Feasibility of Capacity Increase of Batch Distillation by Applying a Second Column

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Increasing the processing capacity of batch distillation is important, for example, in the pharmaceutical industry, where a given amount of waste solvent must be processed in a specific time period, else the remaining amount of waste solvent must be incinerated, which results in solvent loss and considerable environmental impact. It was shown in the previous works of the authors that, depending on the feed composition, the processing capacity of batch distillation can be increased by applying a second, even smaller distillation column as pre-fractionator. Previously, the separation of three specific, highly non-ideal mixtures were studied by the authors: dichloromethane-acetone-water and acetone-water, water-acetic acid. The goal of the present paper is to study whether general conclusions can be drawn regarding the processing capacity increase. In contrast to the previous works, where the columns were operated with constant reflux ratio policy, they are operated with constant distillate composition policy in the present study. A simplified model is developed both for the single- and two-column processes, describing the relationship between the processing capacity, the product specifications (including the recovery), the feed composition and the numbers of trays by assuming constant relative volatility. The equations derived are used to study the influence of each parameter (purity specifications, feed composition, numbers of trays, specified recovery and relative volatility) on the distillation times for both processes. The conclusions obtained by these equations are validated in a case study (separating n-heptane-n-octane) by using a professional flow-sheet simulator.

1. Introduction

Batch distillation is frequently applied in the pharmaceutical industry since it can be used for treating mixtures of variable quantity and composition (Mujtaba, 2004). It is important to increase the processing capacity (Cap) of a solvent regenerator column when a given amount of waste solvent must be processed in a specific time period, else the remaining amount of waste solvent must be incinerated. Cap can be increased by optimising the operational parameters of the existing column. Maximising Cap is equivalent to the minimum time problem as defined by Mujtaba (2004) when a given amount of product must be recovered with a specified purity. However, when the operation of the column is optimal, but further capacity increase is required, a new column must be installed. A cheaper solution, only studied in the literature from the capacity increase point of view by Nemeth et al. (2020b), is to apply an additional, even smaller batch column available in the plant as pre-fractionator before the existing column. In this case, the main cut or still residue of the first column can be processed in the second one. During the operation of the second column, new batch(es) can be processed in the first one, and the two columns can operate simultaneously. If the operation of the first column is fast enough, several batches can be processed, and the main cuts or residues can be united into a single charge of the second column. It is also worth uniting these cuts if their amount is low and the still volume of the second column is large.

Only a few authors investigated batch distillation processes performed in two batch distillation columns, and the question of Cap increase was just briefly mentioned in these works. Adi and Chang (2010) studied the scheduling of a two-column batch heteroazeotropic distillation system by optimisation. The authors did not perform rigorous calculations but used simple material and energy balance equations contrary to the present work, where the columns are also modelled rigorously by using a professional flow-sheet simulator. The

objective of the scheduling optimisation in the work of Adi and Chang (2010) was to maximise the overall profit of a production. The authors concluded that equipment sharing is not beneficial for performing the distillation tasks because it can significantly decrease the overall profit.

Lara-Montaño et al. (2019) studied a two-column process by simulation and experiment for the purification of bioethanol, where a continuous pre-fractionation column was applied before final purification by batch extractive distillation. Different columns were used in each distillation step, but the processing capacity was not studied.

Zhao et al. (2021) used a double-column batch distillation process where the columns are interconnected for the separation of n-hexane/ethanol/butanone. Three different column configurations were studied by minimising the total annual cost and by calculating the global warming potential for each case. Only the operating pressures of the columns were optimisation variables contrary to the present work, where the influence of several operational parameters are studied on the processing capacity for the two-column process.

In the previous works of the authors, the two-column batch distillation process was already investigated. The first study was based on an industrial example (Nemeth et al., 2020a), where the separation task was the regeneration of acetone from an aqueous mixture also containing dichloromethane. The separation was carried out in two columns: the first, pre-fractionator column was the smaller one providing 5 theoretical plates (including the reboiler and the total condenser), while the other column was a larger one providing 22 theoretical plates. In the first column pre-fractionation was performed, then the distillate was further processed in the second one, where acetone was obtained in the specified purity as distillate. Using the smaller, pre-fractionator column resulted in higher processing capacity compared to the case when the separation was performed only in the larger column. In this previous work, the columns were operated with constant reflux ratio policy and only the purity of the final product (distillate of the 2nd Column) was specified.

The configuration of the two-column process and its relationship to Cap was further studied by Nemeth et al. (2020b). Two mixtures were studied: acetone-water (Mixture 1) and water-acetic acid (Mixture 2). For Mixture 1, the distillate of Column 1 was further processed in Column 2, while for Mixture 2, the residue of Column 1 was further processed. In both cases, the columns were operated with constant reflux ratio policy and only one purity was specified: that of the distillate (Mixture 1) or the residue (Mixture 2) of Column 2. The recovery for the two-column process was specified to be equal to that of the single-column process. For both mixtures there were such operational parameters and charge compositions when Cap of the two-column process was higher than that of the single-column process. When the distillate of Column 1 was further processed, the maximum Cao was reached when the operating time of the two columns (including the dead times) were equal. However, when the residue of Column 1 was processed further, the maximum Cap was not reached at the time-equality of the two columns, but far from this point.

In the present work, the two-column process is studied further by setting up a simplified model for calculating the distillation time of both the single- and two-column processes. In this case, the columns are operated with constant distillate composition policy (contrary to the previous works mentioned above), but still, only one purity is specified (for the distillate of Column 2), and the recovery for the whole process is also specified. For calculating the distillation time, the reflux ratio (R) must be calculated, which is a function of time or equivalently a function of the residue composition. For calculating an average reflux ratio, the integral average of the reflux ratio function was taken similarly to the integral average of N_{min} (Betlem et al., 1998). The novelty of this work is that the influence of several operational parameters on the processing capacity is studied both by the simplified model developed and by simulations. Moreover, the results calculated by the simplified model are compared to those obtained from simulations using the professional flow-sheet simulator ChemCAD 7 (Chemstations, 2016). An ideal binary mixture is studied in the simulation case study: n-heptane-n-octane.

2. Vapor-liquid equilibrium (VLE)

The relative volatility (α) of binary mixtures are studied to select one with almost constant α. The mixtures are: n-hexane-n-heptane, whose α changes +/- 6 %; n-heptane-n-octane, whose α changes +/- 5 %; and benzene-toluene, whose α changes +/- 6 %. Based on this, the n-heptane-n-octane mixture with an average α=2.12 (1) is chosen for the simulations. For describing the VLE, the SRK model is used in the rigorous simulation.

3. Separation process and its mathematical description

The possible separation sequences are described for separating zeotropic binary mixtures using single- and two-column systems. The simplified models for calculating the distillation time of the processes are presented.

3.1 Separation process

The separation of the n-heptane-n-octane mixture is performed in a single-column (Figure 1a) and in a two-column process. There are two possible production sequences of the two-column process depending on which product of the first column is processed further:

1. further processing of the distillate of Column 1 in Column 2 (Figure 1b)
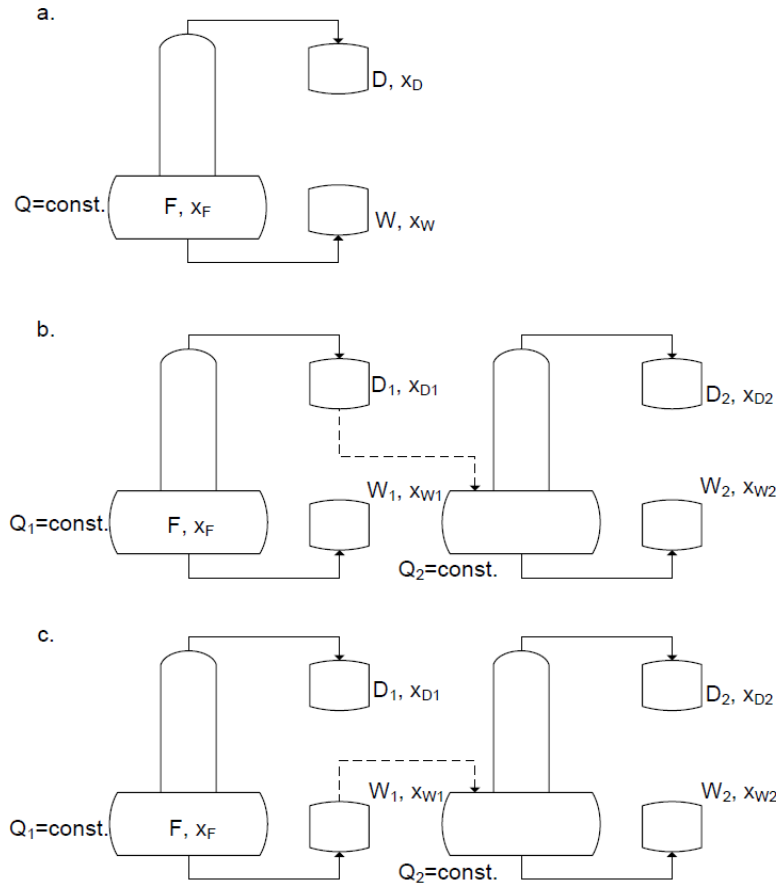2. further processing of the residue of Column 1 in Column 2 (Figure 1c)

a.

b.

c.

Figure 1: Batch distillation using one column (a) and two columns with further processing b. distillate; c. residue

There are further possible cases for the two-column process regarding the number of purity specifications:

1. The purity of both products are specified (For case b. in Figure 1: $x_{D,spec}=x_{D2}$ and $x_{W,spec}=x_{W2}=x_{W1}$, for case c: $x_{D,spec}=x_{D2}=x_{D1}$ and $x_{W,spec}=x_{W2}$)
2. The purity of the distillate is specified (For case b. in Figure 1: $x_{D,spec}=x_{D2}$, for case c: $x_{D,spec}=x_{D2}=x_{D1}$)
3. The purity of residue is specified (For case b. in Figure 1: $x_{W,spec}=x_{W2}=x_{W1}$, for case c: $x_{W,spec}=x_{W2}$)

The recovery of n-heptane (η_{spec}) for the two-column process is also specified in all cases investigated. The same amount of charge (F) is processed with the single- and two-column processes, but only F/2 can be fed processed in Column 1 (e.g. limited volume of the still). Hence, Column 1 must process two charges for the two-column process. This makes it possible to compare its processing capacity to that of the single-column process under the same recovery. The following mathematical description only refers to the single-column process (Figure 1a) and to the two-column process when the distillate is processed further in Column 2 (Figure 1b) and only the purity of the distillate is specified ($x_{D,spec}=x_{D2}$). For the two-column process, x_{D1} and the recovery of n-heptane in Column 1 (η_1, which is calculated from the relative loss in the prefactionator: $\eta_1=1-W_1x_{W1}/(Fx_F)$), which are additional degrees of freedom compared to the single-column process ($x_{D,spec}$ and η_{spec}), are determined to obtain minimum distillation time. These are also the cases for which simulations are performed.

In the calculations performed, the following assumptions are made: 1. Constant α; 2. Each column has constant heat duty ($\dot{Q}$); 3. In the simplified model, the pressure drop and hold-up in the total condenser and in the columns

are neglected. Even without the above assumptions, Eq(1) is always valid for any distillation process, from which $\dot{D}$ (molar flow rate of distillate) can be written as Eq(2).

$$R = \frac{\dot{V} - \dot{D}}{\dot{D}} \tag{1}$$

$$\dot{D} = \frac{\dot{V}}{R + 1} = \frac{\dot{Q}}{\lambda \cdot (R + 1)} \tag{2}$$

where $\dot{V}$ is the molar flow rate of vapor in the column, and λ is the latent heat of evaporation.

3.2 Processing capacity of the single-column process

The processing capacity can be described by Eq(3) for the single-column process, where the distillation time (t_S) is a function of the molar flow rate of the distillate (Eq(4)) assuming constant R_S (subscript S refers to the single-column process). For the single-column process, it is assumed that the larger (second) column is used ($\dot{Q} = \dot{Q}_2$, $N_S = N_2$, $x_{D,spec} = x_D = x_{D2}$ and $\eta_{spec} = \eta = \eta_2$). The subscript 2 refers to the second column.

$$C_S = \frac{F}{t_S} \tag{3}$$

$$t_S = \frac{D}{\dot{D}} = \frac{D \cdot \lambda \cdot (R_S + 1)}{\dot{Q}_2} \tag{4}$$

From Eq(3) and Eq(4), the processing capacity can be written as Eq(5).

$$C_S = \frac{F \cdot \dot{Q}_2}{D \cdot \lambda \cdot (R_S + 1)} \tag{5}$$

The recovery (η) of n-heptane specified can be written as Eq(6):

$$\eta_{spec} = \frac{D \cdot x_{D,spec}}{F \cdot x_F} \tag{6}$$

where x_F is the mole fraction of n-heptane in the charge. By combining Eq(5) and Eq(6), Eq(7) is obtained:

$$C_S = \frac{x_{D,spec} \cdot \dot{Q}_2}{\eta_{spec} \cdot x_F \cdot \lambda \cdot (R_S + 1)} \tag{7}$$

For calculating the capacity, R_S must be determined, e.g. by taking the inverse of the Gilliland equations (Eq(8)).

$$
\begin{aligned}
0.6 < Y < 1.05 \qquad & \lg X = \frac{-0.3397 - \lg Y}{0.0906} \\[2mm]
0.49 < Y < 0.6 \qquad & X = \frac{1.75 \pm \sqrt{16.66 \cdot Y - 8.155}}{8.332} \\[2mm]
0.04 < Y < 0.49 \qquad & X = \frac{0.85 \pm \sqrt{Y + 0.1225}}{0.5}
\end{aligned}
\tag{8}
$$

where $X = \frac{R - R_{min}}{R + 1}$ and $Y = \frac{N - N_{min}}{N + 1}$.

R_{min} and N_{min} can be calculated from the Underwood (Eq(9)) and Fenske (Eq(10)) equations, which can also be used for batch distillation (Mujtaba, 2004) because the batch process can be represented by several continuous distillation operations of short duration. In batch processes x_D and x_W are functions of time. As x_D is kept constant ($x_D = x_{D,spec}$) for practical reasons, R_S becomes a function of time. R_S can also be written as a function of x_W.

$$R_{min}(x_W) = \frac{1}{\alpha - 1} \cdot \left[\frac{x_{D,spec}}{x_W} - \frac{\alpha \cdot \left(1 - x_{D,spec}\right)}{1 - x_W} \right] \tag{9}$$

$$N_{min}(x_W) = \frac{\lg \dfrac{x_{D,spec} \cdot (1 - x_W)}{x_W \cdot (1 - x_{D,spec})}}{\lg \alpha} \tag{10}$$

Substituting X and Y to Eq(8), R_S is obtained as a function of $x_{D,spec}$, x_W, α and N_S.

$$R_S = \frac{X(x_{D,spec}, x_W, \alpha, N_S) + R_{min}(x_{D,spec}, x_W, \alpha)}{1 - X(x_{D,spec}, x_W, \alpha, N_S)} \tag{11}$$

$X(x_{D,spec}, x_W, \alpha, N_S)$ is a piecewise function as given in Eq(8). R_S is also a piecewise function consisting of three equations. To calculate the distillation time using Eq(4), the definite integral of R_S (Eq(12)) must be taken in the range of x_{Wfinal} (calculated by Eq(13)) and x_F.

$$t_S = \frac{D \cdot \lambda \cdot \left(\int_{x_{Wfinal}(x_{D,spec}, x_F, \eta_{spec})}^{x_F} R_S(x_{D,spec}, x_W, \alpha, N_S) dx_W + 1 \right)}{\dot{Q}_2 \left(x_F - x_{Wfinal}(x_{D,spec}, x_F, \eta_{spec}) \right)} \tag{12}$$

$$x_{Wfinal} = \frac{x_{D,spec} \cdot x_F \cdot (1 - \eta_{spec})}{x_{D,spec} - \eta_{spec} \cdot x_F} \tag{13}$$

When plotting R_S as the function of x_W (Figure 2), three equations for R_S in the different intervals of x_W can be identified (marked as R_a, R_b and R_c in Figure 2) based on the Y values (Eq(8)): R_a is valid at high, R_b at medium and R_c at low Y values.
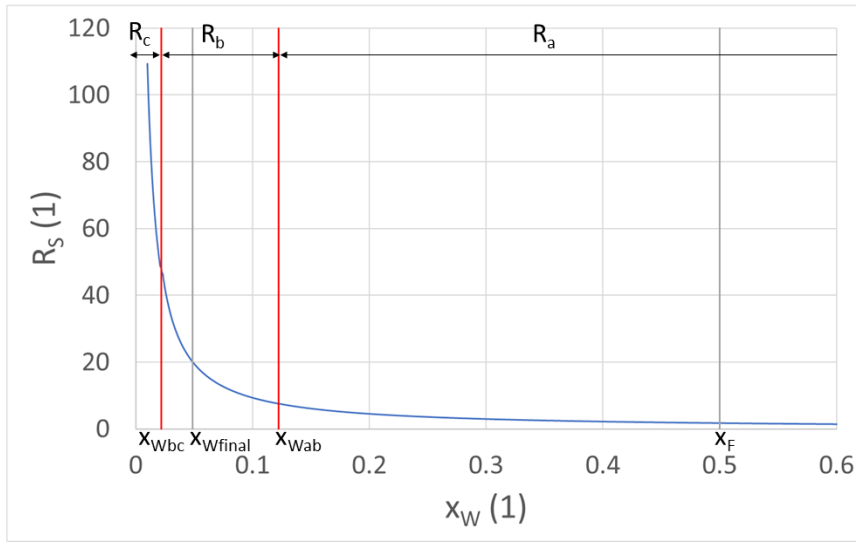

Figure 2: R_S as the function of x_W ($x_{D,spec}$=0.98 (1); α=2.12 (1); N_S=21 (1); η_{spec}=95 %)

In Figure 2, x_{Wab} means the still composition when the validity of R_a and R_b changes in the expression of R_S, while x_{Wbc} means the still composition when the validity of R_b and R_c changes. x_{Wab} and x_{Wbc} are determined by solving the Gilliland equations with the relevant boundaries (Eq(8)) for x_W at given parameters ($x_{D,spec}$, α, N_S). The distillation time (t_S) is calculated as the sum of maximum three integrals of R_S (Eq(14)). The number and the boundaries of the integrals in the expression of t_S depend on the values of x_{Wfinal} and x_F. In Eq(14), the boundaries of each integral are defined so that if x_F or x_{Wfinal} is out of the validity range of R_a, R_b or R_c, its definite integral value becomes zero (for example, if $x_F > x_{Wab}$, but $x_{Wab} > x_{Wfinal}$, the value of the first integral is zero).

$$\int_{x_{Wfinal}}^{x_F} R_S \, dx_W = \int_{\max(x_{Wab}, x_{Wfinal})}^{\max(x_F, x_{Wab})} R_a dx_W + \int_{\max(x_{Wbc}, \min(x_{Wfinal}, x_{Wab}))}^{\min(x_F, x_{Wab})} R_b dx_W + \int_{\min(x_{Wbc}, x_{Wfinal})}^{\min(x_F, x_{Wbc})} R_c dx_W \tag{14}$$

In the example shown in Figure 3a, x_{Wfinal} is higher than x_{Wbc}, but lower than x_{Wab}. Since x_F is higher than x_{Wab}, according to Eq(14) $\int_{x_{Wfinal}}^{x_F} R_S \, dx_W = \int_{x_{Wfinal}}^{x_{Wab}} R_b dx_W + \int_{x_{Wab}}^{x_F} R_a dx_W$ must be used to calculate the distillation time by Eq(12). In the example shown in Figure 3b, following the same logic: x_{Wfinal} is lower than x_{Wbc}. x_F is higher than x_{Wbc}, but lower than x_{Wab}. Consequently, $\int_{x_{Wfinal}}^{x_F} R_S \, dx_W = \int_{x_{Wbc}}^{x_F} R_b dx_W + \int_{x_{Wfinal}}^{x_{Wbc}} R_c dx_W +$ according to Eq(14).

Using the integral of R_S values obtained from Eq(14), the distillation time can be calculated for any parameter combinations using Eq(12). Since minimising the distillation time is equivalent to the maximisation of the capacity (Eq(3)), only the distillation time is discussed in the followings.

3.3 Processing capacity of the two-column process

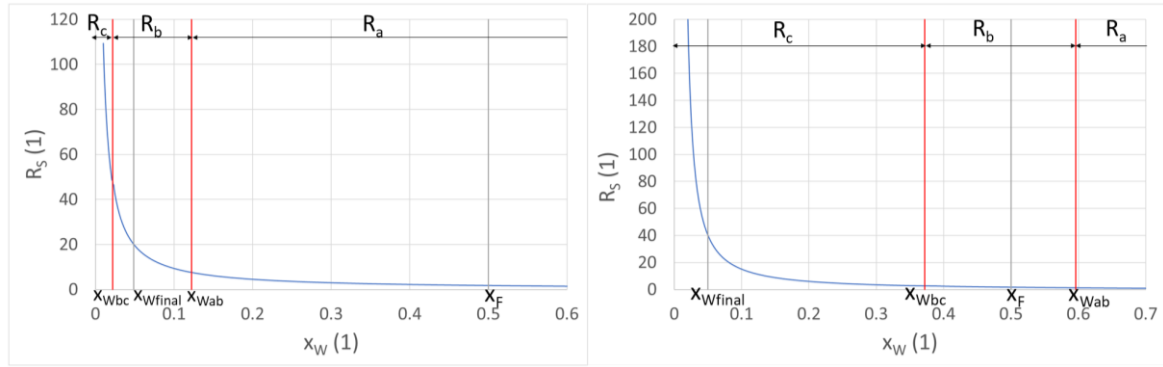For the two-column process, the distillation time of each column i=1,2 can be written as Eq(15).

Figure 3: Examples for choosing the right equation for calculating t_S. a. ($x_{D,spec}=0.98$ (1); $\alpha=2.12$ (1); $N_S=21$ (1); $\eta_{spec}=95$ %), b. ($x_{D,spec}=0.95$ (1); $\alpha=2.12$ (1); $N_S=10$ (1); $\eta_{spec}=95$ %)

$$t_i = \frac{D_i \cdot \lambda \cdot (R_i + 1)}{\dot{Q}_i} \tag{15}$$

The following equations are valid when the distillate of Column 1 is further processed in Column 2 (Figure 1-b). Both R_1 and R_2 can be calculated as R_S, but $D = D_1, x_{D,spec} = x_{D1}, \dot{Q} = \dot{Q}_1, \eta_{spec} = \eta_1$ and $x_W = x_{W1}$ for R_1, and $D = D_2$, $\dot{Q} = \dot{Q}_2$ and $x_W = x_{W2}$ for R_2. For Column 2, η_{spec} is defined as in Eq(6), but with the substitution $D = D_2$. This means that for the two-column process, η_{spec} refers to the recovery for the whole process.
For Column 1, $x_{W1final}$ can be written as Eq(16).

$$x_{W1final} = \frac{x_F \cdot x_{D1} \cdot (1 - \eta_1)}{x_{D1} - \eta_1 \cdot x_F} \tag{16}$$

While $x_{W2final}$ can be written as Eq(17) for Column 2.

$$x_{W2final} = \frac{x_{D1} \cdot x_{D,spec} \cdot (\eta_1 - \eta_{spec})}{\eta_1 \cdot x_{D,spec} - \eta_{spec} \cdot x_{D1}} \tag{17}$$

The same equations can be obtained with the corresponding substitutions for both columns as for the single-column case (Eq(14)).

4. Simulation model and optimization

A simulation model is prepared in ChemCAD 7 for the separation of n-heptane-n-octane mixture. The same model was used as in Nemeth et al. (2020b). Each column is operated with constant heat duty: $\dot{Q}_1 = 360$ MJ/h in Column 1 and $\dot{Q}_2 = 720$ MJ/h in Column 2 and with total condensers. Column and condenser hold-ups are considered in the simulations: the column hold-up is specified as 0.3 dm^3/plate, while the condenser hold-up is 5 dm^3 for both columns. The pressure drop of Column 1 is 0.3 kPa, while it is 0.4 kPa for Column 2. The maximum capacity of the reboilers are different: 5 m^3 for Column 1 and 10 m^3 for Column 2. The charge is always F = 65.1 kmol for the single-column process (using the larger still pot), while it is 32.55 kmol fed two times to the smaller reboiler of Column 1 for the two-column process. The above parameters are kept constants for all calculations, while the other parameters, such as the number of trays (N) and the operational parameters, are varied to study their influence on the distillation times. For the single-column process, Column 2 is used.
For both processes, the columns are operated with constant distillate composition policy. The stopping criterion for the columns is the amount of distillate calculated from the η_{spec} using the $x_{D,spec}$ by Eq(6). The influence of the following parameters is studied on the distillation time by performing sensitivity studies: number of theoretical plates ($N_S=N_2$ and N_1), distillate purity specified ($x_{D,spec}$), recovery (η_{spec}) and charge composition (x_F). For the two-column process, the optimal x_{D1} and η_1 values are determined to get the minimum distillation time for each parameter combination. The optimisation problem both for the simulation and for the simplified model is:

$$\min\left\{OF = \max\left(\left(t_1 + \Delta t_{1,dead}\right)\cdot 2;\ t_2 + \Delta t_{2,dead}\right)\right\}$$

subject to: $\eta_1 > \eta_{spec}$

$\qquad\qquad x_{D1} > x_{D1,crit}$

(18)

where $R_1\left(x_{D1,crit}, x_F, \alpha, N_1\right) = 0$

In Eq(18), $\Delta t_{1,dead}$ = 3.5 h is the dead time for Column 1, while $\Delta t_{2,dead}$ = 1.6 h is that of Column 2. The constraints are: 1. The recovery for Column 1 must be higher than that of the whole process. 2. x_{D1} must be higher than a critical value corresponding to zero reflux ratio at the start of the process ($x_W = x_F$). With the 2nd constraint, negative reflux ratios can be avoided. The optimisation is performed by sensitivity studies with small step sizes. For each distillation column, two distillation steps are specified (apart from the heating-up) for technical reasons to ease the convergence of the simulation. Normally, there shall be only one distillation step after the heating up of the column (with infinite reflux ratio), but due to the constant distillate composition policy, the reflux ratio required at the beginning of the distillation step is too low. Changing from infinite reflux ratio to a small one causes convergence problems. To circumvent this problem, an additional distillation step is defined with a small, constant reflux ratio (R=1) for a short period of time.

5. Results

The results obtained with the simplified model (Chapter 5.1) and the simulation (Chapter 5.2) are compared.

5.1 Results from the equations

First, the influence of the parameters ($x_{D,spec}$, N_S, x_F, η_{spec} and α) on the distillation time is studied for the single-column process. Only one parameter of the base case is varied at a time. For the two-column process, the influence of only one parameter is studied at a time too, but the two additional degrees of freedom are considered as optimisation variables, and their values are calculated to get an optimal (minimal) distillation time for the process. The parameters varied are listed in Table 1 with their lower and upper bounds.

Table 1: Lower and upper bounds of variables.

Process	x_F (1)	$x_{D,spec}$ (1)	N_S (1)	N_1 (1)	η_{spec} (%)	α (1)
Single-column	0.2 – 0.8	0.85 – 0.98	10 – 30	-	86 – 98	1.3 – 4.0
Two-column	0.3 – 0.8	0.88 – 0.98	-	4 – 15	86 – 97	1.8 – 3.5
Base case	0.5	0.98	21	5	95	2.12

The variables x_{D1} and η_1 are optimisation variables in the two-column process with the constraints given in Eq(18). Hence no bounds are given for them in Table 1.

Figure 4 shows the results calculated by Eq(12). For all points belonging to the two-column process, the optimal x_{D1} and η_1 are calculated, which results in the time equality of the distillation times of both columns. This corresponds to the results presented by Nemeth et al. (2020b).

With increasing $x_{D,spec}$, the distillation time of the single-column process increases (Figure 4a), because R_{min} and N_{min} increase, which also raises R_S. For the two-column process, the optimal distillation time of the process does not change significantly on the increase of $x_{D,spec}$, due to the optimisation of x_{D1} and η_1. For decreasing t_1, x_{D1} must be decreased (it is decreased to its lower bound in all cases, which means it is the smallest value to avoid negative reflux ratios at the beginning of the distillation). Besides, the decrease of η_1 also decreases t_1 to a smaller extent, but it highly increases t_2 (to the value, when the distillation times of both columns are equal). As the optimal x_{D1} is the same (the lower bound) for all calculated points, but η_1 can be still slightly decreased to decrease the distillation time of the two-column process; the influence of the decreasing η_1 can be seen in Figure 4a with the almost unchanged (slightly decreasing) distillation time.

With increasing x_F, the distillation time of the single-column process considerably increases (Figure 4b). The increase in x_F slightly decreases the average reflux ratio through the increase in x_{Wfinal} but it also increases the amount of distillate to be collected (for a given η_{spec}), which leads to a significant increase in the distillation time. For the two-column process, the distillation time does not change significantly on the increase of x_F. The reason for this is that the difference between η_{spec} and η_1 is very small. As a result, with the increase of x_F, D_1 and D_2 increase proportionally. Only the influence of the slightly changing η_1 is visible in the slightly changing distillation time (the situation is similar to the case, when $x_{D,spec}$ is increased).

The influence of N_1 on the distillation time is shown in Figure 4c. As Column 1 does not operate in the single-column process, t_S is shown as a horizontal line (for N_S=21 (1)) as the function of N_1. By increasing N_1 for the two-column process, the distillation time decreases because lower R_1 is enough for the separation in Column 1. In Figure 4d, the distillation time is shown as the function of N_S (N_S=N_2). Since N_2 was not varied in the two-column process, its distillation time is shown as a horizontal line. t_S is decreasing on the increase of N_S because lower R_S is required for the separation. When N_S=10 (1), the reflux ratio is negative at the beginning of the distillation with $x_{D,spec}$=0.98 (1).
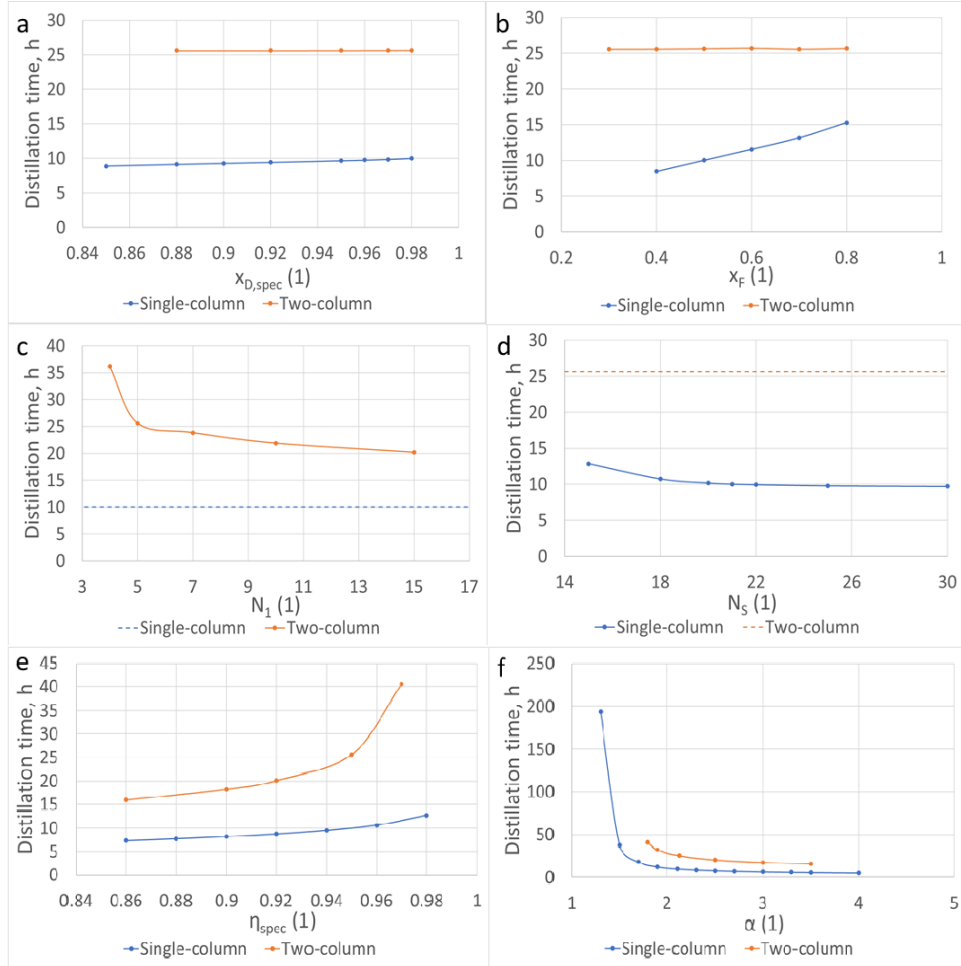
Figure 4: The influence of the parameters (a. x_D, b. x_F, c. N_1, d. N, e. η_{spec}, f. α). on the distillation time (simplified model).

On the increase of η_{spec}, the distillation time increases for both separation processes (Figure 4e). For the single-column process t_S increases due to the increase in D to reach η_{spec}. For the two-column process, D_2 increases similarly to the single-column process. As η_{spec} becomes higher, η_1 must also be increased because of the constraints given in Eq(18), which also raises D_1, and as a result, t_1 increases. It must be noted that for the two-column process, if $\eta_{spec} \geq 0.98$ (1) R_2 (with constant $x_{D,spec}$) is negative at the beginning of the separation.

Figure 4f shows the influence of α (which does not vary for this mixture if the pressure is constant). For the single-column process, t_S decreases on the increase of α because R_S decreases significantly through the decreasing R_{min} and N_{min} required for the separation. The influence of α on the distillation time is the same for the two-column process. The only difference is that the separation cannot be performed below α=1.8 (1) in Column 1 with N_1=5 (1) because of the negative reflux ratios.

5.2 Results from the simulation

Simulations are performed with the same considerations as the calculations by the equations, namely:
1. Only one parameter is varied at a time

2. For the two-column process, the minimal distillation time is determined by optimising x_{D1} and η_1. The simulation results of the single-column process show good agreement with the results calculated by Eq(12). Two examples are given in Figure 5 for the influence of η_{spec} (Figure 5a) and N_S (Figure 5b) on t_S.
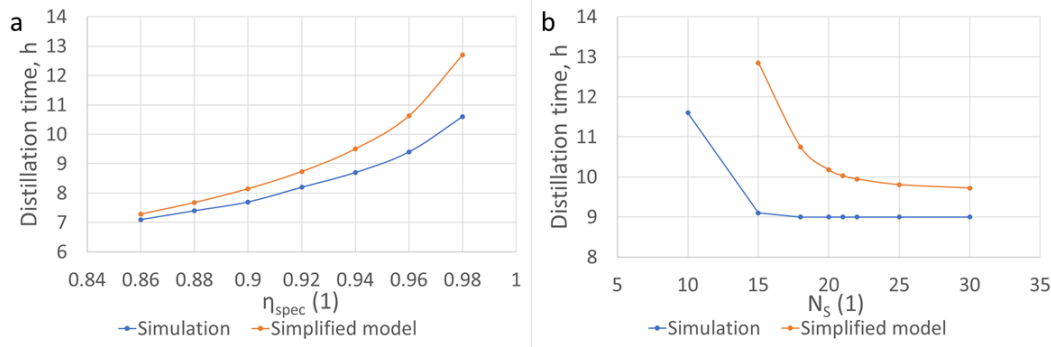

Figure 5: Calculated distillation times of the single-column process by simulation and by the simplified model for the cases when varying a. η_{spec}; and b. N_S

The t_S values calculated by the simulation are always lower because of multiple effects. These effects, which only slightly decrease t_S by themselves, but whose resultant is significant are the following ones: 1. The average reflux ratio in the simulation is lower than that calculated by the simplified model (with the assumption of constant α). 2. An additional distillation step with constant reflux ratio is performed in the simulation (due to the previously mentioned convergence reason). The above points also apply to the two-column process.

For the two-column process, the results generally show good agreement with those calculated by Eq(12), but the time equality is not reached at any of the points calculated due to the high sensitivity of the distillation time of Column 2 (t_2) to varying the specified recovery in Column 1 (η_1): even a very small decrease of η_1 resulted in high increase in t_2. With a smaller step size, the time-equality point could be reached, but decreasing the step size also caused convergence error. There are two cases when the results of the simulations differ considerably from those calculated by the simplified model (Figure 6).
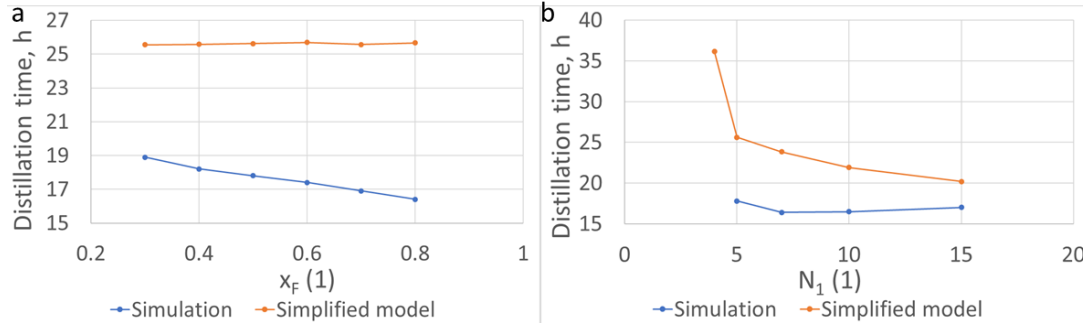

Figure 6: Calculated distillation times of the two-column process by simulation and by the simplified model for varying a. x_F; and b. N_1.

With increasing x_F, the distillation time of the two-column process decreases contrary to the results obtained from the simplified model, where the distillation time is approximately constant (Figure 6a). This might be caused by the effect that with increasing x_F, a lower average reflux ratio is required for the separation in Column 1. The distillation step with constant reflux ratio added to avoid convergence problems has a higher impact on decreasing the distillation time in the simulation.

On the increase of N_1, the distillation time of the two-column process has a minimum by the simulations (Figure 6b). With increasing N_1, R_1 decreases, and t_1 also decreases and the total hold-up of the column increases. The increasing amount of hold-up results in increasing t_1. This effect becomes the dominating one at a critical N_1 (at the minimum of the distillation time). It must be noted that in the simulation, when $N_1 \leq 4$ (1), η_{spec} (0.95 (1)) cannot be reached with $x_{D,spec} = 0.98$ (1). To reach η_{spec}, η_1 must be increased, which means a higher amount of D_1 to be collected. This D_1 cannot be reached with higher values of x_{D1} because at the end of the distillation, R_1 is too high (it tends to infinity). Hence x_{D1} must be decreased, but even at its lowest value (when the negative reflux ratios are avoided at the beginning of the distillation), η_{spec} still cannot be reached in Column 2.

6. Conclusions

A two-column batch distillation process, where the separation is performed in a smaller pre-fractionator and in a subsequent larger column, was further studied as the continuation of the previous works of the authors. In contrast to those previous works where constant reflux ratio policy was applied, both columns were operated with constant distillate composition policy in the present work.

A simplified model (assumptions: 1. constant relative volatility and 2. negligible hold-up and pressure drop) was developed to quickly and easily calculate the distillation time of both the single- and two-column processes. The simplified model is valid for binary mixtures, and for the two-column process, it is valid for the case when the distillate of Column 1 is processed further in Column 2. The results of the simplified model show that the maximum processing capacity (or equivalently the minimum of distillation time) of the two-column process is reached at the time-equality point of the two columns. However, the distillation times of the two columns calculated by simulations were not equal at the maximal processing capacity due to the high sensitivity of the distillation time of Column 2 (t_2) to varying the recovery specified in Column 1 (η_1). Finer tuning of η_1 could also result in the time equality of the two columns in the simulations, but reaching this point is difficult due to convergence difficulties. The results of the simplified model were compared to those of the simulations (for the separation of n-heptane-n-octane). The deviations are relatively high with an average of 38 % for the two-column process and an average of 14 % for the single-column process. The higher deviations between the two models are also caused by the high sensitivity of t_2 to varying η_1. However, the results change similarly for varying the parameters. For the separation of n-heptane-n-octane, the single-column process proved to be by 55-60 % better (depending on the parameter studied) considering distillation times by both the simulations and the simplified model. It must be noted that the constant distillate composition policy is a very strict constraint in Column 1 (providing a low number of theoretical stages) for the pre-fractionation. This is the reason why the distillation times are of the two-column process are higher.

Possible future works could involve searching for parameter combinations, where the two-column process results in higher processing capacity than the single-column one (with the constant distillate composition operating policy) and developing a simplified model for calculating the distillation time when the residue of Column 1 is processed further.

Acknowledgements

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