The prefractionator is also considered as a traditional column if we assume a partial condenser and a partial reboiler while the interconnecting streams are considered as the feed flow-rates for column II and III with superheated vapor and sub-cooled liquid conditions, respectively.

Based on Fig. 2, components A and C are key components and the component B is the distributed component in column I. Therefore, the top of the column I is mainly component A, a part of component B and a little of component C. The bottom of the column I is mainly component C, a part of component B and a little of component A. Column II separates components A and B. Therefore, A and B are key components and component C is heavy non key component. Based on the assumption (7), all of component C leaves from the bottom of column II. Column III separates components B and C. Therefore, B and C are key components and component A is a light non key component. Based on the assumption (7), all of component A leaves from the top of column III.

The FUGK equations of material balance, minimum vapor flowrate of each column (Fig. 2), and number of stages for each section are given in Appendix 1.

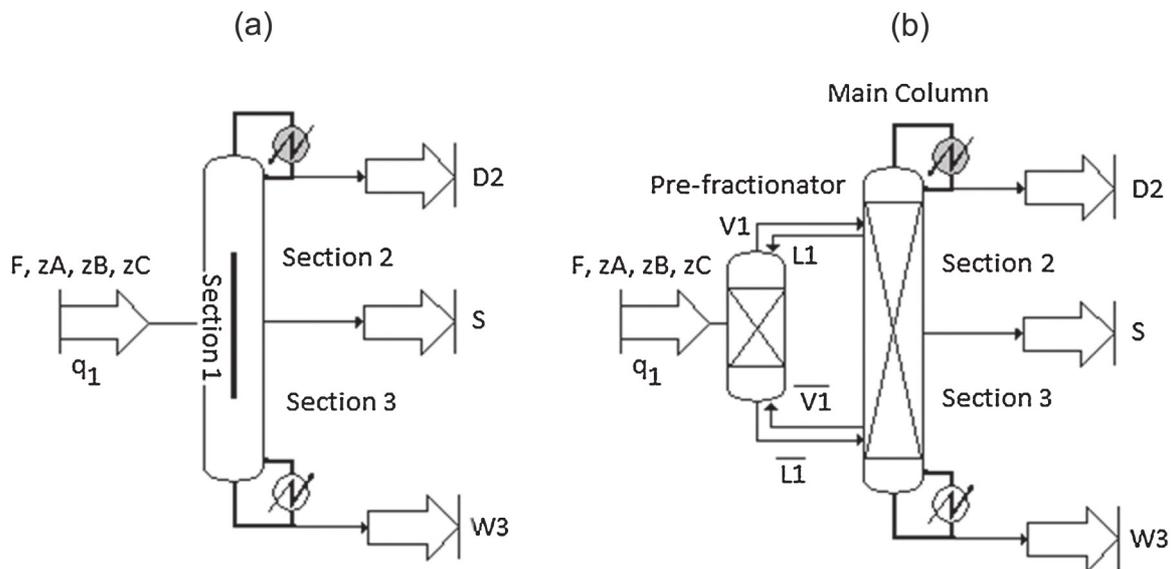


Fig. 1. (a) Divided wall Column; (b) thermally coupled distillation.

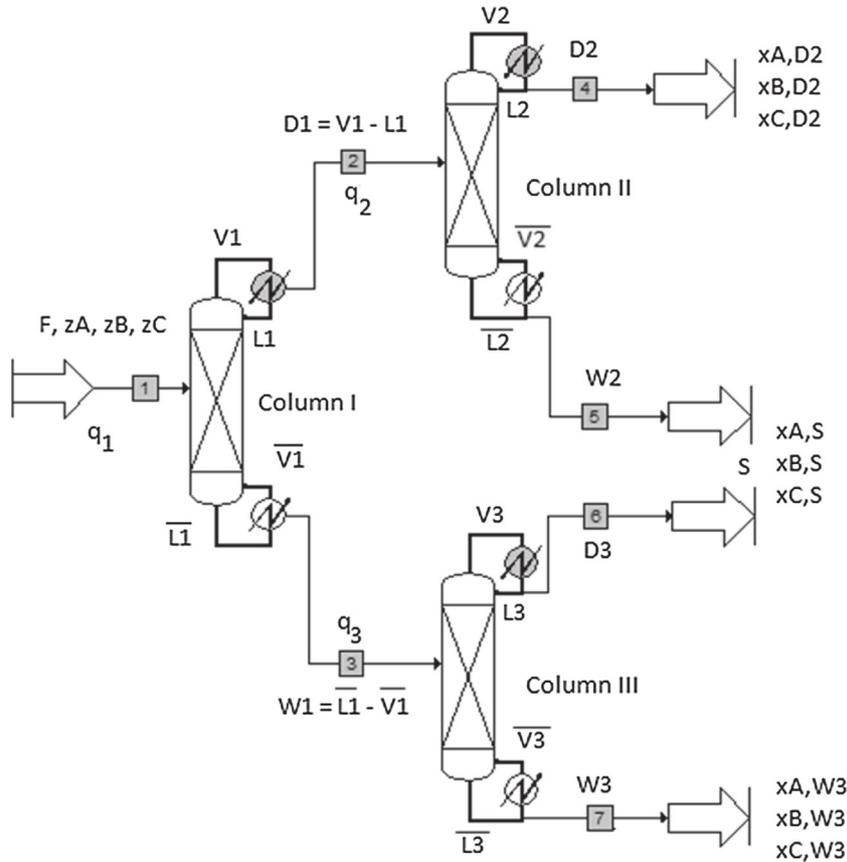


Fig. 2. Simplified model design of divided wall column.

This shortcut procedure is implemented in ProSimPlus software to obtain all parameters of divided wall column.

2.2. Simulation with ProSimPlus software

There is no standard model for the simulation of a divided wall column in commercial software. As shown in Fig. 3, there are four possible models for the simulation: pump around sequence, two-column sequence with prefractionator, two-column sequence with postfractionator, and four-column sequences. For the pump around sequence, Becker et al. [3] reported that the model can lead to convergence problems because in two points of the column entire vapor and liquid are drawn off, and none remains to “flow” to the next tray. The four-column sequence model reflects the actual

best situation, but it is most difficult to initialize, because initial values of more interconnecting streams are required. It is also the slowest model to converge. It is considered for use with dynamic simulations [8].

The structural and operational parameters are determined by shortcut method, they are used as initial parameters for the simulation. Besides the above necessary information, it is noted that the composition, temperature, and flowrates of interconnecting streams (2 and 3) or (4 and 5) must be set in the model. If there is no specification, the simulation cannot be run. Not only because the stream 1 is the feed stream but also because streams 4 and 5 are the feed streams for the prefractionator. Therefore, they must be specified for the initial run of the simulation.

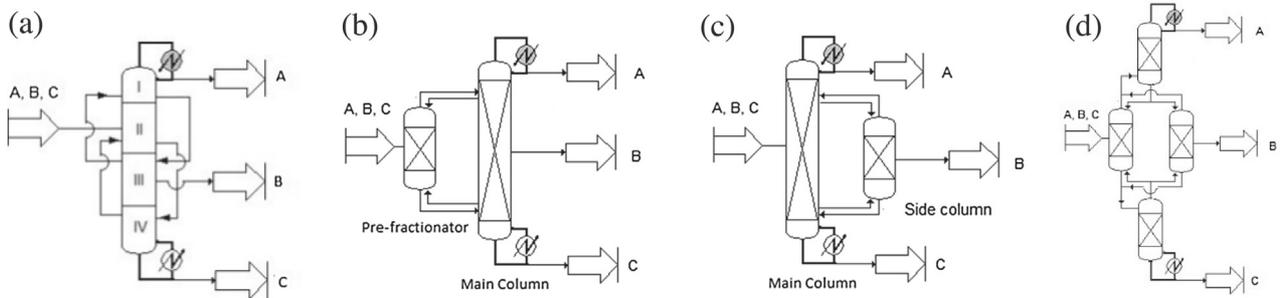


Fig. 3. Models for simulation: (a) pump around sequence (b) two-column sequence with prefractionator (c) two-column sequence with postfractionator (d) four-column sequences.

Based on these reasons, the two-column sequence with prefractionator is used to simulate the system in ProSimPlus. As shown in Fig. 4, the first column is considered as the prefractionator and the second column as the main column. The interconnecting streams 2, 3, 4, and 5 connect the two columns. The top, side and bottom product are the streams 6, 7, and 8, respectively and the feed flow rate is stream 1

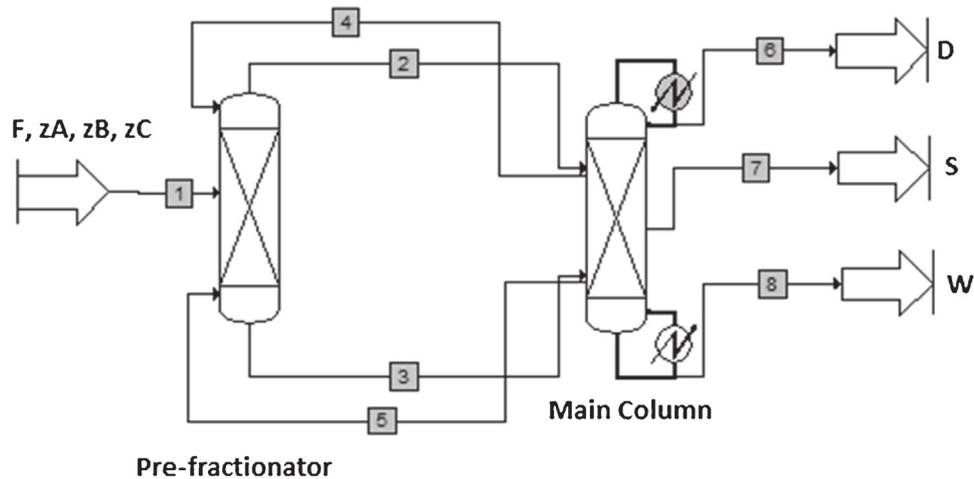


Fig. 4. Configuration used for simulation.

3. Pilot plant

A pilot plant for the divided wall column was set up in our laboratory (LGC, Toulouse, France, 2013). Fig. 5 shows the diagram of the pilot plant for separation of a multi-component mixture separation. Total height of the pilot plant is 5.53 m. It is made of glass and operates under atmospheric pressure. The column is divided into three parts. The upper and lower parts of the column have 6 elements each with a height of 0.3 m and an inner diameter of 80 mm. The middle part of the column is divided into the feed section and the side section. Each section has 4 elements with the height of each element being 0.2 m and having an inner diameter of 50 mm. The height of the connecting elements between the upper part and the lower part with the middle part are Y-shaped and approximately 280 mm in length. The height of the splitting element is 170 mm. The structured packing used in the pilot plant is Sulzer DX for the separation section. Our pilot plant has a parallel structure in the middle section. This was chosen due to the small inner diameter. If we put a dividing wall inside, the liquid distribution will be effected. Moreover, the heat transfer across the dividing wall is not considered in the study. At the top of the column, the condenser is installed and operated with cooling water. The condensate returns to the column due to gravity and a part is taken out as the distillate product through the liquid reflux split valve. The top product is drawn off into a distillate tank. At the bottom of the column, the mixture in the reboiler is heated by a vapor stream. A fraction is taken out as the bottom product. The side product, located at the side section, is cooled by cooling water and is drawn off into the side tank by gravity. The feed stream, from a feed tank through the pump, was heated by a preheater and fed into the feed location in the feed section. The feed flowrate is varied from 5 to 7 kg h⁻¹. To reduce the heat losses through the wall of the column, a jacket is installed along the entire length of the column. The liquid splitter defines the liquid load between the feed section and the side section. The liquid from the top of the column is drawn off via a funnel which is placed in the splitting element and is moved by two electromagnets to facilitate the liquid distribution to each side of the section. The vapor is not controlled but is dependent on the inner diameter of the feed and side sections and the pressure drop inside the packed section. In our pilot plant, the inner diameters of the feed and side section are the same. Moreover the height of the packing of each section is also the same. Therefore, theoretically, the vapor split is around 0.5.

Concerning samples and measurements, the liquid inlet and outlet streams in the pilot plant are measured by weighing the quantity of liquid collected in the product tanks or lost in the feed

tank. The information is noted every 30 min during the steady state experimental runs. The accuracy of a weighing machine is 0.001 g. The pilot plant is equipped with the sixteen temperature sensors (T) along the column, of which, two temperature sensors measure the temperature of cooling water in and out as shown in the flow-sheet in Fig. 5. All temperatures are automatically recorded. The liquid samples (EI) are taken from the feed stream, three products and 11 points along the column. They are analyzed by using gas chromatography. Two pressure sensors record the pressure drop between the top and the bottom of the column during pilot plant operation. The heat duty of the system was calculated by measuring the quantity of liquid leaving as condensate from the bottom of the column.

HETP experiments need to be performed to calculate HETP (height equivalent to a theoretical plate) value of the packing used in the pilot plant. The standards for the experimental method of separating a binary mixture at total reflux that are defined by Fractionation Research Inc. (FRI) and Separation Research Program (SPR) will be applied. A standard cyclohexane and n-heptane mixture is carried out in the divided wall column system at atmospheric pressure with different runs. Firstly, the flooding point was determined, then backing off to roughly 20% of the flood flowrate to unload the bed. Secondly, the tests are run at the targeted reboiler duty. The liquid samples were taken only from EI-7, EI-8, and EI-9 with the height of each unit at 0.6 m as shown in Fig. 5. It is not necessary to analyse more liquid samples as the sample composition has stabilised. The samples are analyzed by a refractometry method in the refractometer to assess the composition of the samples. The number of equilibrium stages is determined by using the Fenske equation. The results show that the average F -factor is equal 2.01 and the number of theoretical stages between EI-7 and EI-8 or EI-8 and EI-9 is 5.21. Thus the average HETP was 0.115

4. Results

4.1. Design and simulation

4.1.1. Three component mixture

The separation of a ternary mixture, methanol, 1-propanol, and 1-butanol, is carried out in divided wall column. The feed flowrate is 100 kmol h⁻¹ and contains 30% mole fraction methanol, 40% mole fraction 1-propanol, and 30% mole fraction 1-butanol. The feed quality q_1 is equal to 1. Operating pressure is 1 atm. The specifications for the product purities for distillate and bottom products are 99% mole fraction and the side product is 98% mole

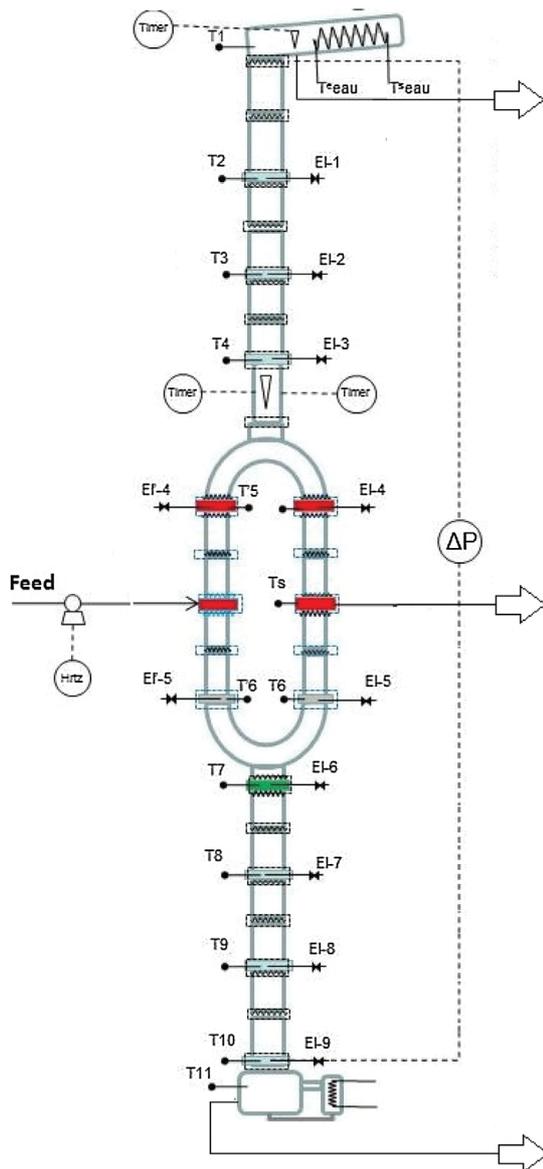


Fig. 5. Flow-sheet of the pilot plant.

LEGEND

Upper section:

6 elements
 $H = 300$ mm
 $D = 80$ mm

Feed and side sections:

4 elements
 $H = 200$ mm
 $D = 50$ mm

Lower section:

6 elements
 $H = 300$ mm
 $D = 80$ mm

Measurement:

16 temperature sensors (T)
 11 liquid sample positions (EI)
 1 pressure drop measurement

fraction. Firstly, the shortcut design procedure determines the structural and operational parameters of the divided wall column. Then, steady-state simulations were carried out in ProSimPlus[®] software. For the thermodynamic model, NRTL model should be used. Fig. 6 provides the results of design parameters for the divided wall column, while Table 3 shows the relative error between the specified product purities and simulation results of the key components. Notice that in order to run simulation with ProSimPlus[®], the information required to initialize a simulation is given in Fig. 6. Based on the volatilities of the components, methanol is the lightest component and is collected as distillate product, 1-propanol is the distributed component collected in the side stream, and 1-butanol is the heaviest component collected as the bottom product.

In Fig. 6, the structure of the divided wall column consisted of 40-stages, with 19-stages in the prefractionator located between stages 8 and 27, the feed location is at stage 17, the side stream at stage 15, a liquid and vapor split of 0.18 and 0.41, respectively, the reflux ratio of 3.8 and a reboiler duty of 1518 kW.

Table 3 compares the specification of key product purities with simulated results. The result indicates that the approach works well and provides both the basis for preliminary optimization and a good initialization for simulation.

4.1.2. Four component mixture

When three-component mixture A, B, and C are separated in the divided wall column, the lightest component A is collected in the distillate product, the middle component B is collected in the side stream, and the heaviest component C is collected in the bottom product. Therefore, three pure components can be obtained in three product streams. However, if the separation of a mixture has more than three components in the divided wall column, it is difficult to obtain each pure component. This section develops the procedure for four component mixtures. The separation of a four-component mixture composed of methanol (A) 40% mole fraction, isopropanol (B) 30% mole fraction, 1-propanol (C) 20% mole fraction, and 1-butanol (D) 10% mole fraction is considered. Feed flowrate is 100 kmol/h, feed quality is 1, and operating pressure is

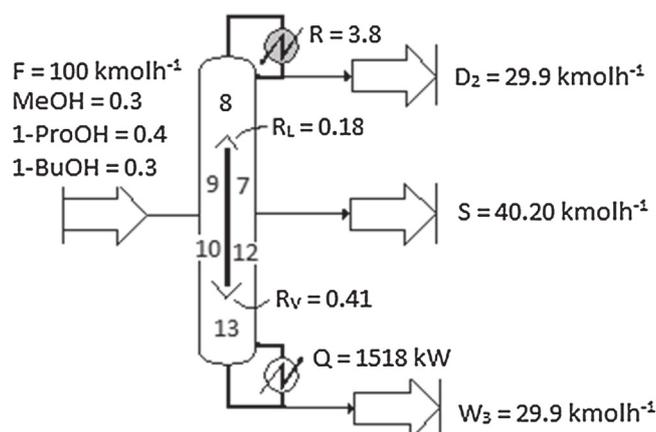


Fig. 6. Structural and operational parameters of DWC system.

1 atm. The desired side product is isopropanol. Therefore, the distillate product contains methanol and a little isopropanol, and the bottom product contains a little isopropanol, 1-propanol, and 1-butanol.

In the case of the four-component mixture, from the balance equations, there are seven equations with fifteen unknown variables. Therefore, to solve the balance equations, 8 variables have to be specified:

$x_{A,D2}; x_{C,D2}; x_{D,D2}; x_{B,S}; x_{A,S}/x_{C,S}; x_{D,S}; x_{A,W3}; x_{B,W3}$ as shown in Fig. 7.

In the top product, $x_{A,D2}$ should be specified because it is a key component while $x_{C,D2}$ and $x_{D,D2}$ are set to zero because we have made the assumption that heavier components are not present in the top product. In the side product, $x_{B,S}$ is the key component so it is specified. The composition of component A and C also should be known. Therefore $x_{A,S}$ or $x_{C,S}$ or $x_{A,S}/x_{C,S}$ should be specified. Finally, in the bottom product, the lightest component A ($x_{A,W3}$) is fixed as zero and the composition B ($x_{B,W3}$) is specified.

In this case, the methanol is specified at 95% mole in the top product, isopropanol is specified at 90% mole in the side stream and isopropanol is specified at 1% mole instead of 1-propanol or 1-butanol in the bottom product as shown in Fig. 8. Firstly, the shortcut design procedure determines the structural and operational parameters of the divided wall column. Then, the simulation of the divided wall column is carried out in ProSim software. The results of structural and operational parameters from the shortcut method are shown in Fig. 8 and relative errors of key components in the product streams are shown in Table 4. The results show that the divided wall column has 43 stages in which the number of stages in the prefractionator is 20 stages. Feed and side positions are located at stage 14. The liquid and vapor splits are 0.5 and 0.69, respectively. The reflux ratio is 2.85 and the reboiler duty of 1268 kW.

Table 4 shows that all relative errors are negatives that means that the simulated results do not reach to the specification. All relative errors are less than -5% .

4.2. Experimental validation

4.2.1. Component system

The procedure for designing a divided wall column applies not only for ternary mixtures but also for four component mixtures. Therefore, to verify the procedure, ternary mixtures and four-component mixtures are investigated in the pilot plant. In the first case, a ternary mixture of methanol, 1-propanol and 1-butanol are chosen for investigation in our pilot plant. This mixture was chosen because the maximum boiling point of the mixture is 118°C while the preheater of the pilot plant can heat the mixture up to 150°C . Moreover, the alcohols can be easily bought in the chemical industry. According to their boiling points from lowest to highest, methanol is obtained in the top product, 1-propanol is obtained as the side product, and 1-butanol is obtained as the bottom product. The different feed compositions of the mixture and different liquid splits will be considered. In the second case, the four-component mixture of the methanol, isopropanol, 1-propanol, and 1-butanol also is carried out in the divided wall column. The distribution of the components to the products is studied. Firstly, isopropanol is a distributed component. Therefore, methanol is obtained as the top product, isopropanol is obtained as the side product, and 1-propanol and 1-butanol are obtained as the bottom product. Secondly, 1-propanol is a distributed component. Therefore, methanol and isopropanol are obtained as the top product, 1-propanol is obtained as the side product, and 1-butanol is obtained as the bottom product.

4.2.2. Ternary mixture

First, a ternary mixture of methanol, 1-propanol, and 1-butanol is carried out in the pilot plant. Table 4 displays the comparison between experimental operating parameters at steady state condition and simulated results. Feed flowrate, feed composition, distillate flowrate, side flowrate, reflux ratio, and liquid splits in simulation are set the same as experimental data while vapor split are adjusted in order to obtain the best fitting between experimental data and simulated results. The result shows that relative errors of key component of three products are less than 5%.

Table 3

Relative errors between specify product purity and simulate result of key component.

Key component	Specification of key component (mole fraction)	Simulation (mole fraction)	Relative error (%)
Methanol	0.99	0.99	0.00
1-Propanol	0.98	0.97	-1.02
1-Butanol	0.99	0.97	-1.02

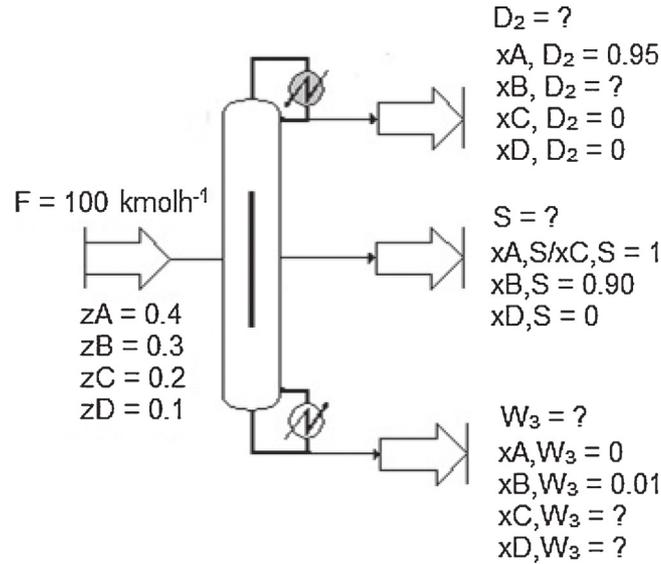


Fig. 7. Specified variables for four-component mixture in divided wall column.

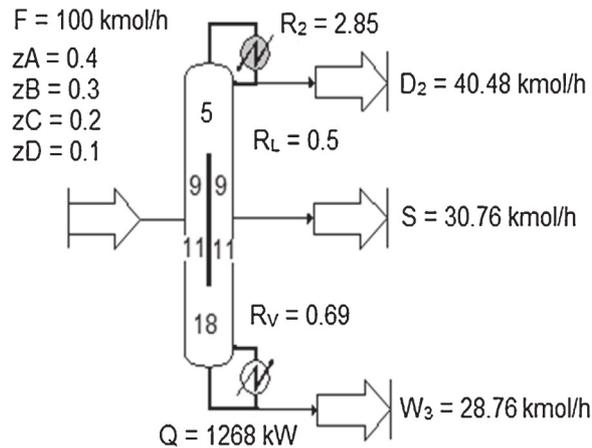


Fig. 8. Design parameters for the divided wall column.

In the simulation, we assume that the heat losses from the column walls are negligible. Thus, heat duty of condenser in experiment is used to compare with heat duty of reboiler in simulation instead of heat duty of reboiler as shown in Table 5. The relative error is equal to 6.31%.

Fig. 9 shows the composition and temperature profiles along the column. This figure indicates that the product purities show very good agreement between experiment and simulation not only for products but also for composition in whole column. The results can indicate two regions: Methanol and 1-propanol are separated in the upper part where mole fraction of 1-butanol is almost zero from a packing height of 2.6–4.4 m. The 1-propanol and 1-butanol are separated in the lower part where methanol is almost zero

Table 4
Relative errors between specified product purity and simulation of key components.

Key component	Specification of products	Simulation	Relative error (%)
Methanol	0.95	0.918	- 3.36
Isopropanol	0.90	0.860	- 4.44
Isopropanol	0.01	0.0096	- 4.00

Table 5
Operation parameters and results for experimental steady-state runs.

Parameters	Experiment	Simulation	Relative error (%)
Feed Stream (kg/h)	6.12	6.12	-
Methanol (wt. %)	0.32	0.32	-
1-Propanol (wt. %)	0.36	0.36	-
1-Butanol (wt. %)	0.32	0.32	-
Distillate stream (kg/h)	1.95	1.95	-
Methanol (wt. %)	0.98	0.97	-1.02
1-Propanol (wt. %)	0.02	0.03	-
1-Butanol (wt. %)	0.00	0.00	-
Side stream (kg/h)	2.12	2.12	-
Methanol (wt. %)	0.00	0.03	-
1-Propanol (wt. %)	0.99	0.97	-2.02
1-Butanol (wt. %)	0.01	0.00	-
Bottom stream (kg/h)	1.93	2.05	-
Methanol (wt. %)	0.00	0.00	-
1-Propanol (wt. %)	0.06	0.05	-
1-Butanol (wt. %)	0.94	0.95	+1.06
Liquid split (-)	0.4	0.4	-
Vapor split (-)	-	0.65	-
Reflux ratio (-)	4	4	-
Heat duty (kW)	2.85	3.03	+6.31
Pressure drop (mbar)	3.1	-	-

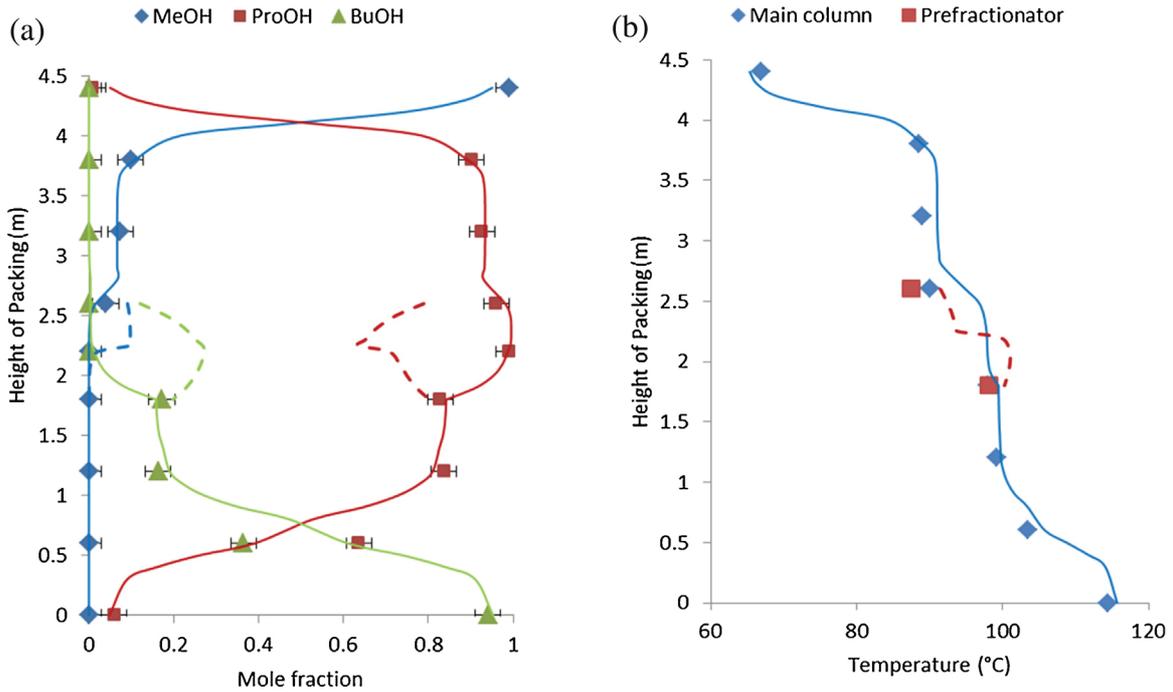


Fig. 9. Composition (a) and temperature (b) profiles of experimental data and simulation results for ternary mixture.

from 0 to 2.2 m. Clearly the upper part is used to separate methanol and 1-propanol and the lower part is used to separate 1-propanol and 1-butanol. In the middle part, 1-propanol reaches a maximum thus it is collected as the side product. Fig. 9 also shows that the content of methanol increases and the content of 1-propanol decreases significantly from 3.8 to 4.4 m and the content of 1-butanol decreases and 1-butanol increases notably at 0–0.6 m.

In all, four steady-state experimental runs are reached of the ternary mixture methanol, 1-propanol, and 1-butanol with different feed compositions, feed flowrates, reflux ratios, liquid splits, and reboiler heat duty. The feed streams can be classified as follows: Case 1 has the same mass fraction of 1-propanol and 1-butanol and a higher mass fraction of methanol; Case 2 has the same mass fraction of methanol and 1-butanol and a higher mass fraction of 1-propanol; Case 3 is represented before with a feed mixture with almost equal mass fraction of all components; Case 4 has the same mass fraction of methanol and 1-propanol and a higher mass fraction of 1-butanol. Cases 1 and 2 have liquid split equal to 0.5, however, Cases 3 and 4 have liquid split of 0.4 and 0.6, respectively. Reflux ratios are determined from simulation results and set into the actual experiments. The pressure drop of each experiment was changed from 2.8 to 6.6 mbar while the heat duty changed from 4.3 kW to 5.4 kW. For all experiments, the product purities show very good agreement between experiment and simulation along the column and at the top and the bottom column, with a maximum deviation of 3%. All results are given in Appendix 1.

4.2.3. Four component mixture

The separation of a four-component mixture in a pilot divided wall column is also investigated. Normally, the divided wall column with a single dividing wall can separate a three component mixture into three high purity products. However, the separation of a mixture of four or more components carried out in a divided wall column achieves only two high purity products and one mixed product. Therefore this section investigates the distribution of components in the divided wall column to see if the fourth

component has an effect on the purity of the products. Two cases are studied. First, the feed stream of the fifth case contains 8% mass fraction methanol, 16% mass fraction isopropanol, 45% mass fraction 1-propanol, and 31% mass fraction 1-butanol. This mixture is prepared because we would like to collect 1-propanol as the side product. Therefore, methanol and isopropanol are collected in the top product and 1-butanol is collected in the bottom product. In the second case, the desired side product is isopropanol. Therefore, the feed stream of the mixture contains 29% mass fraction methanol, 35% mass fraction isopropanol, 22% mass fraction 1-propanol, and 14% mass fraction 1-butanol. The feed flowrate of the studied cases are around 6 kg/h. The liquid split and reflux ratio are 0.5 and 6, respectively. Fig. 10 illustrates the temperature and composition profiles comparison between the experimental data and simulated results

In the first studied case (Fig. 10(a)), a mixture of methanol and isopropanol was obtained as the top product; 1-propanol as the side product with 0.97 mass fraction; and 1-butanol as the bottom product with 0.92 mass fraction. In the second studied case (Fig. 10(a')), methanol is the top product with 72% mass fraction; isopropanol is the side product with 0.967 mass fraction and a mixture of 1-propanol and 1-butanol is the bottom product. The results indicated that a high purity of key components in the side product is obtained. The maximum relative error between experiment and simulation of the key component obtained was less than 10%. Thus it is possible to note that the simulation results and experimental data are in good agreement. The vapor splits of all the studied cases are less than 0.5 as per the trend discussed in section 4.11 when the liquid split is 0.5. The vapor split of Cases 5 and 6 are 0.46 and 0.45, respectively.

5. Sensitivity analysis of divided wall column

In this paper, a design parameter of the divided wall column is determined by our approach. Then, in order to determine the sensitive parameters of divided wall columns, the effects of the structural parameters of the divided wall column such as the

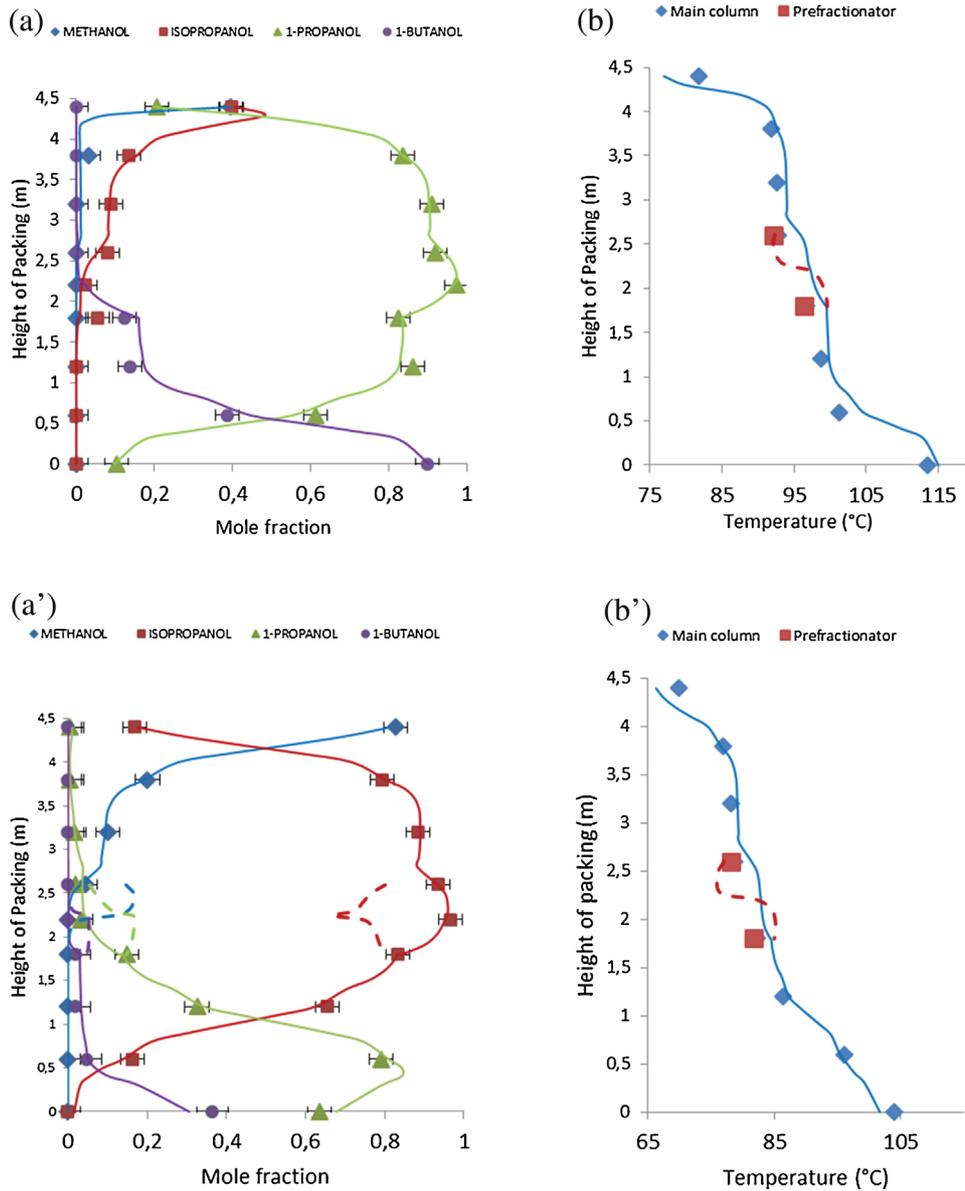


Fig. 10. Composition (a, a') and temperature (b,b') profiles of experimental data and simulation results for the four components mixture.

height of the wall, the vertical position of the wall and number of stages of each section are analyzed. Moreover, this study will also help to analyze the effectiveness of the short cut method to determine the structural parameters minimizing energetic consumption of DWC columns.

Notice that the purity specifications of key components of product streams have to be obtained in all cases. The ternary mixture consisting of benzene 0.33 mole fraction, toluene 0.33 mole fraction and *o*-xylene 0.34 mole fraction is chosen for investigation.

5.1. Effect of the vertical position and height of the wall

The purpose of this section is to investigate how the energy consumption changes when the vertical position and height of the

wall change. Firstly, the vertical position of the wall (Fig. 11) is moved (N3 and N6 change) from the bottom to the top along the column while the height of the wall is constant at 15 stages ($N1 + N2 = N4 + N5$). The total number of stages have not changed ($N3 + N4 + N5 + N6$), and the feed (N1) and side stream (N4) locations are the same as the shortcut results. The position of the dividing wall is marked as zero in Fig. 12 and is the same position that comes from shortcut results.

It is located between stages 9 (N3) and 24 ($N3 + N4 + N5$). In the negative range, the vertical position of the dividing wall is lower than the initial position, and in the positive range, the vertical position of the dividing wall is higher than the initial position. As in Fig. 12, the heat duty of the divided wall column is lower at the initial position $Q_b = 1245$ kW. The lower or higher the position of the wall, the divided wall column has a higher energy demand. The

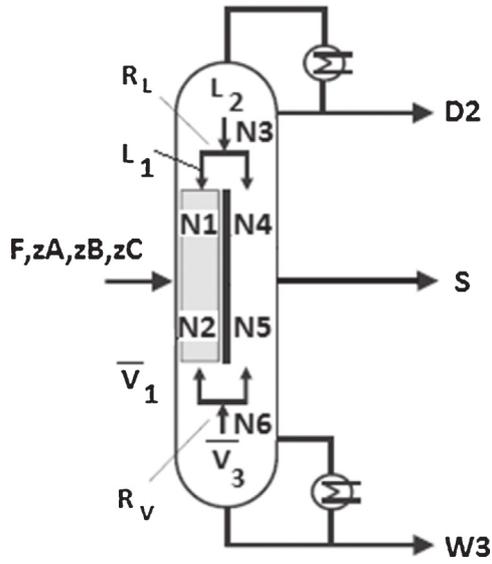


Fig. 11. DWC design parameters.

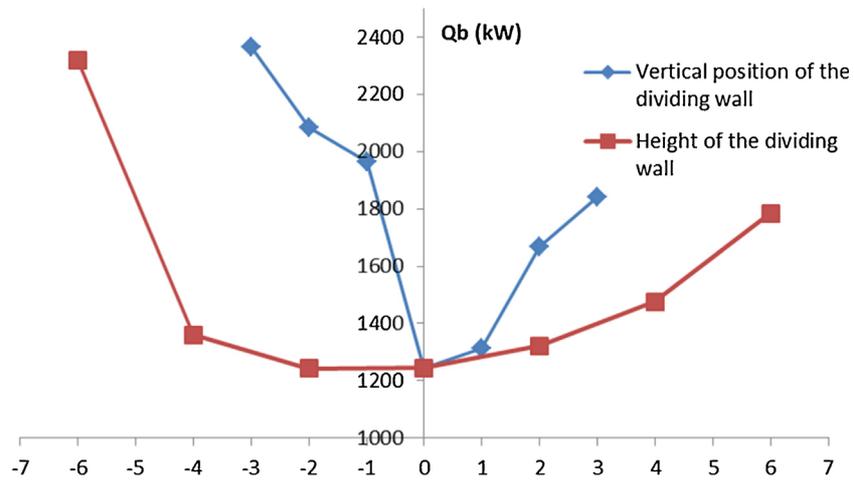


Fig. 12. Effect of the height and vertical position of the wall on the heat duty of reboiler.

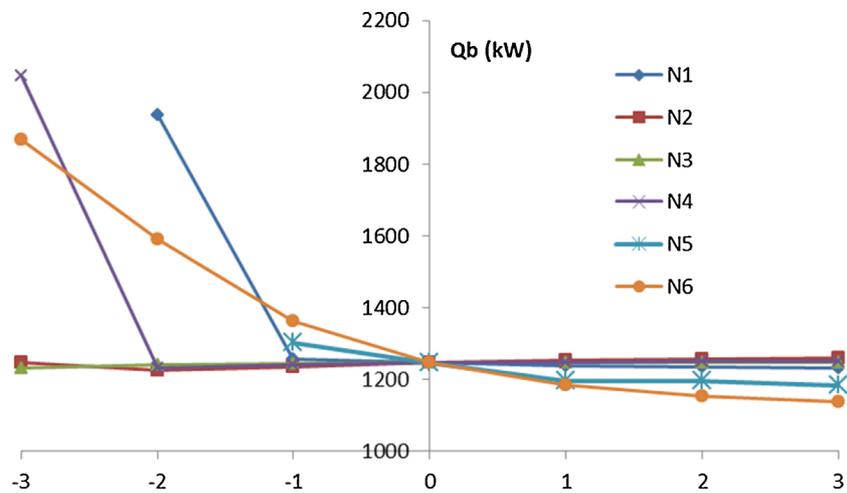


Fig. 13. Effect of number of stages on heat duty of reboiler.

Table 6
Three ternary mixtures with different ESI value.

Mixture	Components A,B,C	ESI	Pressure (atm)
M1	<i>n</i> -Pentane/ <i>n</i> -hexane/ <i>n</i> -heptane	1.04	2
M2	<i>n</i> -Butane/ <i>i</i> -pentane/ <i>n</i> -pentane	1.86	4.7
M3	<i>i</i> -Pentane/ <i>n</i> -pentane/ <i>n</i> -hexane	0.47	2

reboiler heat duty is 2400 kW when the vertical position of the dividing wall is 3 stages lower. It is located between stages 12 and 27. The heat duty of the reboiler is 1850 kW when the vertical position of the dividing wall is 3 stages higher. It is located between stages 6 and 21. The result shows that the vertical position of the dividing wall from the shortcut results requires less energy when the structure changes.

Secondly, the reboiler heat duty is also analyzed with a change of the height of the dividing wall ($N1 + N2 = N4 + N5$ change). The height of the wall is 15 stages, as per the shortcut result, marked

zero in Fig. 12. In the negative range, the number of stages of the dividing wall is decreased while in the positive range, the number of stages of the dividing wall is increased. The feed (N1) and side product (N4) position remains the same as the initial parameters. Fig. 12 shows that the energy consumption of the divided wall column is lower if the number of stage decreases from 15 to 13 stages. The reboiler heat duty is around 1245 kW. The reboiler heat duty increased to 2300 kW when the height of the dividing wall decreases to 9 stages. The reboiler heat duty also increased to 1800 kW when the height of the dividing wall increases to 21 stages. Clearly, our procedure for design of divided wall columns gives good structural parameters; the results are very closed to the minimum energy demand of the column.

5.2. Effect of the number of stages

In this section, the change of the reboiler heat duty is studied when the number of stages of one section has changed while those of other sections are fixed the same as initial parameters. Fig. 13 shows that the heat duty of the reboiler changes with the number

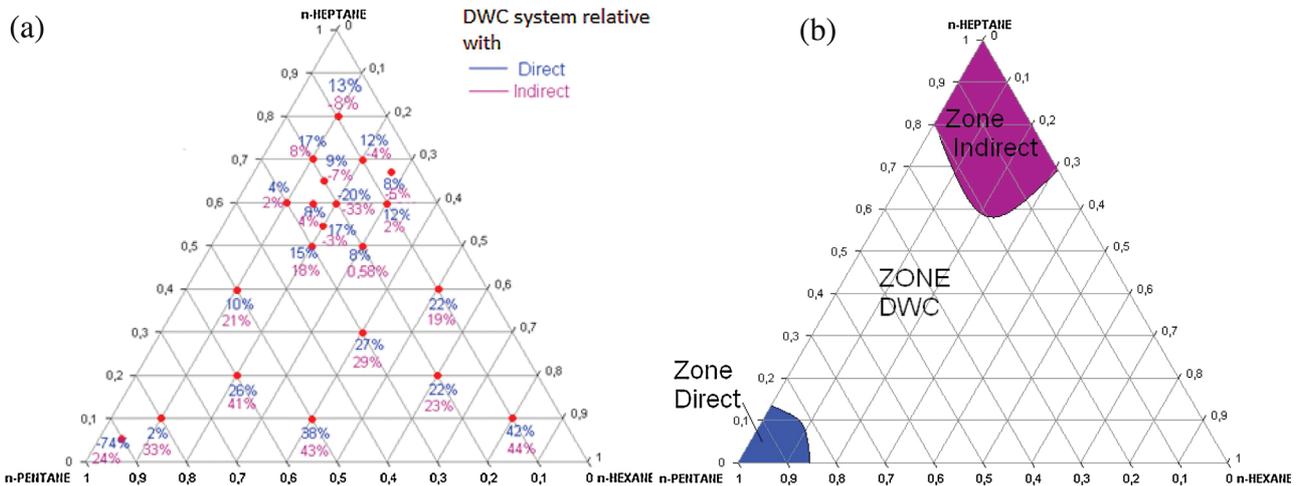


Fig. 14. Energy saving (a) and optimal regions (b) on the composition space for M1.

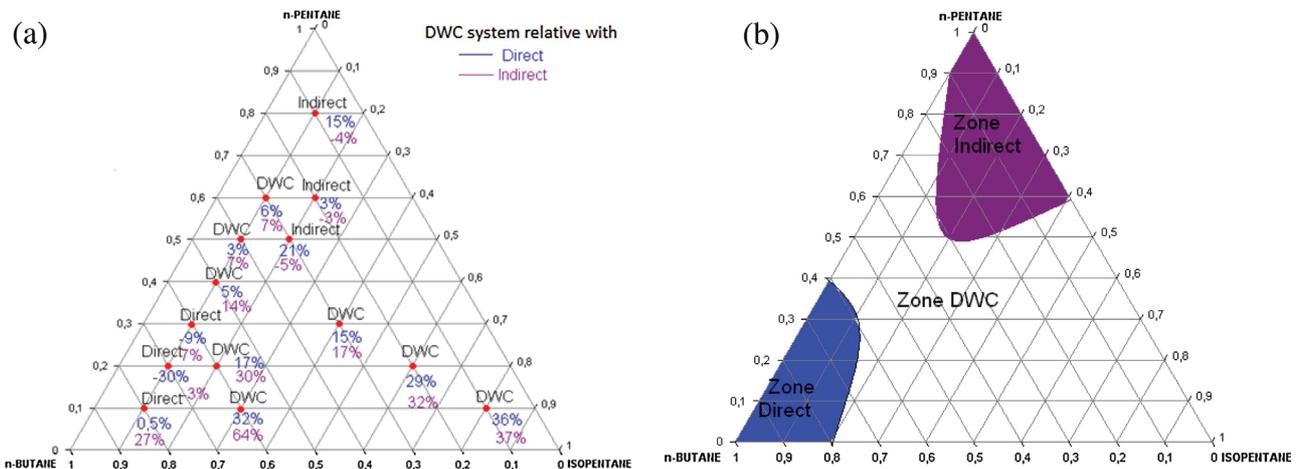


Fig. 15. Energy saving (a) and optimal regions (b) on the composition space for M2.

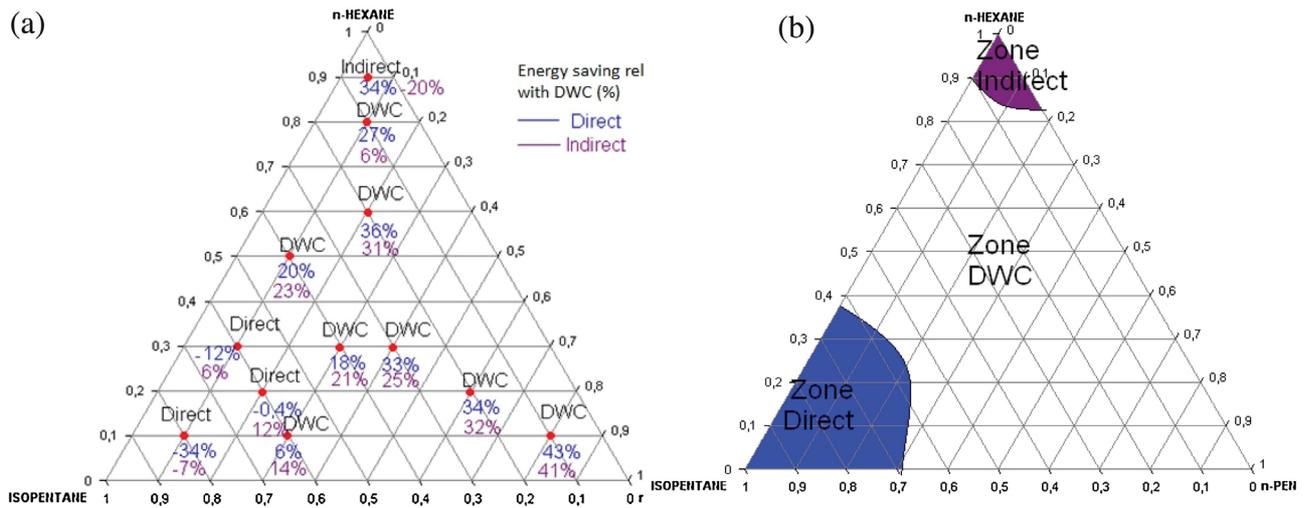


Fig. 16. Energy saving (a) and optimal regions (b) on the composition space for M2.

of stages of each section. The initial parameters from the shortcut results are marked zero as shown in Fig. 13 including N_1 –8 stages, N_2 –9 stages, N_3 –9 stages, N_4 –8 stages, N_5 –7 stages, and N_6 –7 stages. In the negative range, the number of stages decreases and in the positive range, the number of stages increases.

Fig. 13 shows that the heat duty of the reboiler increases when the number of stages of each section decreases. Theoretically, when the numbers of stages decreases, in order to retain the specified product purity, the reflux ratio has to increase. Therefore the reboiler heat duty will increase. In Fig. 13, the numbers of stages in the section 1, 4, and 6 has a significant effect on the heat duty of the reboiler while the number of stages in sections 2 and 3 are not significantly affected. The number of stages in sections 1 and 5 cannot decrease more as the purity specification cannot be reached, regardless of the energy supplied to the column. The number of stages in each section increases, the energy duty of reboiler slightly decreases as shown in Fig. 13. Clearly, it is important to notice that when the number of stages increases, the capital cost of the process will also increase. So, our procedure for design of divided wall columns gives good initial value of number of stage regarding to the minimum energy demand of the column.

5.3. Energy consumption comparison between traditional sequences and DWC system

The idea is to build a ternary diagram and find the boundary where the configuration of distillation is the most economical. In order to do this, the energy used for the traditional distillation column and divided wall column for three ternary mixtures M1, M2 and M3 are considered. Table 6 displays the different values of the ease of separation index (ESI) of the mixture. ESI is defined by Tedder and Rudd [17]: the value ESI equal (or less than, or more than) to 1 that means the split A/B is as difficult as (or more than, or less than) the split B/C.

$$ESI = \frac{K_A K_C}{K_B^2}$$

where K_A , K_B , K_C are volatilities of component A, B, and C.

The feed flowrate is 100 kmol h^{-1} . The operating pressure for each mixture is chosen to ensure the use of cooling water in the

condensers. The lightest component is recovered at the top of the column at 99%, the heaviest component is recovered at the bottom of the column at 99% and the intermediate component is recovered in the middle of the column at 95%. A wide range of feed composition is studied.

Figs. 14 (a), 15 (a), and 16 (a) show the energy saving related to the divided wall column while Figs. 14 (b), 15 (b), and 16 (b) show the distillation zones of the mixture M1, M2, and M3. The red dots are the different feed compositions; the blue numbers and purple numbers are the percentage of energy saving relative to DWC, for direct and indirect sequence (a negative value means an energy gain relative to the column DWC).

The results show that the energy saving of DWC system depends on the amount of intermediate component in the feed. More energy saving consumption in DWC system can be achieved if the mixture has more middle component in the feed composition. For example, in the mixture M1, the energy saving increases from 18% to 44% when the middle component (*n*-Hexane) increases from 20% mole fraction to 80% mole fraction in the feed composition. Kiss et al. [12] which show a procedure to make the right choice between process heat integration or traditional arrangements based on the difference in boiling points between the top and bottom product (ΔT_b), feed flowrate of each component (F_D —product flowrate at the top of the column, F_S —product flowrate of the side product of the column, and F_W —product flowrate at the bottom of the column), and product purity (x_D , x_S , and x_W) conclude the same tendency.

The results show that the distillation zones depends also of the ESI value. The distillation zone for indirect sequence is even larger than the ESI value is high. Indeed, ESI value of 1.86 show the largest zone for indirect sequence zone (Fig. 15b). But there is a different trend for the direct sequence zone. More mixtures should be simulated to conclude with ESI value. The average energy saving for the different values of ESI is 19%, 21% and 24% for mixture M1, M2, and M3 respectively.

Based on the discussion, the divided wall column can save energy duty compared with the traditional sequence. However, the selection of the best arrangement is based on the feed composition and ESI value of the mixture. A representation with a ternary diagram can be useful to select the most economical configuration.

6. Conclusion

The paper has given a procedure for design of DWC system in which both structural and operational parameters of DWC system are rapidly determined. Based on the simulated results, it can be concluded that our procedure can give a good initialization for rigorous simulation. The implementation, startup and operation of a pilot plant to carry out alcohol mixture are achieved in the paper. It indicated that the simulated results are in good agreement with the experimental data at steady state condition and then, the proposed procedure appears to be well adapted to quickly design divided wall columns.

The energy consumption of the traditional arrangements and DWC system are compared. The separations of three different ternary mixtures with different values of ESI are studied. The energy consumption of the DWC system is more favorable than the traditional arrangements if the mixture has more middle component. Clearly, the DWC system is not always the best compared with the conventional arrangements. The selection depends on the feed composition and the ESI value of the mixture.

Acknowledgement

The authors gratefully acknowledge ProSim[®] Company for their support and discussions.

Appendix 1.

FUGK equation for shortcut method of DWC column system

Material balance of divided wall column for ternary mixture

Based on Fig. 2, material balance equations for each component
For the component A

$$Fz_A = D_2x_{A,D_2} + Sx_{A,S} + W_3x_{A,W_3}$$

For the component B

$$Fz_B = D_2x_{B,D_2} + Sx_{B,S} + W_3x_{B,W_3}$$

For the component C

$$Fz_C = D_2x_{C,D_2} + Sx_{C,S} + W_3x_{C,W_3}$$

and

$$x_{A,D_2} + x_{B,D_2} + x_{C,D_2} = 1$$

$$x_{A,S} + x_{B,S} + x_{C,S} = 1$$

$$x_{A,W_3} + x_{B,W_3} + x_{C,W_3} = 1$$

We know the feed flowrate (F) and feed composition (z_A, z_B, z_C). From the above equations, there are twelve unknown variables while there are six equations. Therefore, in order to solve the equations, six of unknown variables must be specified. Unknown variables and specification are listed in Table 2.

We assumed that the component C goes completely to the bottom of the column II and the component A goes completely to the top of the column III. Therefore, we have $x_{C,D_2} = 0$ and $x_{A,W_3} = 0$.

Minimum vapor flowrate of DWC system

Minimum vapor flowrate of column I

In the column I, the recovery ratio of component i in the top product defined as:

$$\tau_{i,T} = \frac{x_{i,D_1}D_1}{Fz_i}$$

Recovery ratio of components A and C should be chosen between:

$$1 > \tau_{A,T} > 1 - \frac{x_{A,S}S}{z_A F}$$

$$0 < \tau_{C,T} < \frac{x_{C,S}S}{z_B F}$$

The recovery ratio of component B is calculated by Stichlmair's equation [16]. It is called the preferred split β_p :

$$\tau_{B,T} = \beta_p = -\frac{(\alpha_A z_A F / \alpha_A - \theta_1) - (\alpha_A z_A F / \alpha_A - \theta_2)}{(\alpha_B z_B F / \alpha_B - \theta_1) - (\alpha_B z_B F / \alpha_B - \theta_2)}$$

where

θ_1, θ_2 —are two roots of Underwood's equation at the minimum reflux condition. They must be following ranges:
 $\alpha_A > \theta_1 > \alpha_B > \theta_2 > \alpha_C$

$$(1 - q_1) = \sum_{i=A}^C \frac{\alpha_i z_i}{\alpha_i - \theta}$$

where

q_1 —Feed quality is the fraction of the feed that is liquid for the column I.

The minimum vapor flow in the prefractionator is determined by Underwood's equation:

$$V_{1,\min} = \sum_{i=A}^C \frac{\alpha_i x_{i,D_1} D_1}{\alpha_i - \theta}$$

and we choose:

$$V_{1,\min} = \max\{V_{1,\min}(\theta_1); V_{1,\min}(\theta_2)\}$$

Minimum vapor flowrate of column II

The interconnecting streams, a saturated vapor stream (V_1) is left from the column I and a saturated liquid stream (L_1) is returned into the column I, can be modified by equivalent feed stream with a superheated vapor condition. Therefore, the quality of the feed for the column II:

$$q_2 = \frac{\bar{L}_2 - L_2}{D_1} = -\frac{V_{1,\min} - D_1}{D_1}$$

At the minimum reflux condition, the Underwood's equation can be written as follows:

$$(1 - q_2) = \sum_{i=A}^C \frac{\alpha_i x_{i,D_1}}{\alpha_i - \theta'}$$

where

θ'_1, θ'_2 are two roots of Underwood's equation at the minimum reflux condition. They must be following ranges: $\alpha_A > \theta'_1 > \alpha_B > \theta'_2 > \alpha_C$

The minimum vapor flow in the column II can be estimated by:

$$V_{2,\min} = \sum_{i=A}^C \frac{\alpha_i x_{i,D_2} D_2}{\alpha_i - \theta'}$$

Therefore, we can choose

$$V_{2,\min} = \max\{V_{2,\min}(\theta''_1); V_{2,\min}(\theta''_2)\}$$

Minimum vapor flowrate of column III

The stream is fed to the column III that is connected from interconnecting streams are a saturated liquid stream (\bar{L}_1) and a saturated vapor stream (\bar{V}_1), can be modified by equivalent feed stream with a sub-cooled liquid condition. The quality of the feed for the column III can be calculated by:

$$q_3 = \frac{\bar{L}_3 - L_3}{W_1} = \frac{V_{1,\min} - D_1 + q_1 F}{W_1}$$

At the minimum reflux condition, the Underwood's equation can be written as follows:

$$(1 - q_3) = \sum_{i=A}^C \frac{\alpha_i x_{i,W_1}}{\alpha_i - \theta''}$$

where

θ''_1, θ''_2 are two roots of Underwood's equation at the minimum reflux condition. They must be following ranges: $\alpha_A > \theta''_1 > \alpha_B > \theta''_2 > \alpha_C$

The minimum vapor flow in the column III can be determined by:

$$\bar{V}_{3,\min} = - \sum_{i=A}^C \frac{\alpha_i x_{i,W_3} W_3}{\alpha_i - \theta''}$$

Therefore, we choose

$$\bar{V}_{3,\min} = \max\{\bar{V}_{3,\min}(\theta''_1); \bar{V}_{3,\min}(\theta''_2)\}$$

Minimum vapor flow rate of DWC system

The minimum vapor flowrate of DWC system should be chosen by Halvorsen and Skogestad [7,10]

$$V_{\min,DWCs} = \max\{V_{2,\min}, \bar{V}_{3,\min} + (1 - q_1)F\}$$

Number of stages for each section of DWC system

The minimum reflux ratio of the DWC system can be calculated as follows:

$$R_{\min} = \frac{V_{\min,DWCs}}{D_2} - 1$$

The operating reflux ratio of the DWC system can be chosen as:

$$R = \left(\frac{1,2}{1,5}\right) R_{\min}$$

The liquid and vapor splits between prefractionator and main column can be defined as:

$$R_L = \frac{L_1}{L_2}$$

$$R_V = \frac{\bar{V}_1}{\bar{V}_3}$$

Starting from the structure as shown in Fig. 2, an evaluation of NET for each section and reflux ratio for each column are computed based on traditional shortcut method of Fenske, Underwood, and Gilliland and Kirkbride equations by Kister [9]. The minimum number of stages can be determined by Fenske equation for

column i :

$$N_{i,\min} = \frac{\ln(S)}{\ln(\alpha_{av})}$$

where S is given by equation

$$S = \left(\frac{x_{LK}}{x_{HK}}\right)_{D_i} \left(\frac{x_{HK}}{x_{LK}}\right)_{W_i}, i = \frac{1}{3}$$

Then, we calculate number of stages by using Gilliland equation:

$$Y = 0,75 \cdot (1 - X^{0,5668})$$

where X and Y are given by equation

$$X = \frac{R - R_{\min}}{R + 1}$$

$$Y = \frac{N - N_{\min}}{N + 1}$$

Feed location in each column can be calculated by Kirkbridge equation

$$\left(\frac{N_R}{N_S}\right)_i = \left\{ \left(\frac{z_{HK}}{z_{LK}}\right)_i \left(\frac{x_{LK,W_i}}{x_{HK,D_i}}\right)^2 \frac{W_i}{D_i} \right\}^{0,206}$$

Appendix 2.

Experimental results for ternary mixture

Operating parameters and results for experimental steady-state runs

Parameters	Case 1	Case 2	Case 3	Case 4	
Feed (kg/h)	5.41	5.77	6.12	5.97	
Methanol (wt. %)	0.4	0.29	0.32	0.3	
1-Propanol (wt. %)	0.3	0.46	0.36	0.24	
1-Butanol (wt. %)	0.3	0.25	0.32	0.46	
Distillate (kg/h)	2.66	2.00	1.95	1.80	
Methanol (wt. %)	0.814	0.85	0.98	0.93	
1-Propanol (wt. %)	0.186	0.15	0.02	0.07	
1-Butanol (wt. %)	0	0	0	0	
Side stream (kg/h)	1.038	2.17	2.12	0.918	
Methanol (wt. %)	0	0	0	0	
1-Propanol (wt. %)	0.995	1	0.998	0.96	
1-Butanol (wt. %)	0.005	0	0.002	0.04	
Bottom stream (kg/h)	1.872	1.7	1.93	3.144	
Methanol (wt. %)	0	0	0	0	
1-Propanol (wt. %)	0.21	0.114	0.06	0.19	
1-Butanol (wt. %)	0.979	0.886	0.94	0.81	
Liquid split (-)	0.5	0.5	0.4	0.6	
Reflux ratio (-)	3	6	4	4	
Heat duty (kW)	5.17	5.1	4.3	5.4	
Heat condenser (kW)	2.67	2.2	2.24	2.5	
Pressure drop (mbar)	2.8	6.6	3.1	2.4	
Relative error (%) of	Total	-2.92	-1.73	1.96	-1.80
mass balances	Methanol	0.47	-1.59	2.40	7.51
	1-Propanol	2.12	-0.11	-3.04	-7.12
	1-Butanol	-12.48	-4.88	7.15	2.81

Composition and temperature profiles of experimental data and simulation results for ternary mixture Fig. A1-A4 .

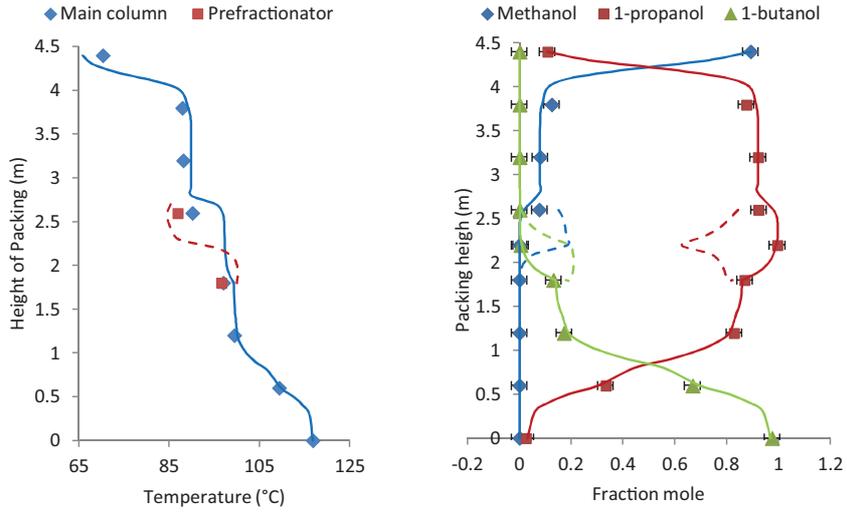


Fig. A1. Case 1.

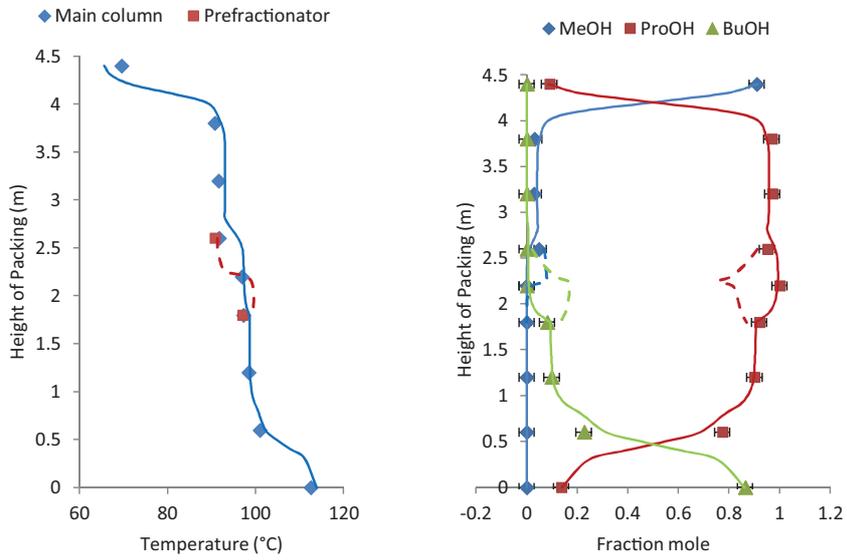


Fig. A2. Case 2.

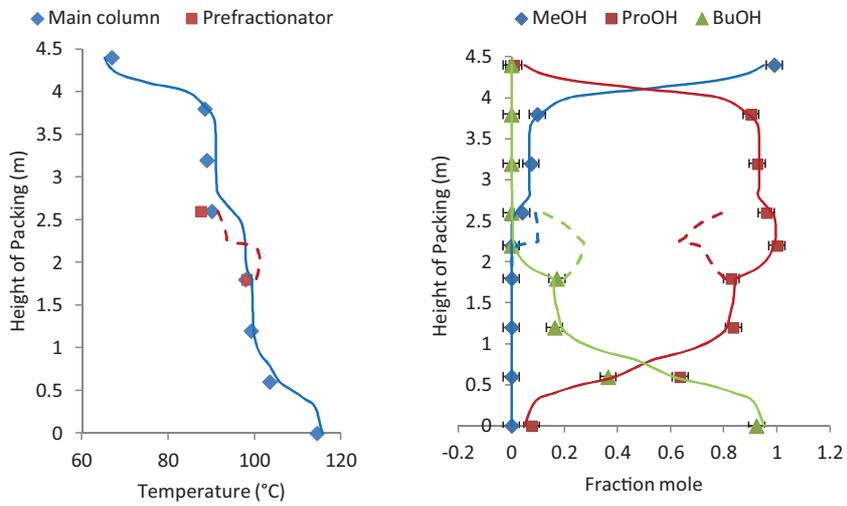


Fig. A3. Case 3.

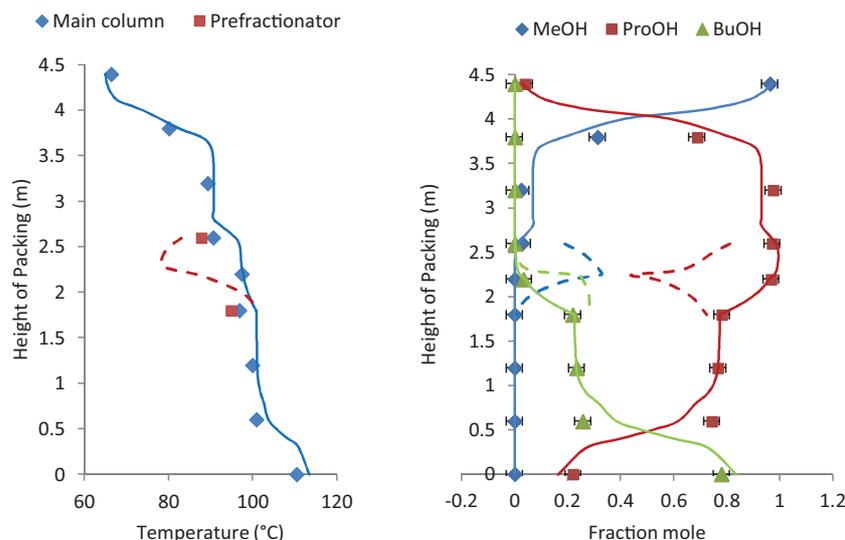


Fig. A4. Case 4.

References

- [1] K.A. Amminudin, R. Smith, Design and optimization of fully thermally coupled distillation columns: part 2: application of dividing wall columns in retrofit, *Chem. Eng. Res. Des.* 79 (7) (2001) 716–724.
- [2] K.A. Amminudin, R. Smith, D.C. Thong, G.P. Towler, Design and optimization of fully thermally coupled distillation columns: Part 1: Preliminary design and optimization methodology, *Chem. Eng. Res. Des.* 79 (7) (2001) 701–715.
- [3] H. Becker, S. Godorr, H. Kreis, Partitioned distillation columns—why when and how, *Chem. Eng.* 108 (1) (2001) 68–74.
- [4] C. Triantafyllou, R. Smith, The design and optimization of fully thermally coupled distillation columns, *Inst. Chem. Eng.* 70 (March (A2)) (1992) 118–132.
- [5] C.J. Calzon-McConville, M.B. Rosales-Zamora, J.G. Segovia-Hernandez, S. Hernandez, V. Rico-Ramírez, Design and optimization of thermally coupled distillation schemes for the separation of multicomponent mixtures, *Ind. Eng. Chem. Res.* 45 (2) (2006) 724–732.
- [6] K.T. Chu, L. Cadoret, C.C. Yu, J.D. Ward, A new shortcut design method and economic analysis of divided wall columns, *Ind. Eng. Chem. Res.* 50 (15) (2011) 9221–9235.
- [7] I.J. Halvorsen, S. Skogestad, Minimum energy consumption in multicomponent distillation. 3. More than three products and generalized Petlyuk arrangements, *Ind. Eng. Chem. Res.* 42 (3) (2003) 616–629.
- [8] Hao Ling, William L. Luyben, New control structure for divided wall columns, *Ind. Eng. Chem. Res.* 48 (13) (2009) 6034–6049.
- [9] Henry Z. Kister, *Distillation Design*, McGraw-Hill, 1992.
- [10] I.J. Halvorsen, S. Skogestad, Minimum energy consumption in multicomponent distillation: 2. Three-product Petlyuk column, *Ind. Eng. Chem. Res.* 42 (2003) 605–615.
- [11] Y.H. Kim, Structural design and operation of a fully thermally coupled distillation column, *Chem. Eng. J.* 85 (2) (2002) 289–301.
- [12] A.A. Kiss, S.J. Flores Landaeta, C.A. Infante Ferreira, Towards energy efficient distillation technologies—making the right choice, *Energy* 47 (1) (2012) 531–542.
- [13] Nelly Ramirez-Corona, Arturo Jiménez Guíérrez, Angel Castro Agüero, Vicente Rico-Ramirez, Optimum design of Petlyuk and divided wall distillation systems using a shortcut model, *Chem. Eng. Res. Des.* 88 (2010) 1405–1418.
- [14] Noori Sotudeh, Bahram Hashemi Shahraki, A method for the design of divided wall columns, *Chem. Eng. Technol.* 30 (9) (2007) 1284–1291.
- [15] N. Sotudeh, B.H. Shahraki, Extension of a method for the design of divided wall columns, *Chem. Eng. Technol.* 31 (1) (2008) 83–86.
- [16] J. Stichlmair, *Distillation and rectification*, Ullmann's Encyclopedia of Industrial Chemistry B3, (1988) 4-1-4-94.
- [17] D.W. Tedder, D.F. Rudd, Parametric studies in industrial distillation: Part I. Design comparisons, *AIChE J.* 24 (2) (1978) 303–315.
- [18] D.B. Van Dongen, M.F. Doherty, Design and synthesis of homogeneous azeotropic distillations. 1. Problem formulation for a single column, *Ind. Eng. Chem. Fundam.* 24 (4) (1985) 454–463.