An Effective Method for Establishing the Stage and Reflux Requirement of Three-product Dividing Wall Columns+

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This paper introduces an effective method for determination and optimisation of stage and reflux requirement of a three-product dividing wall column, arranged within a commercial software package by combining a fundamentally sound shortcut method providing reliable initial guesses and a detailed, four-column model, which appeared to be the most flexible among possible configurations, for both steady state and dynamic simulations. The applicability and usability of the method is demonstrated using an industrially relevant, platformate splitter application in an aromatics plant of a petroleum refinery as the base case. Results of this study indicate that compared to actual, two-columns-in-series configuration, a dividing wall column requires approximately 43 % less energy to deliver three fractions at required product specifications.

Key words:
Distillation, energy saving, thermally coupled columns, dividing wall columns

Introduction

Energy used for continuous distillation processes comprises approximately 40 % of total energy consumption in the chemical process industry.[1] Indeed, distillation is both most widely used and most energy-intensive among the methods for separation of liquid mixtures.[2] Also, there is no doubt that distillation is the most mature among separation technologies. However, the fact that it was invented and developed by experience alone,[3] during the times when related utilities consumption was not a concern, makes distillation still an amenable subject for process intensification.

The most impressive development in this respect is implementation of the so-called dividing wall column (DWC) concept, which not only leads to energy saving but also to capital saving. Upon establishing itself as packed column for separations of various chemicals performed mainly under vacuum, DWC is now making an inroad into the refinery world dominated by large tray columns,[2] where in many situations multicomponent feeds are separated into three products. In case of sharp product specifications, this often means use of two columns connected in series. Fig. 1 shows schematically a typical (indirect sequence) two-columns-in-series arrangement for obtaining three pure products from a three-component feed. Where appropriate, this sequence can be realized as a so-called Petlyuk column, which, as shown in Fig. 2, consists of a prefractionator column fully thermally coupled to the main column. This configuration implies that only one condenser and one reboiler are sufficient, which means a reduction in the number of main equipment and accompanying investment costs. A DWC, shown schematically in Fig. 3, is a single-shell thermodynamic equivalent of a fully thermally coupled column, which allows separation of three or more components into high purity products within one shell. This is achieved by using a vertical partition wall that divides mainly the central

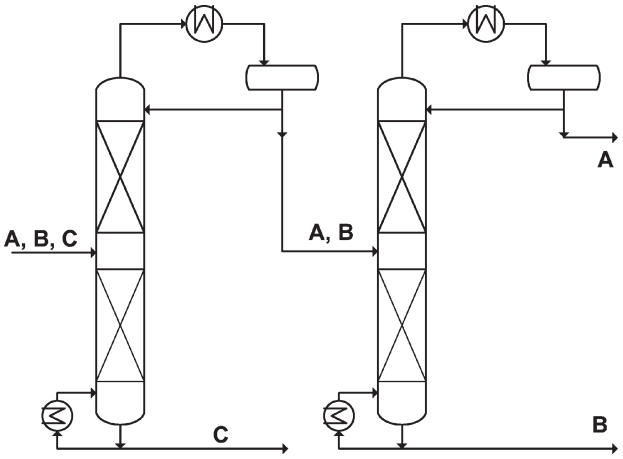

Fig. 1 – *Conventional three-component separation arrangement*

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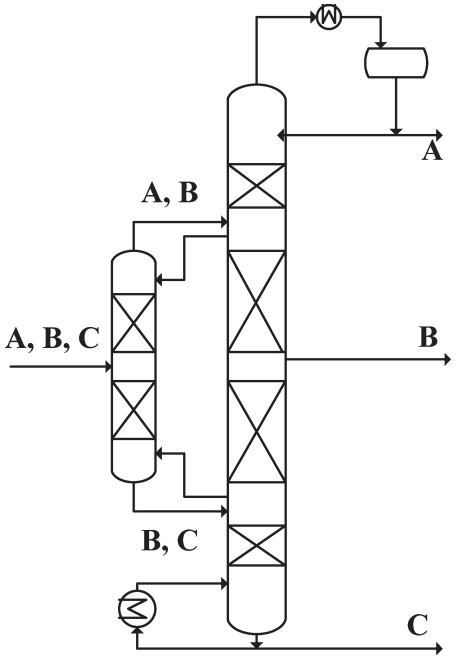


Fig. 2 – *Petlyuk arrangement with thermally coupled prefractionator and main column*

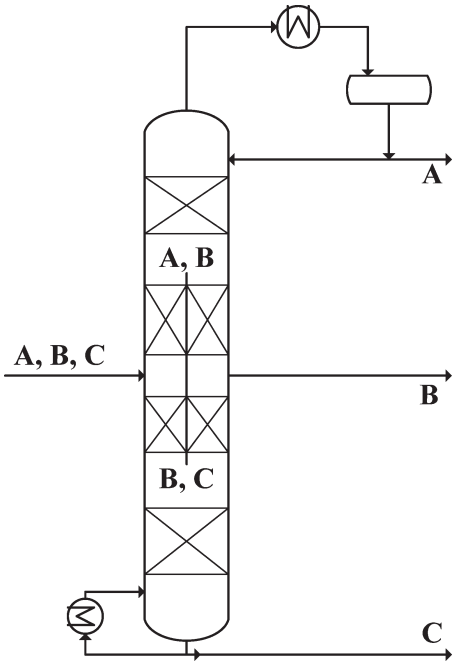


Fig. 3 – *A three-product DWC arrangement*

part of the column into prefractionator and main column sections. In each section, two components with greatest difference in volatility are separated, while others are allowed to distribute.

The overall thermodynamic efficiency of Petlyuk column and its thermodynamic equivalent DWC is significantly larger than that of the conventional configuration. This is mainly because its configuration allows avoidance of the entropy of mixing formation caused by composition differences between feed stream and feed stage, as well as minimization of re-mixing effect of middle boiling components in separation of mixtures with more than 2 components,[4] which is effectively a direct loss of separation work. Although the idea of thermal (heat) coupling is older, F. Petlyuk was instrumental in its implementation and therefore, this and more complex fully thermally coupled columns are known as Petlyuk columns.[5] Most of the work, which Petlyuk and co-workers have devoted over the years to distillation theory and its application in optimal design of columns and column configurations or sequences, can be found in a recently published book by Petlyuk.[6]

Recent advances, including non-welded partition walls that can be placed in off-centre positions to allow optimal design for unsymmetrical (with respect to composition), and partly vaporized feeds, are described elsewhere.[7,8] Indeed, as mentioned by Olujić *et al.*[2] and Dejanović *et al.*,[5] implementation of non-welded wall, which allows maximization of the application window for DWC and allows building DWCs for obtaining four or even more products, can be considered as a milestone in the development of this energy-, space- and capital-saving technology.

A number of papers have been published on the subject of DWC design, with various degrees of complexity. Short-cut design methods, based on some combination of Fenske-Underwood-Gilliland--Kirkbride set of equations, are presented in papers by Triantafyllou and Smith,[4] Muralikrishna *et al.,*[9] Sotudeh and Shahraki[10,11] and Ramirez-Corona *et al.*[12]

Amminudin and Smith[13] and Amminudin *et al.*[14] proposed a semi-rigorous design method based on equilibrium stage composition concept, previously applied for synthesis of azeotropic distillation sequences.

Other authors propose rigorous optimization based approach, to simultaneously obtain optimal operating parameters and optimal structure leading to minimal total annualized costs. Such approaches are presented in papers by Dünnebier and Pantelides,[15] Wenzel and Rohm,[16] Gomez-Castro *et al.*[17] and Gutierrez-Antono and Briones-Ramirez.[18]

Although there are in open literature many publications addressing simulation of the performance of conventional, three-product DWCs, there is still no standard model, and performing such simulations requires experience and certain skill, required to adapt in an effective way provisions available in commercial software to arrange a configuration resembling the performance of a DWC. These

are more or less computationally demanding, depending on the configuration chosen. A thorough evaluation of literature has indicated that a four-column model allows for maximum flexibility regarding specifications for different column sections, and vapour and liquid splits.[5] Indeed, it appears that this model reflects the actual situation best, but it appeared to be most difficult to initialize. As demonstrated further in this paper, by utilising a simple but fundamentally sound short-cut method for this purpose in conjunction with a detailed, sequential four-column model, an effective simulation tool was obtained that allows determination and optimization of the stage and reflux requirement of a three-product dividing wall column.

The objective of this paper is to demonstrate the usability of the proposed model arranged using provisions available within commercial simulation software for performance evaluation and conceptual design of a DWC column for a refinery application, concerning separation of a multicomponent aromatics-rich mixture into three fractions, according to given specifications.

Design case

As it can be seen in a comprehensive state-of-the-art review on DWC technology,[5] separation of three-component aromatics mixtures, mainly benzene-toluene-xylene (BTX) into individual products is one of the prominent examples used in academic papers regarding various aspects of heat coupling and DWC performance simulation. However, this is an idealization with respect to the actual situation as encountered in aromatic complexes of petroleum refineries. According to actual plant data delivered for the purposes of this simulation study by INA Sisak Refinery, Croatia, a member of MOL Group, the product stream from the platforming process (platformate) comprises approximately 40 components. This complex feed mixture is separated into three fractions by employing a conventional indirect two-column sequence, shown in Fig. 1. The product streams and the related specifications: the distillate (C5-C6) is a gasoline fraction containing less than $w = 1.5$ % of benzene, the middle boiling product stream is a benzene-rich cut (BRC) containing minimum $w = 68$ % benzene, and the heavy end (T-plus or heavy reformate) containing no more than $w = 0.5$ % of benzene, includes toluene and all heavier components.

For the sake of convenience, we reduced this number to a 15-component mixture by lumping components of similar volatilities, while maintaining those of economic interest, like benzene, toluene, ethylbenzene, xylenes, etc. Table 1 shows the composition of the feed mixture as well as the compositions of three product streams as obtained by simulation, performed in ChemCAD. The next step was to set up a model and simulate performance of a DWC that was expected to comply with specifications, i.e. the product stream compositions given in Table 1.

Table 1 – *Base case feed and product compositions*

Stream name	Component tag	Feed	C5-C6	BRC	T-plus
Total flow [t h^{-1}]		31.74	6.94	3.70	21.10
Component mass fractions [–]					
N-Butane	A	0.019	0.088	0.000	0.000
Isopentane	B	0.064	0.291	0.000	0.000
N-Pentane	C	0.045	0.206	0.000	0.000
2-Methylpentane	**D**	**0.080**	**0.351**	**0.026**	**0.000**
N-Hexane	E	0.043	0.050	0.270	0.000
Benzene	**F**	**0.086**	**0.013**	**0.680**	**0.005**
3-Methylhexane	G	0.020	0.000	0.024	0.026
Toluene	**H**	**0.247**	**0.000**	**0.000**	**0.373**
Ethylbenzene	I	0.035	0.000	0.000	0.053
P-Xylene	J	0.042	0.000	0.000	0.064
M-Xylene	K	0.122	0.000	0.000	0.183
O-Xylene	L	0.055	0.000	0.000	0.083
M-Ethyltoluene	M	0.047	0.000	0.000	0.071
1-3-5-Trimethylbenzene	N	0.077	0.000	0.000	0.116
1-4-Diethylbenzene	O	0.017	0.000	0.000	0.025

DWC performance modelling

Regarding the simulation approach, as indicated in Fig. 4 a DWC has significantly more design variables than a conventional distillation column. Additional design variables, when compared to a conventional side-stream column are the number of stages in two sections of prefractionator, and the liquid and vapour split ratios.

That is why a good initialization method (shortcut model) is essential to give rough design estimates, which can be used to set up more detailed simulation. A thorough evaluation of literature[5] indicated that this can be accomplished in a rather simple, but effective way by employing a fundamentally sound short-cut method introduced recently by Halvorsen[19] and Halvorsen and Skogestad.[20,21]

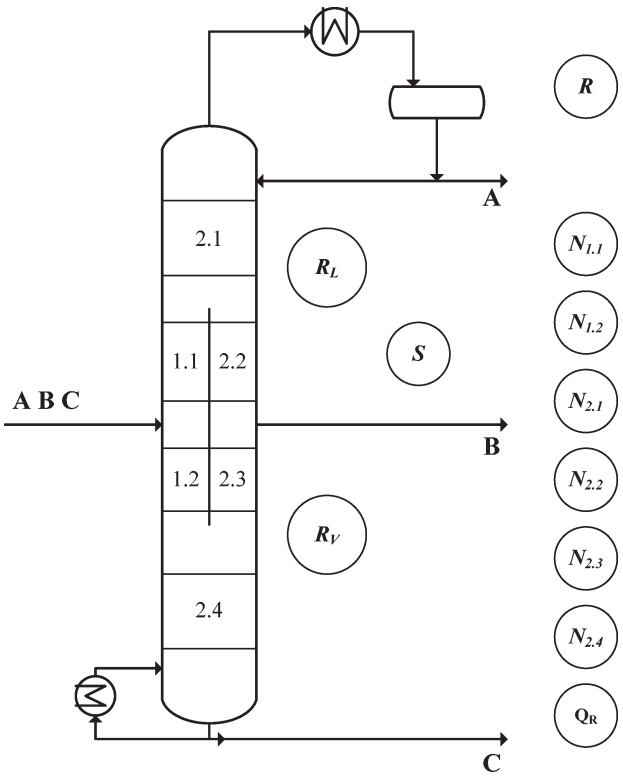


Fig. 4 – *DWC design parameters*

V_{min} diagram method

Halvorsen and Skogestad[21] proposed a design method based on Underwood's equations to determine minimum vapour and liquid flows needed to perform all binary separations of a specified feed mixture, which constitute product splits occurring in each subcolumn, assuming an infinite number of theoretical stages.

Their basic postulate is that the minimum vapour flow required for separation of *n* components feed into *n* pure products in any conceivable distillation arrangement, corresponds with that required for the most difficult binary split. Halvorsen's method is available as a commercial software package, which can be used to gain insight into the requirements to separate *n*-component feed mixture into *n* products. Being a constant relative volatility model, this method is obviously limited to analysis of ideal zeotropic mixtures. However, V_{min} diagram can also be developed by performing a series of binary split calculations using rigorous distillation models which are a common part of commercial process simulators, as described elsewhere.[20] This was the approach used in this case, leaving only the assumption of an infinite number of stages as simplification.

To proceed accordingly, the feed mixture shown in Table 1 was represented by three key components: 2-methyl pentane, benzene and toluene. Corresponding pure components mole fractions and representative K-values are shown in Table 2.

Table 2 – *Key components used to construct V_{min} diagram*

Tag	Component	Mole fraction	K-value on feed stage	Product
A	2-methylpentane	0.2516	1.6298	**D**
B	benzene	0.1058	0.9417	**S**
C	toluene	0.6426	0.4154	**B**

In order to determine the key points of the V_{min} diagram, a series of three binary distillation calculations of the specified feed were performed. All calculations were performed using ChemCADs TOWER module – rigorous equilibrium distillation model, in conjunction with SRK method for calculation of vapour-liquid equilibrium. The specified recoveries of key components, and calculated (required) vapour and distillate flow rates, together with the minimum number of stages and feed stage location, are shown in Table 3. To approximate conditions of an infinite number of stages, as required by V_{min} diagram, the number of stages for each simulation was set to at least $4N_{min}$, as calculated by Fenske equation. This is the value suggested by Halvorsen,[19] and was also confirmed by simulation, showing no decrease in reboiler heat duty by further increase in the number of stages. The values shown in Table 4 were used to construct V_{min} diagram, shown in Fig. 5.

Table 3 – *V_{min} diagram values*

		Specified		Calculated			
Section	Cut	Recoveries		V_T/F	D/F	N_{min}	N_f
$\mathbf{C_1}$	$\mathbf{P_{A/C}}$	r_D(C) = 0.01	r_B(A) = 0.01	0.62	0.310	9	19
$\mathbf{C_{2.1}}$	$\mathbf{P_{A/B}}$	r_D(B) = 0.01	r_B(A) = 0.01	1.32	0.253	20	40
$\mathbf{C_{2.2}}$	$\mathbf{P_{B/C}}$	r_D(C) = 0.01	r_B (B) = 0.01	0.93	0.385	13	28

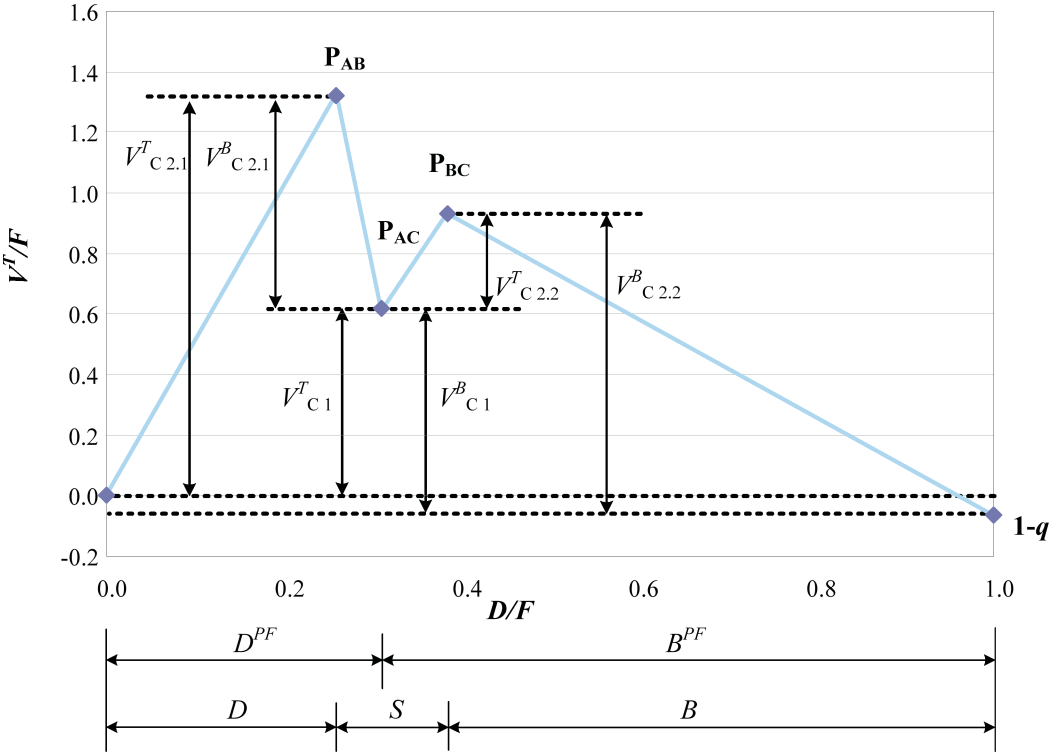


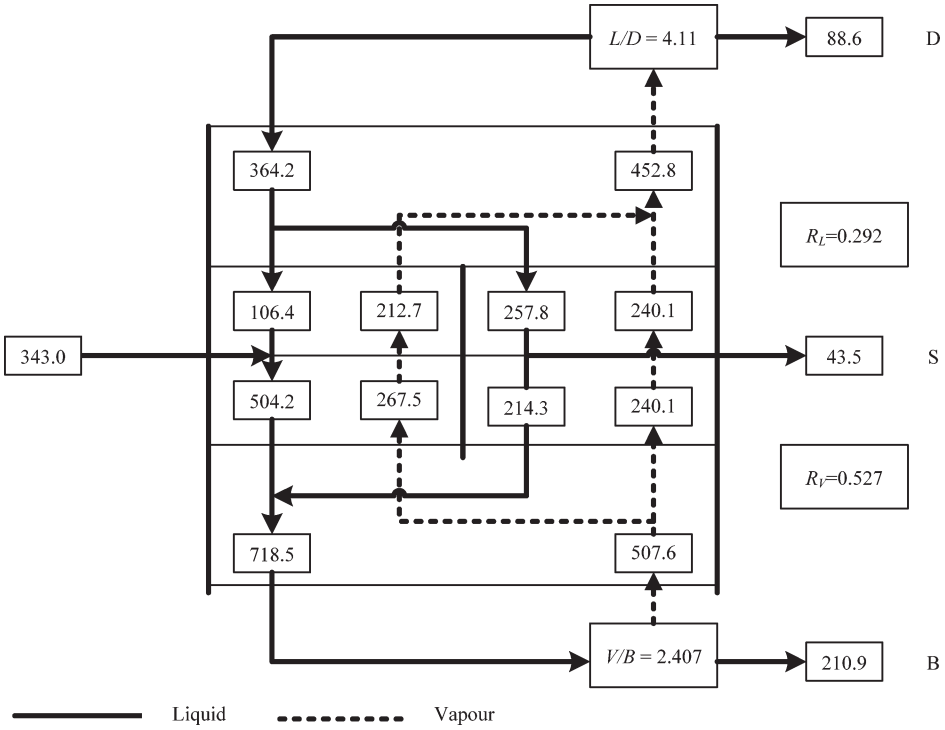
Fig. 5 – V_{min} *diagram for three-product case*

Table 4 – *Base case and DWC simulation results*

		Column 1	Base case	Column 2	DWC
Product specifications					
C5-C6	*w* %		1.31		1.30
BRC	*w* %		68.00		68.12
Heavy reformate	*w* %		0.51		0.50
Top pressure	bar	1.7		2.7	2.7
Number of stages	–	40		38	64
Feed stage	–	20		19	36
Reflux ratio	–	1.70		2.39	2.80
Condenser duty	MW	–3.16		–2.51	–2.76
Total	MW		–5.67		–2.76
Reboiler duty	MW	3.55		2.63	3.50
Total	MW		**6.18**		**3.50**

From the diagram, all necessary information on internal flow rates of vapour and liquid in different column sections can easily be calculated, as described in detail in a paper by Halvorsen and Skogestad,[21] as well as minimum reboiler vapour flow rate, denoted as the highest peak on the diagram. Calculated internal vapour and liquid flow rates, together with calculated vapour/liquid splits, reflux/boil-up ratio and product flow rates are shown in Fig. 6. These values can be used as highly reliable initial estimates of parameters of the rigorous model.

One should realize that the data summarised in Fig. 6 also provides a basis for conceiving the internal configuration of a DWC. For convenience, the partition wall here is placed in the middle. However, a closer inspection of liquid and vapour flow rates in each section indicates that this may not be the case. Namely, the liquid flow leaving the rectification section on prefractionator column (feed) side is much smaller than that on main column side. However, a roughly factor-three larger feed stream is a slightly sub-cooled liquid (q = 1.06), which means that the specific liquid load of the stripping section on prefractionator side will be much larger than that on main column side. A significant part of this liquid will result from direct condensation of ascending vapour immediately below the feed stage. With somewhat larger vapour load at the bottom of the stripping section and a roughly factor-two larger liquid load, a considerably larger sectional area will be required below the feed on the prefractionator side. This is a good indication of initial configuration, which can be completed quantitatively upon determining the actual stage requirement of each section using detailed calculations.



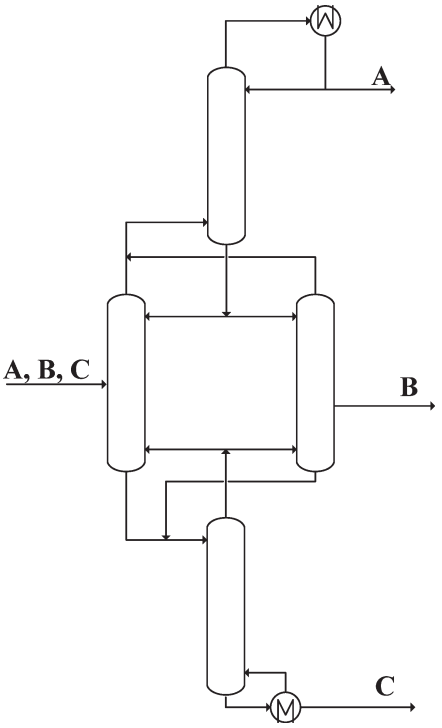
F i g . 6 – *Internal values of molar flow rates and phase splits according to V_{min} diagram, initial values for rigorous simulation*

Detailed model calculations

Detailed, tray-by-tray calculation must be performed to determine the actual number of stages in each section (at $R > R_{min}$), and to get a converged column profile to serve as a basis for dimensioning and cost estimation, which is not in the scope of this paper. The goal was to see whether widely available commercial process simulators could be used for this task.

Process simulators such as ChemCAD, Aspen or HYSYS still do not incorporate even the simplest, three-product DWC as a distinct model. Instead, a DWC can be modelled as a sequence of simple columns. Several approaches can be used, ranging from one to four column models, each exhibiting some good and bad sides.[5] As a rule, one-column pump-around models have better convergence properties than models with more columns. Four-column model is more difficult to initialize and exhibits a slower convergence, but it is easier to inspect obtained results and perform sizing, considering that each column section is represented individually. Fig. 7 shows schematically four-column model configuration implemented in ChemCAD to simulate the performance of the DWC considered in this study.

In simulating DWC according to initial design parameters from $V_{\min}$ diagram, the first step was to restore feed composition to full 15 components. The initial values for the number of stages in each



F i g . 7 – *Four-column DWC model*

section and heat coupling locations were taken from *short-cut* simulation, as calculated by Fenske equation. Side-product flow rate was taken from base case simulation. To obtain compositions of interconnecting streams needed for initialization, the

most straightforward approach is to first specify the vapour and liquid streams flowing from prefractionator to main column, which composition can easily be estimated from specified product distribution in the prefractionator. The flow rate of streams is set according to the V_{min} diagram, and temperatures set at dew point for vapour stream, and boiling point for liquid stream, at governing pressure. Condenser and reboiler were initially specified using reflux ratio and boil-up ratio calculated from V_{min} diagram, and the remaining interconnecting streams compositions were calculated by individually simulating all four column sections.

Once the initial profile of the column was obtained, condenser and reboiler were set to automatically maintain required benzene content in distillate and bottom product, respectively.

The next step was to determine the actual number of equilibrium stages in each section. This was done by incrementally reducing the number of stages in the sections, keeping mass fractions of benzene in distillate and bottoms, as well as the liquid side draw flow rate constant. For every case, an optimization was performed using optimization tool built in ChemCAD, the objective function being Min Q_R, and liquid and vapour split ratios the independent variables. To determine the optimal ratio between energy cost and number of stages, another objective function, Min $N(R+1)$ was used, which approximates minimum of total annualized cost of a conventional distillation column.

As shown in Fig. 5, peaks A and B, representing A/B and B/C splits respectively, are of significantly different heights. According to Halvorsen,[20] this gives certain flexibility regarding the prefractionator operation, meaning that the prefractionator can be operated away from the preferred A/C split, along P_{AC}-P_{BC} line, without influencing the total energy requirement. To examine the influence of split ratios on energy requirement of DWC, a parametric sensitivity study was performed.

The calculation procedure can be summarized as follows:

(1) Define separation task: feed composition and state, key components' recoveries.

(2) Calculate reboiler vapour flow and distillate flow rate for every possible key components' binary split, for $N > 4N_{min}$, as using Fenske equation to estimate the N_{min}.

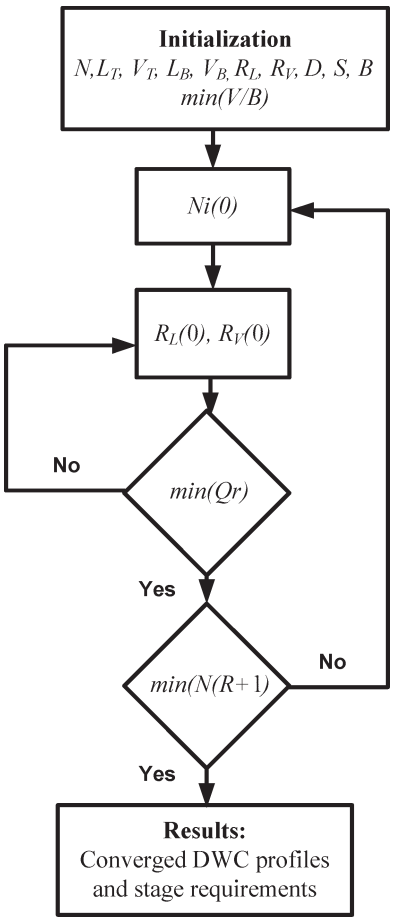
(3) Construct V_{min} diagram and calculate product flow rates, as well as internal liquid and vapour flow rates.

(4) Initialize rigorous sequential simulation using values from V_{min} diagram.

(5) Optimize vapour and liquid split ratios until Q_R is minimized, while maintaining product purities.

(6) Repeat calculation gradually reducing the number of stages in each section until $N(R+1)$ is minimized.

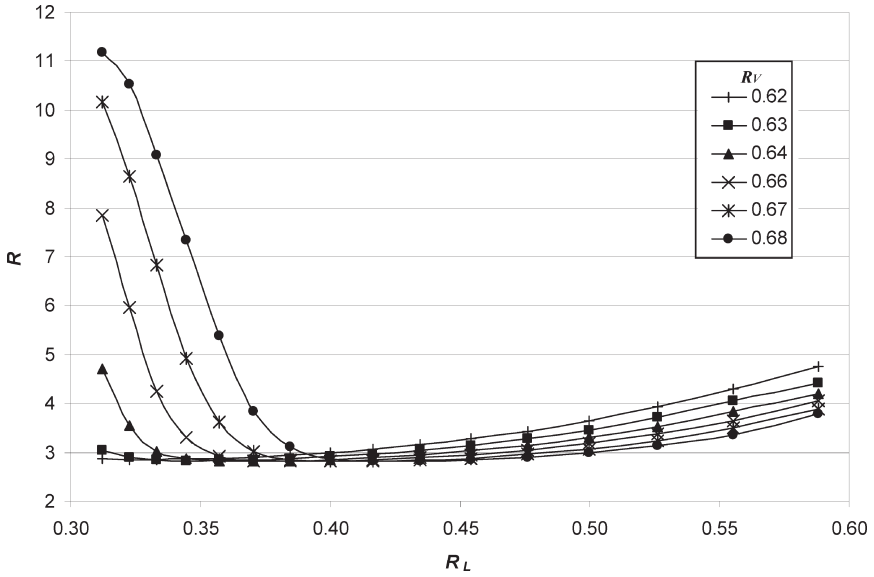
The algorithm of the detailed model, e.g. steps (4) to (6), is shown in Fig. 8.



F i g . 8 – *Algorithm of the detailed DWC model*

Results

Fig. 9 shows the results of a parametric study indicating reflux ratio values, as a measure of energy requirement at different vapour and liquid split ratios. Vapour split, denoted as R_V, represents a ratio of molar flow rate of vapour in the bottom of the prefractionator and total vapour flow rate below the wall. Similarly, liquid split denoted by R_L represents a ratio of molar flow rate of liquid in the top of the prefractionator, divided by total molar flow rate of liquid above the wall. The location of the minimum values of R is at values of R_L and R_V corresponding to the preferred split in the prefractionator (point P_{AC} in Fig. 5). When prefractionator operates in this point, a pinch zone is formed in the

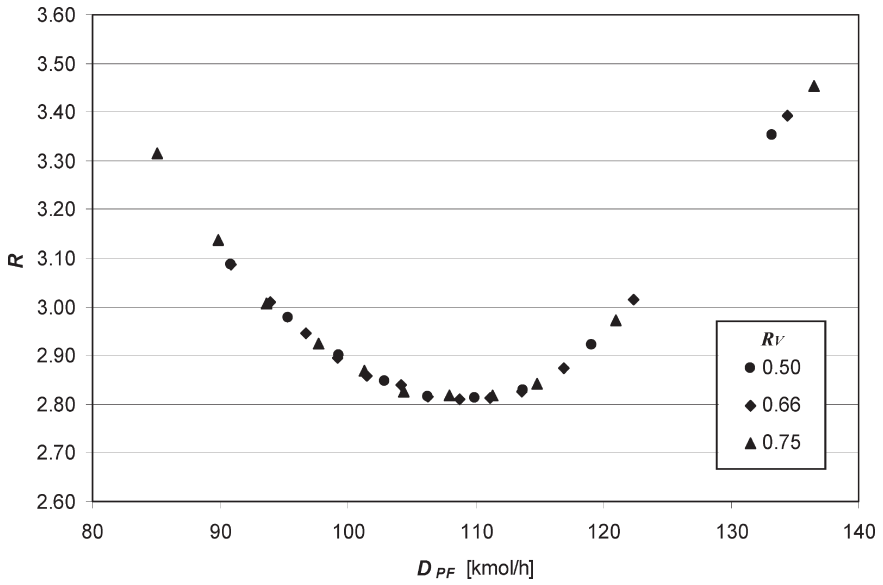


F i g . 9 – *Operating reflux as a function of the liquid split with vapour split as a parameter*

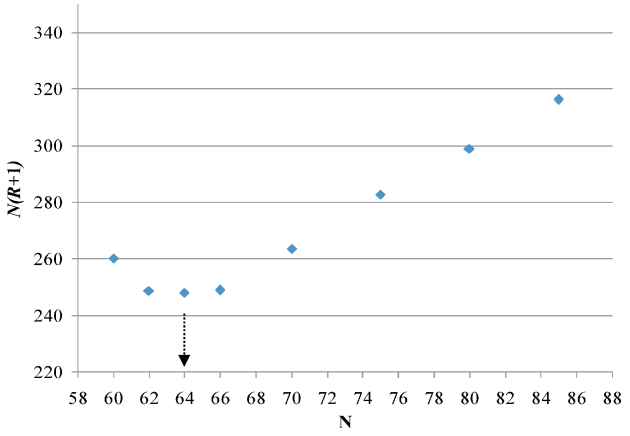
feed section of the main column, i.e. the compositions of interconnecting streams and corresponding stages of the main column are the same, leading to avoidance of additional entropy of mixing formation and minimum energy requirement.[19]

A practically flat optimality region can be observed in Fig. 9, resulting from the properties of feed mixture and specified product splits. This indicates that there is certain flexibility regarding the prefractionator operation, which can be operated away from the preferred split without increasing boil-up.

Fig. 10 shows how the minimal value of reflux ratio actually depends on the net flow in the prefractionator, defined as $D_{PF} = V_T - L_T$, for three different values of vapour split ratio. It can be seen that for each value of vapour split ratio, reflux ratio reaches approximately the same minimum value, at the same value of D_{PF}. This minimum corresponds to the net flow in the prefractionator, corresponding to the preferred split (see Fig. 5). This practically means that, for this particular case, there can be more than one combination of liquid and vapour split ratios that give almost the same minimum energy requirement. This fact gives the designer certain freedom in choosing the appropriate vapour split ratio, which is then fixed in the design stage by arranging the dividing wall position and design of the internals.



F i g . 1 0 – *Operating reflux as a function of net flow rate in the prefractionator for various vapour split values*

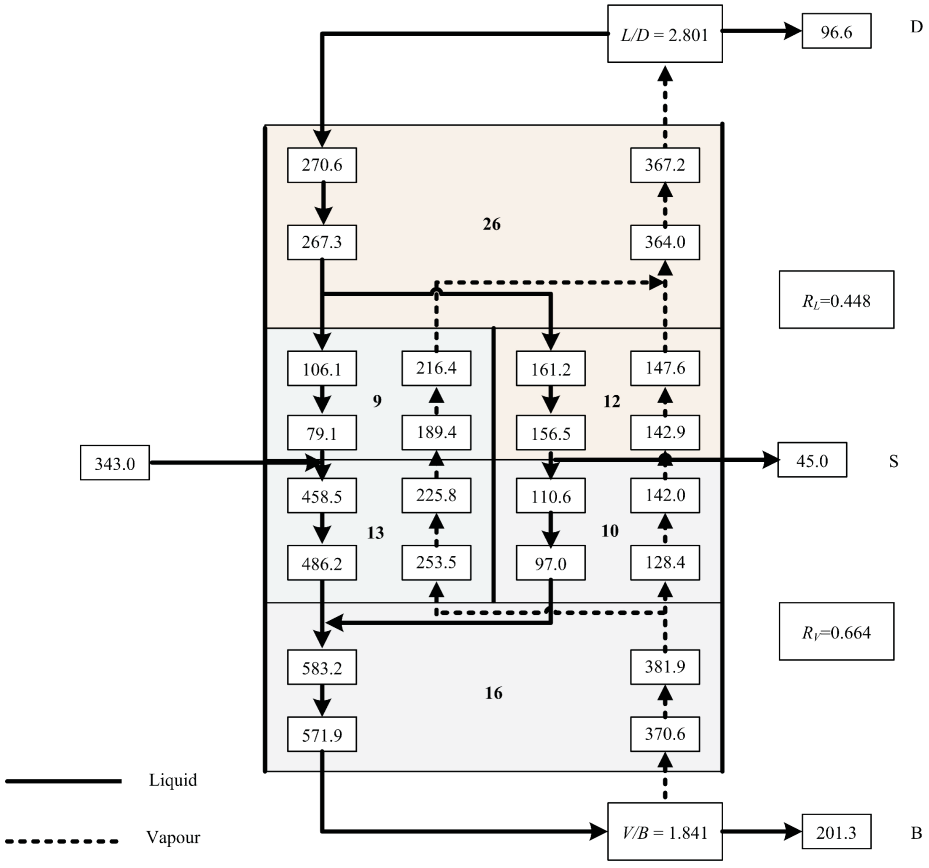


F i g e 1 1 – *Determination of the optimum number of equilibrium stages for a DWC*

Proposed objective function used to approximate minimum of total annualized cost of DWC is shown as a function of the number of stages in the main column in Fig. 11, indicating 64 equilibrium stages as a minimum value in the present case for the main column to perform accordingly. The minimum is a result of the trade-off between energy cost approximated by (R+1), and capital cost approximated by N.

Fig. 12 summarizes the mass balance of the DWC obtained by rigorous simulation, indicating the number of equilibrium stages contained in each column section. Top and bottom boxes represent top and bottom stages in a section, respectively. One should note that in the present case, the same number of stages is required at prefractionator and main column side of the DWC. This means that 86 equilibrium stages are required in a DWC, which is eight stages more than the total number of stages employed in two columns of the conventional configuration. However, with 22 stages in the prefractionator, the main column contains 64 stages, which means that the actual height of the DWC will be significantly lower than the combined height of two conventional columns containing 78 stages.

The internal molar flow rates of vapour and liquid at the top and bottom of each section are shown, as well as resulting liquid and vapour splits, above and below the partition wall, respectively. Although the prefractionator operates at similar flow rates as obtained by short-cut calculation (see Fig. 6), vapour and liquid flows in main column are practically halved. This is due to a lower external reflux and boil-up ratio requirement associated with



F i g . 1 2 – *Internal configuration with material balance of a DWC, indicating liquid and vapour flow rates at the top and bottom, and the number of stages of each section as well as resulting liquid and vapour splits*

separation of a 15-component feed into three fractions with specifications requiring much less separation effort than separation of a three-component feed into three pure products. The latter was arranged to comply with requirements of the short-cut calculation method used to obtain reliable start values for rigorous calculation initialization. As experienced during this and other similar studies, the detailed four-column model, complemented by V_{min} diagram method as a reliable means for initialisation of rigorous simulations, allows easy identification and optimization of internal configuration of a three-product DWC.

Comparison of simulation results of base-case and DWC design is shown in Table 4, indicating that a DWC would require approximately 43 % less energy, based on reboiler duties.

In the present case, such a reduction in energy requirement will translate directly not only in reduced operating costs, but also in a significantly reduced capital investment. The reason for the latter is twofold, i.e. elimination of one reboiler and one condenser as well as a reduction in overall column dimensions compared to the conventional, two columns case. A quick estimate indicated that based on total annualised cost (TAC) a DWC enables a saving with respect to conventional configuration equivalent to energy saving. However, to quantify this properly, a detailed sizing of the proposed configuration needs to be performed, which is not in the scope of this study. A detailed elaboration on the dimensioning method for three-products DWCs can be found elsewhere.[22]

Conclusions

In this paper, we have demonstrated that distillation column simulation facilities within a commercial simulator can be arranged to simulate performance of a DWC in an efficient way. A four-column model was used in conjunction with initial guesses for governing variables obtained from a simple but theoretically founded short-cut method, to generate without computational difficulties an optimized internal configuration of a DWC.

Compared to the conventional two-column-in-series configuration for obtaining benzene- and toluene-rich fractions from a 15-component feed, a DWC requires 43 % less energy for the same product specifications. This, as well as the fact that less capital and space will be needed to install a new DWC, makes this option highly interesting for implementation in aromatics processing plants as encountered in the complex petroleum refineries.

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Nomenclature

A, B, C – components (in feed, products)
B – bottom product flow rate, kmol h^{-1}
D – distillate flow rate, kmol h^{-1}
F – feed flow rate, kmol h^{-1}
L – liquid flow rate, kmol h^{-1}
n – number of products
N – number of equilibrium stages
N_f – feed stage
N_{min} – number of equilibrium stages
q – thermodynamic state of the feed
Q – heat duty, MW
R – reflux ratio/split ratio
R_L – liquid split ratio
R_V – vapour split ratio
r – component recovery
S – side product flow rate, kmol h^{-1}
V – vapour flow rate, kmol h^{-1}
w – mass fraction
x – mole fraction

Subscripts

B – bottom
D – distillate
i – component
L – liquid
PF – prefractionator
R – reboiler
T – top
V – vapour
1.1, 1.2,... – column sections

Superscripts

B – bottom
T – top

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