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EXPLORATION OF THE DESIGN OF DISTILLATION COLUMNS — BEYOND REFLUX AND TRAY COUNT

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INTRODUCTION

The starting point of this work can be found in an interesting problem proposed by Seader et al.,[1] where some features of multicomponent distillation, from the behavior of the condenser to the thermal condition of the feed stream, are posed. The complete answer to the problem requires the use of different tools available in chemical processes simulators, as ChemCAD from Chemstations, Inc.[2] or Aspen HYSYS® and Aspen Plus™ from Aspentech.[3] Students not only have to be able to recognize and to know what unit to choose and when and how to use it, but also to select the best way to extract and analyze the results. The process knowledge acquired by solving the problem permits us to develop a case study in order to perform a comparison among the different ways to select the optimal feed stage, as well as to study the effect of the feed temperature on the column behavior.

The above-mentioned exercise corresponds to problem number 10.30 of the textbook by Seader et al.[1] and focuses on the design of a depropanizer with the problem statement as follows:

A depropanizer distillation column is designed to operate at a feed stage pressure of 315 psia (21.7 bar) for separating a feed into distillate and bottoms with the flowrates shown in Table 1. The feed is 66 mol% vapor at tower pressure. Steam at 315 psia (21.7 bar) and cooling water at 65 °F (291 K) are available for the reboiler and condenser, respectively. Assume a 2-psi (0.138 bar) column pressure drop.

For this separation:

a. *Should a total condenser be used for this column?*

b. *What are the feed temperature, the equilibrium ratios (K-values), and relative volatilities with reference to n-butane (C4)?* (Author's note: It is worth mentioning that in the original statement, the requested volatilities referred to propane (C3), but in this case it has been changed because in this type of problem, the relative volatilities are usually referred to respect to the heavy key component (C4)).

c. *If the reflux ratio is 1.3 times the minimum reflux, what is the actual reflux ratio, R? How many theoretical plates, N, are needed in the rectifying and stripping sections?*

d. *Compute the separation of species. How will the separation differ if a reflux ratio of 1.5, 15 theoretical plates, and feed in the ninth plate are chosen?*

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e. *Compute the temperature and concentration in each stage. What is the effect of feed plate location? How will the results differ if a reflux ratio of 1.5 and 15 theoretical plates is used?*

TABLE 1
Proposed separation corresponding to problem 10.30 from Seader et al.[1] with the original units changed from lbmol/h to kmol/h.

	kmol/h		
Component	**Feed**	**Distillate**	**Bottoms**
Methane (C1)	26	26.0	—
Ethane (C2)	9	9.0	—
Propane (C3)	25	24.6	0.4
n-Butane (C4)	17	0.3	16.7
n-Pentane (C5)	11	—	11.0
n-Hexane (C6)	12	—	12.0
Total	100	59.9	40.1

In this work the original flowrate units have been changed from lbmol/h to kmol/h in order to obtain larger column diameters, in the range of the available correlations for calculating the costs of columns.[2,3] In the following sections the answer to these five points, as well as the analysis of the best feed stage location and the effect of the thermal feed phase, are provided and analyzed. The detailed calculation procedures can be found at http://bit.ly/3rQft6X. In this paper we will focus on the interpretation of the results.

The mixture and the separation shown in Table 1 have been called Case 1, and four additional cases described in Table 2 have also been considered. The flowrate of the consecutive components as well as the separation between the key components are the same as in Table 1. For each case the five thermal conditions of feed in Table 3 have been studied. The partially vaporized mixture of Case 1 corresponds to the original problem statement proposal. q is defined as the liquid fraction of the feed[4,5] and varies from < 0 for a superheated vapor (SHV) to > 1 for a subcooled liquid (SCL).

INTENDED LEARNING OUTCOMES

The problem proposed in this work addresses several objectives. It is an exercise included in an Advanced Separation Processes course that is part of the Chemical Engineering Master's program of the University of Alicante (Spain). Some parts of the exercise also could be useful for undergraduates. Multiple problems can be extracted from this material and used in multiple forms covering different learning strategies. Instructors can select parts of the analysis in order for the students to master basic separation concepts or to gain more in-depth knowledge.

Usually these exercises focus on the search for the optimum combination of number of stages and reflux ratio. In this case the influence of the feed stage location and the feed thermal condition are considered. The proposed case study allows the students to learn the importance of knowing and comparing the different alternatives for a particular process and developing conclusions. Moreover, they can discover the utility of using the results of the simulation software for

TABLE 2
Cases to be considered. The flowrates of the consecutive components and the keys recoveries are the same as in Table 1 (98.40 % and 98.24% of recovery of light key, LK, in the distillate and heavy key, HK, in bottoms, respectively). Ci represents a linear hydrocarbon with i carbon atoms. Expected distillate and bottoms temperatures are their bubble or dew temperatures calculated from the compositions obtained from the preliminary mass balance.

Case	Components	LK	HK	Column Pressure (bar)	Condenser type	Feed bubble/dewpoint temperatures (K)	Expected distillate temperature (K)	Expected bottoms temperature (K)
1	C1, C2, C3, C4, C5, C6	C3	C4	21.7	Partial	217.3 / 389.7	294.1	426.6
2	C6, C7, C8, C9, C10, C15	C8	C9	2.17	Total	410.7 / 496.7	390.4	479.8
3	C5, C6, C7, C8, C9, C10	C7	C8	2.17	Total	376.0 / 426.9	356.2	446.3
4	C5, C6, C7, C8, C9, C10	C5	C6	2.17	Total	376.0 / 426.9	333.6	411.6
5	C5, C6, C7, C8, C9, C10	C9	C10	2.17	Total	376.0 / 426.9	370.5	478.9

TABLE 3
Feed phase conditions at column pressure (indicated in this Table) taken into account for each case. $\alpha_{LH, HK}$ is the relative volatility of light key component with respect to the heavy key.

		Case 1 P = 21.7 bar		Case 2 P = 2.17 bar		Cases 3, 4 and 5 P = 2.17 bar			
Feed phase condition	**q**	**T (K)**	$\alpha_{LK,HK}$	**T (K)**	$\alpha_{LK,HK}$	**T (K)**	$\alpha_{LK,HK}$ (Case 3)	$\alpha_{LK,HK}$ (Case 4)	$\alpha_{LK,HK}$ (Case 5)
Superheated vapor (SHV)	< 0	399.8	1.840	520.0	1.488	440.0	1.762	1.808	1.733
Saturated vapor (SV)	0	389.7	1.916	496.7	1.554	426.9	1.826	1.873	1.791
Partially vaporized (PV)	0.34	357.1	2.204	447.2	1.734	410.9	1.913	1.964	1.866
Saturated liquid (SL)	1	217.3	6.362	410.7	1.922	376.0	2.155	2.207	2.088
Subcooled liquid (SCL)	> 1	205.4	7.277	400.0	1.988	360.0	2.294	2.346	2.200

purposes other than the mere simulation or design of units and the importance of not using these simulation packages as a black box. Similarly, the instructor can highlight the usefulness of the binary distillation concepts, well known from previous courses, for understanding multicomponent distillation behavior. There are several points useful for reinforcing the idea of the appropriate use of the simulator. These include the need to know which thermodynamic models should be selected and why, the importance of checking the fulfillment of assumptions of approximate methods, and how to locate and export results that are useful for carrying out additional calculations.

We recommend that the problem be solved by the whole class with each student or small group of students devoted to one of the different parts of the work. It is most appropriate to be assigned as an out-of-classroom activity, guided by the instructor who balances the tasks for each student or team and takes into account the time needed for their resolution. Additional data necessary for some calculations, such as the cost of columns, should be provided to students, highlighting the importance of considering aspects such as the equipment sizes and the required properties of heating or cooling agents. Finally, the results can be shared, analyzed, and discussed in the classroom with all the students.

METHODOLOGY

In order to solve the proposed exercise, the following procedures and tools have been used:[1,4,5]

- The Soave-Redlich-Kwong (SRK) equation of state (EOS), which is applicable to hydrocarbon mixtures at all pressures and non-cryogenic temperatures,[1] has been selected for calculating the vapor-liquid equilibrium (VLE) of mixtures.
- VLE calculations have been performed with the Flash unit of ChemCAD v7.1.1.
- The approximate design or simulation of distillation columns has been carried out using the Fenske-Underwood-Gilliland (FUG) method, with the Kirkbride or the Fenske equations for the optimal feed stage location, and using the Shortcut distillation unit of the above-mentioned ChemCAD version.
- The rigorous simulation of distillation columns uses the rigorous simultaneous correction distillation (SCDS) column of ChemCAD, which is a rigorous multi-stage vapor-liquid equilibrium module that simulates any single column calculation of two-phase or three-phase distillation systems. It applies a Global Newton or simultaneous correction method.[6]

It is assumed that students have previously learned how to select the best thermodynamic model or ChemCAD unit to perform the different calculations involved.

According to King,[7] for a given number of equilibrium stages, the optimal feed stage location is the one requiring the lowest reflux to obtain a given separation between key components. Moreover, several rules of thumb are widely used for determining the optimum feed location stage in simple and efficient ways. Among them, Kister[6] suggests the following for the optimum feed stage:

- **It is the stage where the ratio of key-component mole fractions in the liquid on the feed stage (x_{LK}/x_{HK}) is the closest to this ratio in the liquid portion of the feed stream at tower pressure.** The key components, i.e. light key (LK) and heavy key (HK) components, are defined as those which are being separated in the column. The values for calculating this ratio in the feed stream have been obtained with the Flash unit of ChemCAD from the VLE calculation of the feed steam at the specified thermal conditions. In the liquid phases through the column, x_{LK}/x_{HK} is calculated from the liquid mole fraction profiles resulting from the rigorous simulation using the SCDS unit of ChemCAD.

- **It is the stage provided by the intersection of the operative lines on a Hengstebeck diagram.** The method of Hengstebeck, which is described by Kister,[6] is an approximated method for designing multicomponent distillation columns. It is based on defining equivalent binary streams having only the key components in the same relative amount as in the original multicomponent mixture. The method of Hengstebeck provides tools to calculate the binary equivalent equilibrium x_e - y_e curve as well as the effective liquid and vapor streams in the rectifying (L_e and V_e) and stripping (L_e' and V_e') sections, where molar overflow is considered constant. After that, the problem is solved in exactly the same way as the McCabe-Thiele method. In this work the flowrate and composition profiles resulting from the SCDS-rigorous simulation of the columns have been used to calculate the equivalent binary equilibrium curve and internal flowrates.
- **It is the stage yielding the most equal slopes on both sides of the feed stage in a key ratio plot.** The key ratio plots, i.e. the plots of the log (x_{LK}/x_{HK}) profile through the column, are described by Kister[6] as a tool to check the appearance of "retrograde" or "reverse" distillation. They indicate when the column operates near total reflux or minimum reflux, and the feed stage is reflected by the appearance of some extent of reverse distillation. The reverse distillation is very well explained by King[7] and Kister[6], and results in the increase of the HK and the decrease of the LK when we go up the column, i.e. the reverse of the expected behavior. This is due to the presence of multiple heavy non-key components in the rectifying section such that the behavior of the HK is that of a light component; similarly, there may be many light non-key components in the stripping section yielding the behavior of the LK as that of a heavy component. In this work the LK and HK mole fractions needed for the construction of key ratios graphics have been obtained from the SCDS-rigorous simulation of the columns.
- **It is the stage resulting from the application of every approximate or rigorous analytic technique.** In this work the results of the application of the approximate equations of Fenske and Kirkbride,[1,4] provided by the Shortcut distillation tool of ChemCAD, have been considered.
- **It is the stage yielding the lowest values of reboiler duty or the lower reflux.** Students usually consider that the optimal feed stage is the one minimizing the heat supply needed in the reboiler, understanding that it should be the one yielding the lowest operating costs and, therefore, the lowest total cost. This approach, as well as that suggested by King[7] based on the location giving the lowest reflux flowrate, will be also checked.
- **The true optimal feed stage has been considered as that yielding the lowest column costs.** For a given number of theoretical stages, it seems reasonable that the true optimal feed stage will be that yielding the lowest column cost. This work considers that this criterion gives the "true" result, and it is compared with results given by other methods.

All these concepts should be known by the students, who have previously been instructed in the correct use of the simulator to solve phase equilibrium and multicomponent distillation problems, as well as in the different methods for selecting the feed stage.

RESULTS AND DISCUSSION

The following discussion focuses on the results obtained and their interpretation. More information about calculations and details of the results can be found at http://bit.ly/3rQft6X.

Answers to the Proposed Questions

a) Should a total condenser be used for this column?

The condenser requires a sufficient difference of temperature between the available refrigerant, in this case cooling water at 291 K, and the distillate, at higher temperature, in order to provide the required driving force for the heat transfer. Total condensers yield a liquid distillate at its bubble point or as a subcooled liquid. In this case the bubble point temperature of the distillate in Table 1 at 21.6 bar is 194.6 K, lower than that of cooling water (291.5 K); therefore, the use of a total condenser is not possible. The condenser pressure has been estimated considering that the pressure drop specified in the problem statement only occurs between the feed stage and the condenser.

Partial condensers yield a vapor distillate at its dew point. They are used when there are very light components, as it occurs in this case with methane and ethane in the feed stream. Consequently, high column pressures and/or low condenser temperatures are required to condense these very volatile components in a total condenser, and the use of a partial condenser avoids the use of costly refrigeration. In this case the difference between the dew point temperature of the distillate (294.0 K) and that of the available cooling water is very small (only 2.5 K) and it cannot be used as a refrigerant for a partial condenser either. Thus, we can conclude that this separation will require a partial condenser operated with another cooling agent at a sufficiently lower temperature.

b) What are the feed temperature, the equilibrium ratios (K-values), and relative volatilities with reference to C4?

The VLE calculation for the feed stream in Table 1, 66% vaporized at 21.7 bar, yields a temperature of 357.1 K and the equilibrium ratios, K_i, shown in Table 4. For each component i, the relative volatilities with respect to the HK component (C4), calculated as K_i/K_{C4}, are also shown. The main conclusion is that the relative volatility of the LK is high enough to achieve the separation by distillation. Moreover, the volatilities of the non-key components permit the separation suggested in the preliminary mass balance on Table 1.

TABLE 4
Equilibrium ratios (K_i) and relative volatilities ($\alpha_{i,C4}$) for the feed stream 66% vaporized at 21.7 bar (T = 357.1 K).

Component	C1	C2	C3	C4	C5	C6
K_i	8.610	2.976	1.351	0.613	0.287	0.137
$\alpha_{i,,C4}$	14.05	4.855	2.204	1.000	0.468	0.223

c) If the reflux ratio is 1.3 times the minimum reflux, what is the actual reflux ratio? How many theoretical plates are needed in the rectifying and stripping sections?

In order to answer this question, the approximate design of the column was performed using the Shortcut unit of ChemCAD, using both the Kirkbride and Fenske equations for the feed stage location and the needed specifications given in the initial problem statement. The results obtained are:

- Reflux ratio, R = 1.171
- Number of theoretical stages (condenser and reboiler included), N = 23.9 (it will be rounded up to 24 for the rigorous simulation)
- Feed stage (considering the condenser as first stage) given by the Kirkbride equation, NF = 12.7 (it will be rounded up to 13 for the rigorous simulation). Consequently, the column has a partial condenser (stage 1), 11 equilibrium stages in the rectifying section (stages 2 to 12), 11 equilibrium stages in the stripping section (stages 13 to 23, with the feed stage, 13, as the first one in this section), and the reboiler (stage 24). According to Seader et al[1], the Fenske equation should only be used for symmetrical mixtures and separations, which gives NF = 11.8 (rounded up to 12).

As the students have been instructed, these results provide the starting point for the rigorous simulation and, moreover, will allow the comparison proposed in the next question. This column is further referred to as Column A.

d) Compute the separation of species. How will the separation differ if a reflux ratio of 1.5, 15 theoretical plates, and feed in the ninth plate are chosen?

The separation of components predicted by the Shortcut module for Column A is practically the same as that shown in Table 1. The column proposed for comparison has been simulated with the Shortcut module in ChemCAD and with the same specifications for the feed stream, condenser, column pressure and C4 split as Column A, with the exception that N = 15, NF = 9, and R = 1.5. This column is further referred to as Column B. The main differences are that Column B yields lower C3 recovery in the distillate and higher heat duties than Column A. The interpretation of these results is not easy, because Column B has a higher reflux ratio but a lower number of stages than Column A. Thus, the study of cases available for the Shortcut module in ChemCAD is very useful for this comparison. The study of the requested separation of C3 in Column A with 15 theoretical stages needs $R/R_{min} \approx 2.88$, i.e. $R \approx 2.6$, much higher than the value specified, R = 1.5. Thus, the lower reflux ratio, the lower the separation. The same result is obtained from a sensitivity analysis on Column B, modifying the reflux ratio to determine the corresponding C3 flowrate in the distillate.

e) Compute the temperature and concentration in each stage. What is the effect of feed plate location? How will the results differ if a reflux ratio of 1.5 and 15 theoretical plates are used?

The column profiles are obtained from a rigorous simulation of the column that provides the values of the variables corresponding to the two phases leaving each theoretical stage. This is accomplished using the SCDS unit within ChemCAD. Columns A and B have been simulated, specifying the key components recoveries shown in Table 1 as separation requirements. It is worth mentioning that the results obtained for the distillate and bottoms products as well as for the condenser and reboiler duties are very close to those obtained by the approximate Shortcut calculations, thus showing that the FUG method assumptions (constant molar overflow and approximately constant volatilities) are adequately fulfilled.

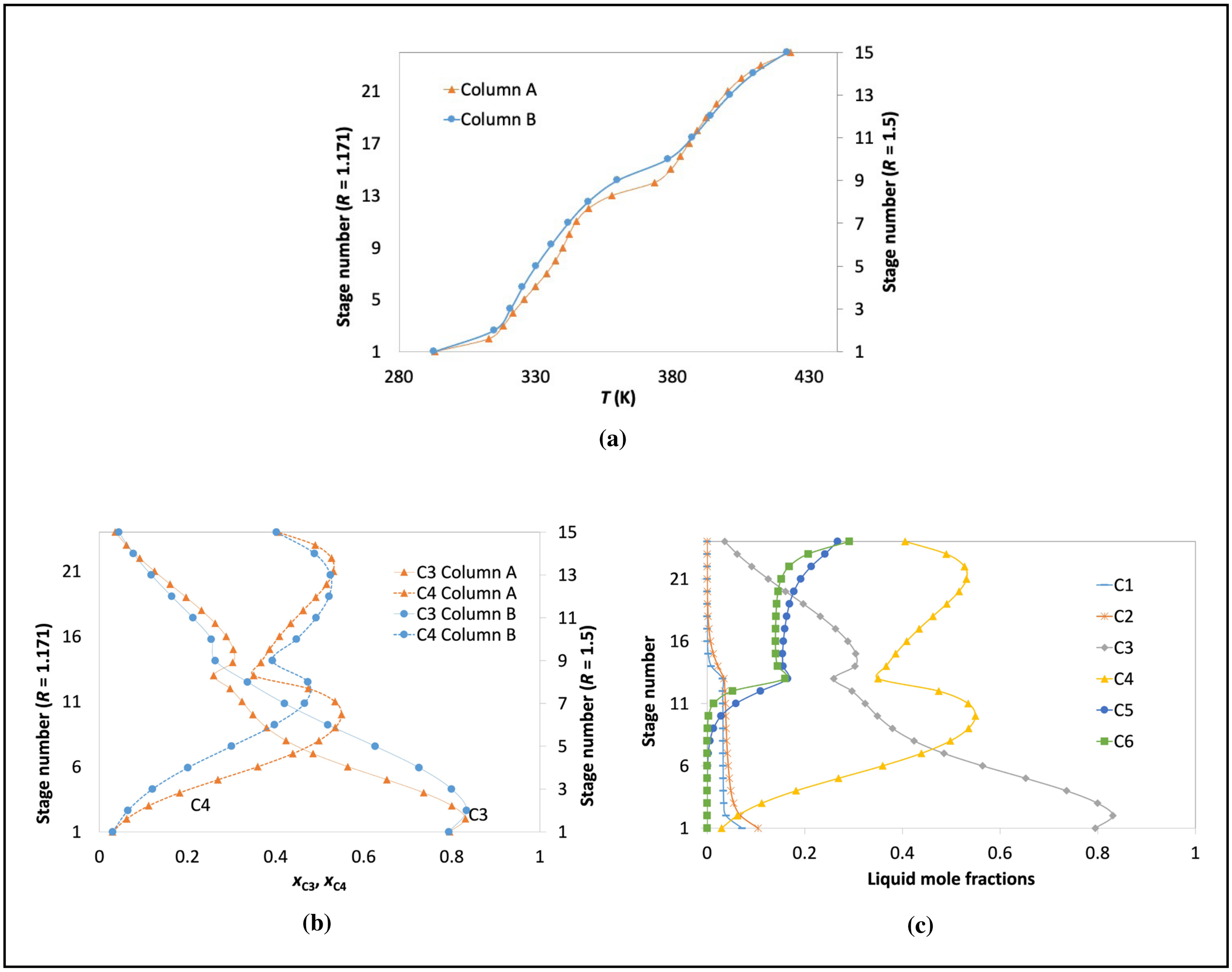
Figure 1 shows the temperature and key component liquid mole fraction profiles corresponding to Columns A and B. As seen in Figure 1a, the temperature profiles are very similar, but Column A shows a higher temperature in the rectifying section and only slightly lower (or equal) than Column B in the stripping section. This is also reflected by the inner streams of Column A having higher C4 and lower C3 contents in the rectifying section than Column B, and the opposite in the stripping section, as Figure 1b reflects. These profiles can be qualitatively explained based on a McCabe-Thiele diagram; for Column A, with lower reflux ratio, the operative lines are closer to the equilibrium curve. As a result, the driving force for the separation in each equilibrium stage is lower, and the rectifying section, where streams are enriched in the LK component, allows lower C4 removing and C3 enriching. This is the reason why more stages are required to achieve the same separation. Similarly, in the stripping section, where streams are enriched in the HK component, the lower reflux ratio results in the lower driving force and lower separation, so streams in Column A have higher C3 and lower C4 contents. The students learned the binary separation concepts and calculations in previous courses on Separation

Processes. Here, the McCabe-Thiele diagram is used to justify and interpret multicomponent behaviors, which the instructor can take advantage of to highlight and reinforce this previous knowledge.

Figure 1c shows the composition profiles of column A, while Column B exhibits a similar behavior. The obtained profiles reflect the expected behavior on a column with good separation between key components, as the following statements indicate:

- The maxima of C3 and C4 approaches to the top and bottom of the column, respectively, which are characteristic of key components.[4,6] They appear near the condenser due to the scarcity of components heavier than C3, with the consequence that C3 shows the behavior of a heavy component. Similarly, C4 behaves as a light component near the reboiler because the scarcity of compounds lighter than C4.
- Components lighter than C3 practically do not appear in the stripping section and show a near constant concentration in the main part of the rectifying section. Similarly, components heavier than C4 are practically absent in the rectifying section and constant in the stripping section. This is the behavior assumed by the Hengstebeck method,[6] which is applied in the next section.

Figure 1c also shows that there is some reverse distillation around the feed stage due to the existence of so many heavy components above of the feed stage, producing C4 as a light component, and so many light components under the feed stage, producing C3 as a heavy component.



(a)

(b)

(c)

Figure 1. *(a) Comparison between the temperature and (b) the liquid C3 and C4 mole fraction profiles in Columns A and B. (c) Liquid composition profile in Column A. Stage 1 is the partial condenser and the last stage is the reboiler. The vapor profiles follow similar patterns to (b) and (c).*

Comparison Among Different Criteria to Select the Optimal Feed Stage

The analysis of the influence of the operating variables on the behavior of a distillation column is not an easy task because different results are obtained depending on the variables selected. For instance, given a column with the same number of theoretical stages where the reflux ratio and the reboiler duty are specified and the feed stage changes, different distillate and bottoms products are obtained with similar operating costs. Nevertheless, if the specifications are based on mole fractions, flowrates, or recoveries of key components, the product streams obtained when the feed stage is modified will be very similar, but the requirements of reflux and condenser and reboiler duties, and consequently the operating column cost, will be different. The study carried out in this section is based on the specification of the number of theoretical stages and the required separation, and different ways to locate the optimal feed stage will be studied. The column selected is the one previously designed with the Shortcut method, with 24 theoretical stages to obtain the separation shown in Table 1 with $R/R_{min} = 1.3$. The required simulations for the application of the different criteria for selecting the optimal feed stage have been performed with SCDS, varying the feed stage location in the range of 2-23 (stage 1 is the partial condenser and stage 24 is the reboiler). The complete optimization process would require the calculation of the cost of a series of columns with different number of stages, each one of them with the optimal feed stage location.

Feed stage from the comparison between x_{LK}/x_{HK} in each stage and in the liquid portion of the feed stream. The VLE calculation of the feed stream provides the keys ratio value in the liquid phase, i.e. 0.2030/0.2284 = 0.8888, for the comparison with the values obtained from the liquid mole concentration profiles in the column. The C3 and C4 concentration profiles corresponding to each simulated column, each one with a different feed stage location, have been extracted from the SCDS simulation, and the stage showing the keys ratio closest to 0.8888 has been identified. Consequently, the column for which this stage was the nearest to the feed stage has been identified as that having the optimal feed location, according to this criterion. As an example, Table 5 shows the results when the feed stage is moved between stages 7 and 11, allowing us to conclude that the optimal feed stage appears at *NF* = 9. It is worth mentioning that this criterion is highly time consuming because not only do 21 simulations have to be completed, but also the liquid profiles for each one of them have to be extracted for the keys ratio calculation and comparison.

TABLE 5
Location of the stage with the closest keys ratio to that in the liquid portion of the feed stream when *NF* changes from 7 to 11.

Feed stage, *NF*	7	8	9	10	11
Stage with x_{C3}/x_{C4} closest to 0.8888	12	11	8	8	8

Feed stage from the intersection of the operative lines in a Hengstebeck diagram. The method of Hengstebeck[6] provides tools to determine the equivalent binary flowrates of the streams in each section of the column from the calculated limiting compositions of the non-key components, i.e. from the composition of non-key components in the zone where they have constant mole fraction. Figure 1b shows that this assumption is adequately fulfilled in this case, therefore the method of Hengstebeck can be applied.

Once the equivalent binary composition of feed, distillate, and bottoms, the equivalent binary equilibrium curve, and the equivalent operative lines have been calculated, the problem is solved in exactly the same way as the McCabe-Thiele method for binary mixtures. The details of the application of the Hengstebeck method in this example can be found at http://bit.ly/3rQft6X, and the obtained Hengstebeck diagram is shown in Figure 2.

As in the McCabe-Thiele method, the feed stage corresponds to the intersection of the operative lines. The results from Figure 2 suggest that the optimal column has 16 theoretical stages, with *NF* = 8. Therefore, considering the same ratio of the number of stages in each section, it can be concluded that for a 24 theoretical stages column, *NF* = 12. Note that this method is substantially more time consuming than the previous one and, moreover, requires multiple assumptions. Thus, at least a priori, it should not be selected as the best method based on convenience.

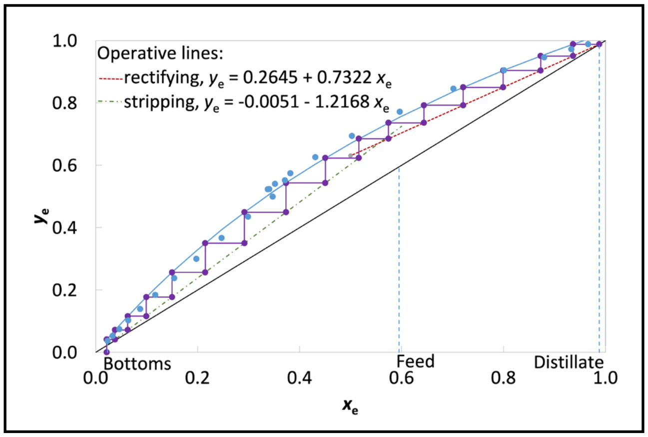
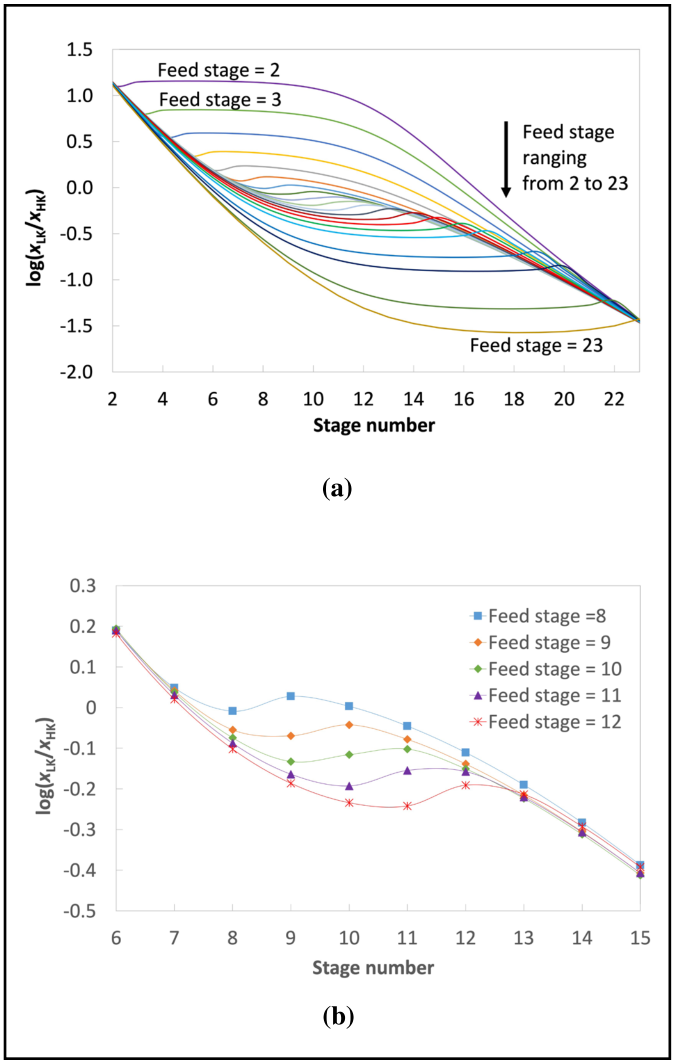


***Figure 2.** Hengstebeck diagram corresponding to the separation shown in Table 1. x_e and y_e represent the equivalent binary compositions of multicomponent mixtures.*

Feed stage from a key ratio plot. Using this criterion, the optimal feed stage is the one yielding the most equal slopes on both sides of the feed stage in a key ratio plot. Figure 3 shows the key ratio plots obtained when the feed stage moves from stage 2 to stage 23. When the feed stage is between stages 2 and 7, the slope of the key ratio plot in the upper part of the rectifying section is almost horizontal, thus suggesting the existence of a zone with almost no changes in the key ratio, i.e. almost no separation between keys. Similarly, when the feed stage approaches the bottom section of the column, in the range of 13 to 23 stages, the last stages of the rectifying section show quite horizontal profiles. Therefore, the optimal feed stage should be in the range of 8 to 12 stages. The enlarged key ratio plots in this range suggest that the optimal location seems to be stage 10. Note that this way of selecting the optimal feed stage is also very time consuming, and the results are not very clear because the differences among the cases with *NF* equal to 9, 10 and 11 are negligible.

Feed stage from the Kirkbride and Fenske equations. These are approximated equations, very well described in textbooks focused on multicomponent distillation,[1,4] which calculate the optimal feed stage exclusively from a preliminary mass balance, as shown in Table 1. According to the results described previously, the optimal feed stage locations provided by equations of Kirkbride and Fenske are, respectively, 13 and 12. These calculations are easy and fast and can be performed by hand. Nevertheless, the validity is relative because the approach depends on the applicability of the empirical equations to each particular case. Seader et al.[1] report a comparison of the results of both equations with the exact result, showing noticeable deviations.

Feed stage selected as that yielding the lowest reflux flowrate. This criterion is the first one suggested by King.[7] It can be justified taking into account that, as can be expected and as shown in the next sections, the lowest reflux flowrate also corresponds to the situation with the lowest energy requirements and the lowest column operating costs. Consequently, this criterion could be considered as equivalent to the one giving the true result, i.e. based on the most economical column. Moreover, by using chemical process simulation software, this criterion can be easily applied by performing a sensitivity study where the feed stage location is selected as the independent variable and the reflux rate as the dependent variable. In this case NF has been varied from 2 to 23 in 21 equal steps, and the calculated molar reflux rate has been obtained. Figure 4a shows the results obtained, yielding stage 10 as the optimal feed stage. The pathway observed in Figure 4a will be repeated when variables other than the reflux rate are considered, thus appearing as alternative variables to be taken into account for the optimization of the feed stage location. Moreover, the fact that the area surrounding the minimum of Figure 4a was so flat indicates that small – or not so small – deviations from the optimal feed stage will not have a noticeable influence on the operation of the column.



(a)

(b)

Figure 3. *Key ratio plots corresponding with (a) columns varying the feed stage from stages 2 to 23 and (b) enlarged version for columns varying the feed stage from stages 8 to 12.*

Feed stage selected as the one yielding the lowest reboiler duty. As in the previous case, this criterion can be easily applied with simulation software by performing a sensitivity study and choosing reboiler duty as the independent variable. In this work the exchanged heat at the reboiler, Q_{reboiler} , and the condenser, $Q_{\text{condenser}}$, have been checked. As expected, the sum ($Q_{\text{condenser}} + Q_{\text{reboiler}}$) has almost the same value for all the cases because it corresponds to the difference between the enthalpy of the outlet and inlet streams, which is very similar in all the cases because the products are always almost the same. Some deviations observed when the feed stage approaches the column top and bottom could be explained considering that, in such cases, there are not enough theoretical stages to eliminate the heavy non-key components from the distillate or the light non-key components from the bottoms product. The results obtained are represented in Figure 4b. According to this criterion, the optimal feed stage is *NF* = 10, which also yields the lowest condenser duty and reflux flowrate. Moreover, the profiles shown by these variables follow exactly the same pattern of variation. Therefore, both criteria could be considered as equivalent.

Feed stage selected as the one yielding the lowest total column cost. Given a theoretical number of stages, the exact result for the optimal feed stage location should be the one corresponding to the economic optimum. The column costs calculation is a long process that requires the sizing of the column, condenser, and reboiler to obtain the capital costs, as well as the selection of the adequate utilities, i.e. the adequate cooling and heating agents and their corresponding loads, in order to evaluate the operating costs. In this work the column sizing has been performed by following the procedure shown of Sinnott and Towler.[8] The capital costs have been calculated according the equations, figures, and relationships provided by Turton et al.[4] based on the module factor approach, and the operating costs have been obtained as Ulrich and Vasudevan[5] proposed. The criteria and considerations considered to complete these calculations are shown in http://bit.ly/3rQft6X, and the results obtained are represented in Figure 4c. The lowest cost corresponds to the column with the feed stage at stage 10, with very small cost changes when the feed stage location moves between stages 8 and 13. In this example the utility costs are noticeably higher than the fixed costs, which is very common because of noticeably high energy costs. Therefore, it can be concluded that the feed stage location involving the lowest reflux flowrate and, thus, requiring the lowest utilities consumption, should also

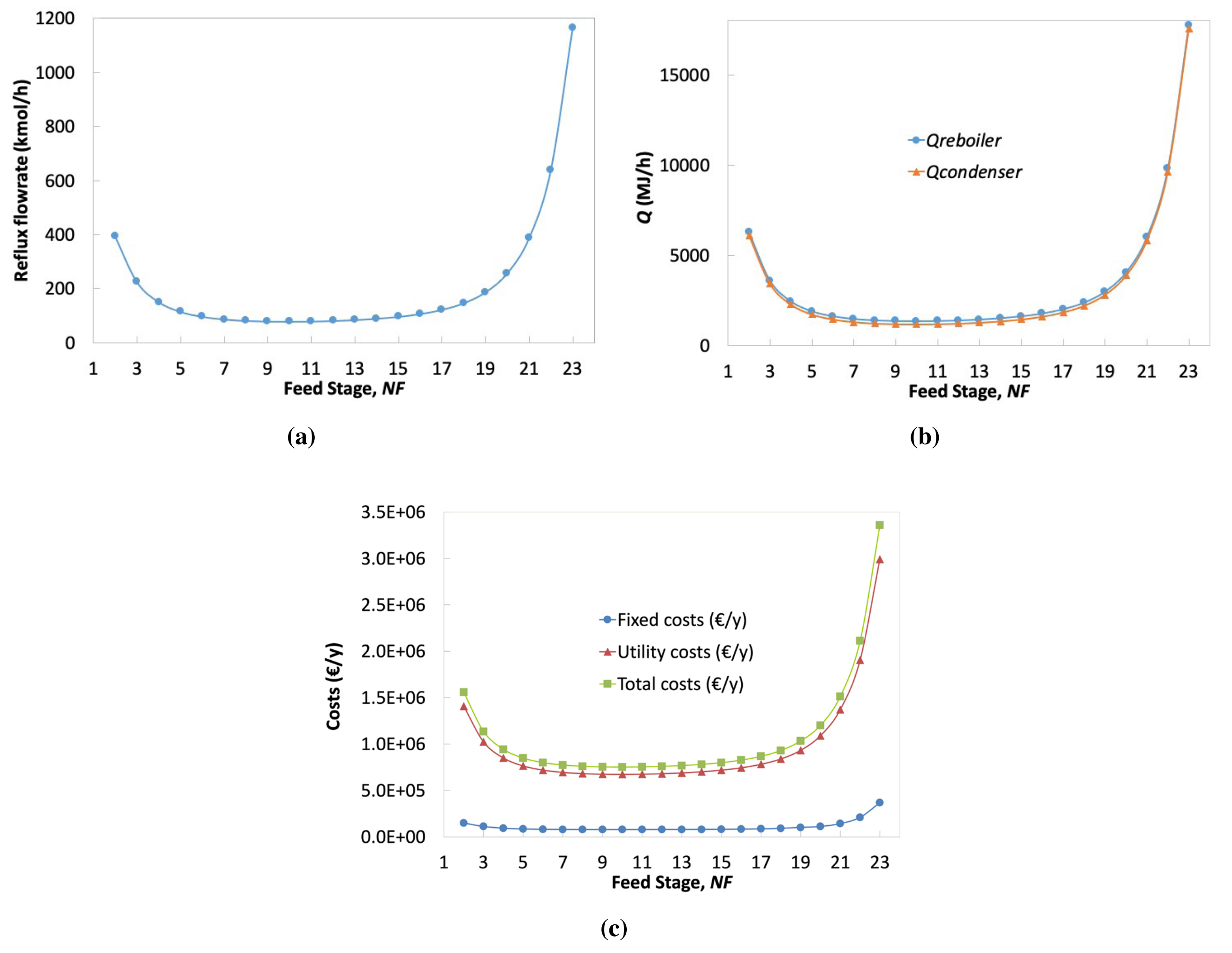


Figure 4. *(a) Calculated reflux flowrate, (b) Energetic requirements and (c) Costs for columns with N = 24 and ranging the feed stage location from 2 to 23.*

be the one yielding the lowest total cost, i.e. the optimum. Moreover, the column having the lowest reflux flowrate also corresponds to the one with the lowest reflux and reboil ratios (the reboil ratio, *V/B*, is defined as the ratio between the flowrates of vapor from the reboiler and bottoms), liquid and vapor flowrates through the column, column diameter, and heat exchanger sizes. Consequently, it not only has the minimum utility cost, but it also shows the minimum fixed cost. As seen in Figure 4, the pattern followed by costs is exactly the same of that shown by the reflux flowrate and duties, as well as by the other above-mentioned variables. Thus, despite this pattern not being generalized, it makes sense to consider that the three criteria for selecting the optimal feed stage could be considered as equivalent.

Final comparison among the different criteria for locating the optimal feed stage. Table 6 summarizes the results yielded by the different methods considered for selection of the optimal feed stage. Considering that the true value corresponds to the lowest column costs, criteria based on looking for the lowest reflux flowrate or energetic requirements are completely equivalent. These criteria avoid the need of long and tedious cost calculations but involve the rigorous simulation of several columns that, in the case of separations showing convergence problems or columns with high number of stages, could be very time consuming. Despite the fact that these results cannot be generalized, it seems that the Hengstebeck and keys ratio comparison methods, also based on rigorous simulation results, tend to give worse results and, moreover, their application requires significant effort. The method based on the key ratio plots is also time consuming and, although in this case yield the correct result, the selection of the adequate plot is not so clear. The application of the Kirkbride and Fenske equations do not give the best results, but they are within the range of acceptable values, as Figure 4 reflects. Moreover, they involve very fast and simple calculations, only needing the preliminary mass balance of the column.

TABLE 6
Optimal feed stage location from different criteria.

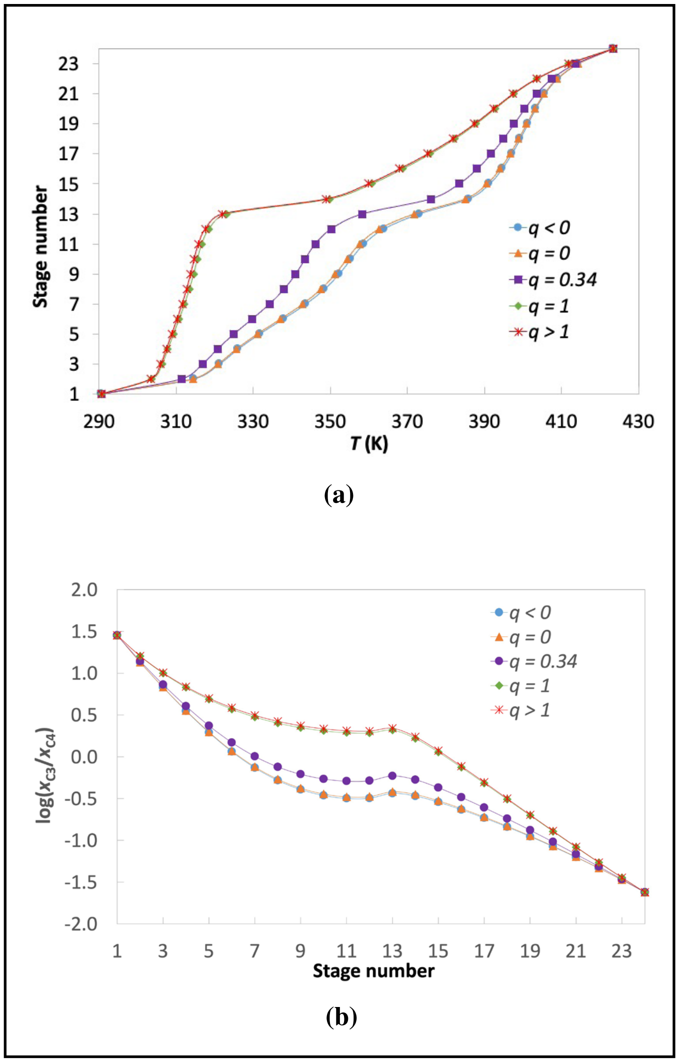
Method	Feed stage, *NF*
Comparison of keys ratio	9
Hengstebeck method	12
Key ratio plot	10
Kirkbride equation	13
Fenske equation	12
Lowest reflux flowrate	10
Lowest reboiler duty	10
Lowest column cost	10

As a conclusion, the use of the Kirkbride and Fenske equations should be limited to very preliminary design steps or by hand calculations when a chemical processes simulation package is not available. In this case, whenever is possible, we recommend performing a further optimization. This can easily be done by using the sensitivity study tool in the simulation package, with the feed stage as the independent variable and the reboiler duty or the reflux flowrate as the dependent variable. As Figure 4 reflects, the optimal feed stage, at the minimum of the curves, appears in a wide zone that is almost flat. This pattern suggests the possibility that small displacements of the feed stage location near the minimum would not have noticeable influence on the operation and cost of the column. Therefore, the deviations involved in the use of the approximated methods for locating the optimum feed stage location should have not a significant impact. This behavior has been also observed when the feed phase condition was modified and for other mixtures with different ranges of VLE temperatures. Other variables, such as the liquid and vapor flowrates, the diameter of each section of the column, the flowrates of cooling and heating agents, the heat exchange areas of condenser and reboiler, and the reflux and reboil ratios, also exhibit the same pattern of variation. Consequently, any of the variables described could be selected in order to locate the optimal feed stage.

Effect of the Thermal Feed Phase Condition

In order to check the effect of the feed phase condition, we performed a study of a distillation column to give the separation proposed in the five cases described in Tables 1 and 2 with the five thermal characteristics shown in Table 3. First, we compare the results for Case 1, at the different thermal conditions considered, for a column with $N = 24$ theoretical stages and the feed stage location at stage $NF = 13$ to give the required keys recovery of Table 1. Next, we consider the effect of the Case 1 feed temperature on the preliminary column design, i.e. on the number of stages and the required reflux ratio to give the specified separation. Finally, the behavior of Cases 2 to 4, with the five thermal conditions specified in Table 3, is summarized and analyzed. In all the cases, the comparison among the patterns of variation of the operation variables with the feed stage location has been checked, showing the same behavior than that of Figure 4.

Effect of the feed phase condition for a given number of theoretical stages and feed stage location. Figure 5 shows the temperature profiles and the key ratio plots obtained when a column with 24 theoretical stages ($N = 24$) with feed stream entering at stage 13 ($NF = 13$) is simulated with the feed stream corresponding to Case 1 (Tables 1 and 2) and the five phase conditions considered in Table 3. The tray temperatures increase as the feed temperature increases (Figure 5a). Moreover, the temperature profiles for cases SHV and SV (the vapor phase feeds) are nearly coincident, as well as those for cases SL and SCL (the liquid phase feeds). As expected, the temperatures of stages 1 (condenser) and 24 (reboiler) are the same for the five cases because the specified separation is also the same. The temperature range in the column is between 294.2 K and 426.6 K; the partially vaporized feed temperature (Table 2) is within this range and near the mean of the end point values, but the liquid feeds are colder than the distillate, and the vapor feeds are relatively close to the bottoms temperature. Therefore, the vapor feeds tend to heat and the liquid feeds tend to cool the column. The higher temperature profile corresponds to higher heavy key concentration profiles, i.e. lower x_{C3}/x_{C4} values through the column (see Figure 5b). This behavior will have consequences on both the reflux ratio and the rest of related variables required for obtaining the specified separation. According to Figure 5b, in this specific case, the feed temperature does not seem to significantly affect the reverse distillation phenomena near the feed stage.



(a)

(b)

Figure 5. *Temperature profiles (a) and key plots (b) corresponding to a column with N = 24 and NF = 13, fed with the mixture corresponding to Case 1 with the different phase conditions considered in Table 3.*

Figure 6 shows the liquid and vapor profiles corresponding to the separation of mixtures with the five feed phase conditions of Case 1. As expected, the liquid (subcooled or saturated) entering the column mixes with the liquid coming from the rectifying section, giving higher liquid flowrate in the stripping section than that corresponding to merely the sum of both flowrates, because the liquid, at lower temperature than that of the feed stage, causes the condensation of a part of the vapor coming from the stripping section. Consequently, if the bottoms flowrate is constant, the vapor flowrate in the stripping section will be increased.

Similarly, when the feed is a saturated or superheated vapor at higher temperature than that of the feed stage, it mixes with the vapor coming from the stripping section and increases the vapor flowrate in the rectifying section because of the flow of vapor feed and the vaporization of a part of the liquid. Thus, if the distillate flowrate is constant, the rectifying section will show higher liquid flowrates, resulting in higher reflux ratios. It is important to highlight that this behavior is not only related to the VLE characteristics of the feed stream but also to the difference between the temperatures of the feed and of the feed stage. The calculated diameters of each section fit with this behavior, and the diameters in the stripping section increase when the feed temperature decreases.

A comparison of the effect of the feed phase condition based on the condenser and reboiler duties or on the column costs should include the energetic or economic costs involved in heating or cooling the feed stream to specified thermal condition. As the feed temperature increases, i.e. as the enthalpy of the feed stream increases, the energetic requirements at the reboiler decrease because part of the energy required to accomplish the separation is supplied by the feed stream. At the same time, the condenser duty increases because the column temperature has increased but the temperature of the distillate is the same – it has the same composition at the same pressure. Nevertheless, the energy savings at the reboiler would be misleading if the feed stream must be previously heated.

The results obtained reflect that the analysis of the influence of the feed phase condition for a column where *N* and *NF* are specified depend more on the difference between the feed and column temperatures than on the actual phases of the feed stream. One important effect is related to the flowrates circulating through the column. These flowrates affect the column diameters and, consequently, the column's fixed costs. Nevertheless, the total column costs are frequently dominated by the operating costs and, moreover, the influence of the diameter on the column cost is usually less than that of the column height. With respect to the total column

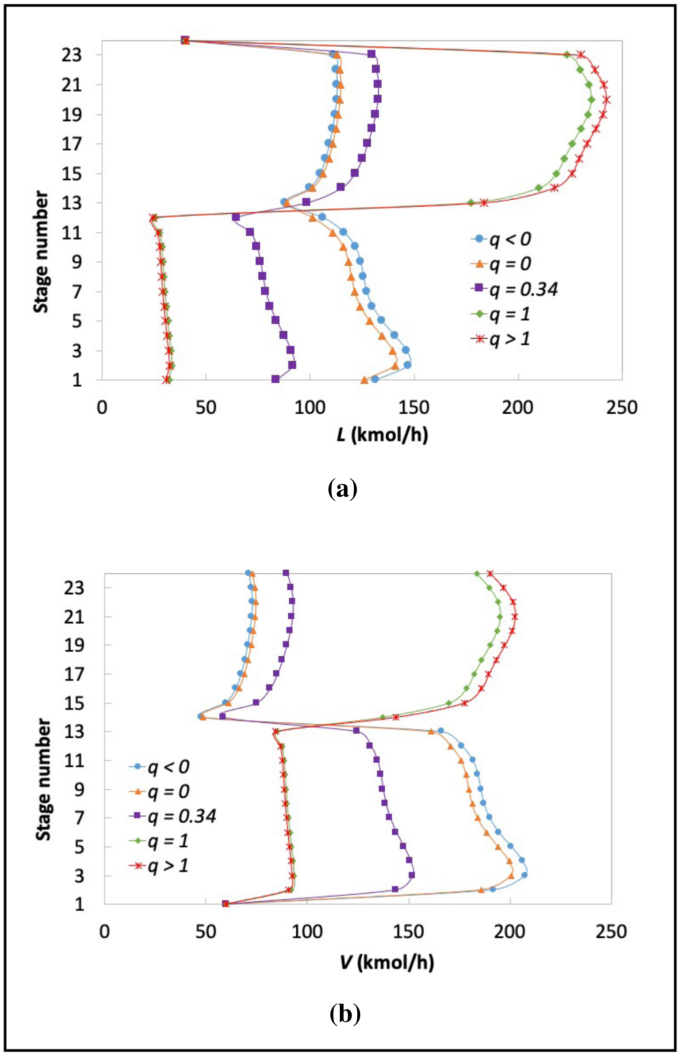


Figure 6. *Liquid (a) and vapor (b) profiles corresponding to a column with N = 24 and NF = 13, fed with the mixture corresponding to Case 1 with the different phase conditions considered in Table 3.*

costs, we must consider whether the feed stream is available at the specified temperature and pressure or if it has to be heated or cooled. In that case, the cost of this energetic change must be also included in the calculations. Another interesting conclusion is that the increase of the reflux ratio and decrease of the reboil ratio as the feed temperature increases suggest that hotter feeds could make the separation between keys in the stripping section easier, whereas colder feeds could make the separation in the rectifying section easier.

When the feed temperature is below the condenser temperature, the hot oil consumption in the reboiler increases dramatically, which is a situation to be avoided. Also, when the feed is at a temperature near that of the reboiler, both the reflux ratio and the refrigerant consumption increase significantly, which is undesirable. With respect to the cost calculation, we must consider whether to include the costs required to modify the feed phase condition.

Effect of the feed phase condition on the design of the column for a given separation. The preliminary design of the columns to achieve the separation given in Table 1 with $R/R_{\min}$ = 1.3 and considering Case 1 (Table 2) with the five feed phase conditions shown in Table 3 has been performed applying the FUG method with the Kirkbride equation for the feed stage location and using the Shortcut unit of ChemCAD. The results are shown in Table 7. As expected, the five cases show the same values of $N_{\min}$ and NF/N because the feed phase condition does not play any role in the Fenske equation for $R_{\min}$ nor in the Kirkbride equation for NF. However, there is a slight decrease in the required number of theoretical stages as the feed temperature increases, but this decrease is not significant, reflected in the 2.4 theoretical stage difference between the extreme cases SHV and SCL. The observed increase of the minimum reflux ratio as the feed temperature increases is related to the previously described effect of the feed stream on the flowrates circulating through the column.

TABLE 7

Preliminary design of columns giving the separation on Table 1 with the five feed phase options on Table 3 and with $R/R_{\min}$ = 1.3.

Case	$R_{\min}$	$N_{\min}$	N	NF	$-Q_{\text{condenser}}$ (MJ/h)	Q_{reboiler} (MJ/h)
SHV	1.2836	10.7	22.9	12.2	1507.5	655.8
SV	1.2538	10.7	23.0	12.2	1472.5	732.9
PV	0.9008	10.7	23.9	12.7	1058.1	1223.5
SL	0.5892	10.7	25.3	13.4	692.5	3120.1
SCL	0.5845	10.7	25.3	13.4	686.9	3234.3

At this point, it is important to remember that the FUG method is an approximate method whose validity is restricted to cases fulfilling the assumptions of the model, i.e. constant molar overflow and small changes of relative volatilities through the column. Figure 6 shows that it is reasonable to accept the hypothesis of constant molar overflow. With respect to the relative volatilities, Figure 7 shows the profiles of relative volatility of the light key obtained for the five columns simulated with $N = 24$ and $NF = 13$. Table 3 shows the relative volatility of the light key component at the five considered feed phase conditions. Despite the assumption of constant volatilities profiles, another aspect should be highlighted. The Underwood equations for the minimum reflux calculation[1] assumes that, for class 2 separations, volatilities are constant in the region between the two pinch-zones (at the rectifying and stripping sections) and would be adequately represented by the relative volatilities at the feed thermal condition. As shown in Figure 7, cases SL and SCL do not fulfill this condition and, therefore, the results obtained with the Shortcut tool should be carefully analyzed. The instructor can take advantage of these situations in order to emphasize the importance of having adequate knowledge of the characteristics of the equations and their application. When they are used as a part of a simulation package, students may tend to view them as a black box without recognizing the underlying assumptions.

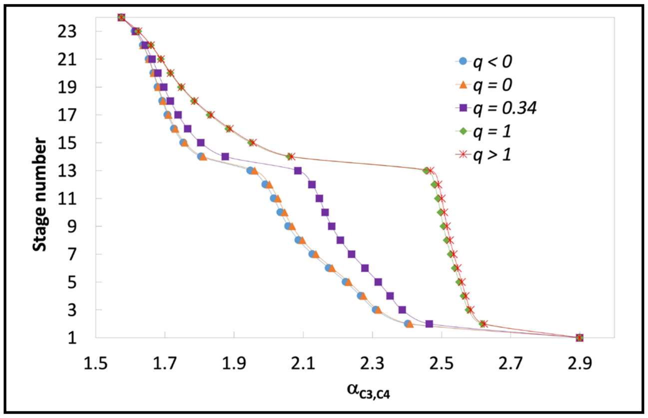


Figure 7. *Light key relative volatility profiles for columns with N = 24 and NF = 13, fed with the five mixtures described in Table 3.*

Can the previously observed behavior be generalized to other mixtures? The answer to this question is not easy because it requires an extensive study of mixtures with different VLE properties being separated over a wide range of operational variables. In this work the proposed study has been extended to other hydrocarbon mixtures covering different VLE temperature ranges and showing different type of separations, shown in Tables 2 and 3. Thus, the following situations have been considered:

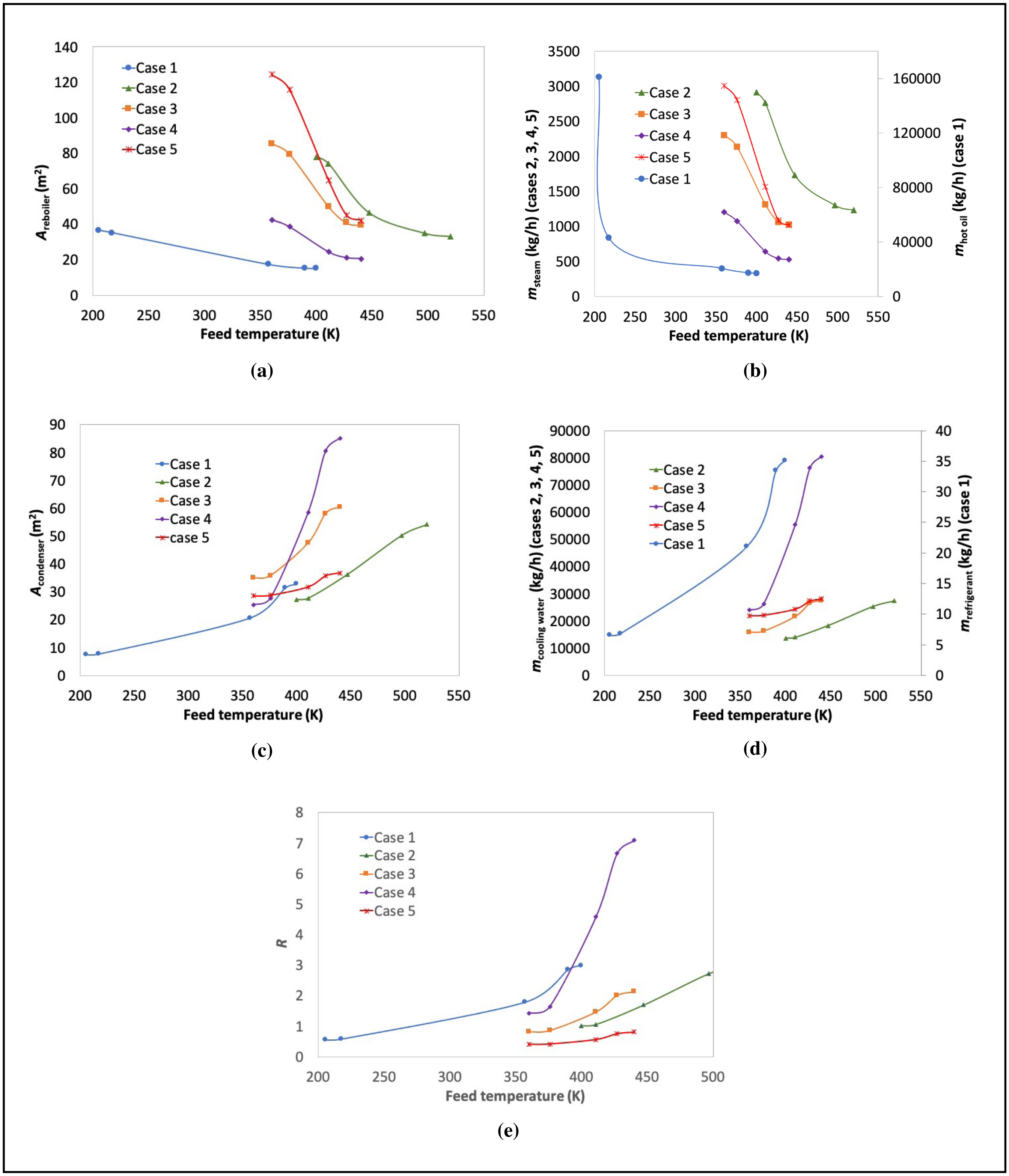
- Case 1: The bubble point of the feed is significantly lower than the distillate temperature
- Case 2: The dew point of the feed is higher than the bottoms temperature
- Case 3: The bubble and dew point of the feed are within the range of the column temperatures

Moreover, the separation specified in Table 1 could be considered relatively close to a symmetrical separation. The study also includes:

- Case 4: The light key component is the most volatile in the feed mixture
- Case 5: The heavy key component is the least volatile in the feed mixture

In Cases 2 and 3 the same flowrates appearing in Table 1 are used. The feed component flowrates for Cases 4 and 5 are also the same as in Table 1; however, the distillate and bottoms compositions have been established on a preliminary mass balance based on the same recovery of key components as in the other cases: 98.40 % of light key in the distillate and 98.24 % of heavy key in the bottoms. According to the VLE characteristics of mixtures corresponding to Cases 2 to 5, the column pressure has been fixed at 2.17 bar in order to use cooling water at the condenser and saturated steam at the reboiler.

The same analysis carried out for Case 1 in order to study the optimal feed stage location and the effect of the thermal condition of the feed stream has been performed for Cases 2 to 5. The results confirm the previously discussed comments and conclusions. Figure 8 shows the evolution with feed temperature of some interesting operating variables for the five considered cases. As expected, the heat exchange area of the reboiler (A_{reboiler}) decreases as the feed temperature increases, as shown in Figure 8a. The largest drop in heat exchange area appears when the feed changes from liquid to vapor. Case 5 shows the more pronounced slope, i.e. the highest difference between the extreme values, thus suggesting that this behavior could be related to the type of separation that, in this case, requires obtaining a bottoms product that is nearly pure HK. Consequently, despite the fact that the bottoms temperature of Cases 2 and 5 is almost the same, the separation is not. Case 5 requires a significantly higher reboil ratio than Case 2, resulting in higher reboiler area. The required flowrate of heating agent (hot oil in Case 1 and steam at different pressures in the other cases), shown in Figure 8b ($m_{\text{hot oil}}$ and m_{steam}, respectively) follows the same trend. Case 1 is the only exception, and it shows the previously discussed dramatic increase of heating agent when the feed temperature is significantly lower than the distillate temperature. Likewise, the heat exchange area of the condenser increases as the temperature increases, with a pattern of variation similar to that at the reboiler. As Figures 8c and 8d show, there is a substantial difference between the areas obtained for liquid and vapor feeds. The highest differences correspond to Case 1, with the highest difference between the feed bubble and dew point temperatures, and Case 4, yielding nearly LK pure distillate. Therefore, it seems that, besides the expected variation of the heat exchange areas, the type of separation is also a factor, and the reboiler and the condenser areas increase when the composition of HK and LK, respectively, approach nearly pure streams. Finally, Figure 8e shows that the reflux ratio (R) increases when the feed temperature increases, which is the biggest difference observed for Case 4. This variation agrees with the corresponding evolution of the R_{min} values. It not only depends on the effect of the feed stream on the vapor and liquid flows inside the column, but also the ease of separation of the key component, i.e. their relative volatilities, the feed composition, and the specified recoveries or concentrations at the distillate and bottoms.



(a)

(b)

(c)

(d)

(e)

Figure 8. *(a) Reboiler heat exchange area, (b) required flowrate of heating agent, (c) condenser heat exchange area, (d) required cooling agent flowrate, and (e) reflux ratio versus feed temperature for the five cases considered in this section.*

CONCLUSIONS

In this work, a problem statement focusing on the behavior of a multicomponent distillation column has been used as a starting point in order to perform a deep study of the different procedures to select the optimal feed stage location and thermal feed phase. This problem allows us to teach students the importance of knowing and comparing the different approaches to solving a problem and developing their own conclusions. The discussion of the results emphasizes the importance and usefulness of the knowledge of the behavior of binary distillation, deeply studied in previous courses, for interpreting and understanding multicomponent separations. The application of approximate and rigorous methods in the design of distillation columns also highlights the importance of not using the simulation packages as a black box. It is critical to check if the assumptions of approximate methods are fulfilled and to know the available tools to make use of the column profiles obtained by rigorous methods.

The analysis performed is based on the assumption that the true optimal feed stage location is the one corresponding with the lowest column costs. This study indicates that this situation is reached when several variables — such as the reflux flowrate, reflux ratio, reboiler or condenser duties, reboil ratio, among others — reach their minimum values. Consequently, the true optimal feed stage can also be considered as the one giving the lowest value of some of these variables. According to this criterion, seeking the optimal feed stage requires the simulation of several columns with different feed stages. This could be time consuming, especially in cases with convergence problems or with a high number of stages. Nevertheless, it should be considered that other methods, such as the key ratio comparison, the key ratio plots, or the Hengstebeck method, are also based on rigorous simulation results but do not give the best result; moreover, they require additional calculations and/or graphical representations. Therefore, if a simulation package is available and the column simulation does not show convergence problems, the optimal feed stage can be obtained from a sensitivity study where the feed stage location is selected as the independent variable and the dependent variable is selected from among of the above-mentioned variables. The approximate Kirkbride and Fenske equations do not give sufficiently good results, and their use should be limited to preliminary design steps or hand calculations. The results obtained reflect that small changes of the feed stage location around the optimum value do not have a noticeable effect on the column behavior. Therefore, the deviations involved in the use of the approximate equations are acceptable.

With respect to the study of the influence of the feed phase condition on the operation of a distillation column, situations in which the feed temperature is not within column temperature range should be avoided because it results in an undesirable increase of some operating variables. The observed increase of the reflux ratio and decrease of the reboil ratio as the feed temperature increases allow us to conclude that hotter feeds could make the separation between keys in the stripping section easier, whereas colder feeds could make the separation in the rectifying section easier. Besides the influence of the feed phase condition on the operating variables, the type of separation also affects the behavior of the column, and the areas of the reboiler and the condenser increase when the required contents of HK and LK, respectively, approach nearly pure streams.

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