



Sultan Qaboos University

College of Engineering

Petroleum and Chemical Engineering Department

Chemical Reaction Engineering

Distillation Column Calculation Theory

Submitted by	ID
Muayad Al-Abri	133322

Supervisor: Dr. Mohammed Al-Abri

Date of Submission: 5/4/2024

Table of Contents

Introduction:	4
Steps needed to solve:	5

Results:.....	6
Part A: Determining the average relative volatility.	6
Step 1: identifying the given information.....	6
Step 2: Calculating the Fractional Recovery of the Components (FR,i):	7
Part B: Determine the minimum number of equilibrium stages using the Fenske equation.....	8
Part C: Estimate the degree of separation of the non-key components using the Fenske equation and compare this with the separation specifications given to see whether they are satisfied.	9
Part D: Revise the separation to meet the required specifications using Fenske equation.	13
Part E: Calculate the minimum reflux using Underwood equation.....	14
Part F: Calculate the actual number of stages using Gilliland correlation.	15
Part G: Calculate the compositions of the top and bottom products.	15
Part H: Estimate the location of the feed tray.	16
Part I: Determine the actual number of stages of a binary mixture using McCabe-Thiele method.	16
Part J: How can analytical procedure be improved to achieve better results.	19
Discussion of results:.....	20
Conclusion:.....	20
Reference.....	21
Appendix 1	21

Figures:

Figure 1: Calculate the number of stages. 17
Figure 2: Calculating Rmin..... 18

Tables:

Table 1:mass balance equations 7
Table 2: Identifying the variables..... 7
Table 3: Solving table 1 mass balances. 8
Table 4:New Nmin..... 10
Table 5:The new fractional recovery 11
Table 6:Second trial. 12
Table 7:Fractional recovery table for second trial 12
Table 8:Change from mole % to weight % 13
Table 9:Final Vmin. 14
Table 10:Composition of top and bottom..... 15

Introduction:

In industry, the distillation process, the oldest and most significant separation technology, is employed for separating liquid mixtures, with a particular emphasis on achieving high component purity. This separation relies on both the differences in boiling points of individual components and their distribution between liquid and gas phases. Various types of distillation, such as simple, fractional, and vacuum distillation, are utilized. Simple distillation, suited for liquids with substantial boiling point differences, involves the evaporation of the most volatile component, followed by its condensation in a condenser. Fractional distillation, on the other hand, is employed to separate components with nearly equal relative volatilities and slight boiling point discrepancies. Vacuum distillation is preferred for mixtures with temperatures below their atmospheric boiling points, particularly for high-boiling-point solvents like ethylene glycols and glycerol. The distillation process takes place in a continuous column, which is classified into binary and multicomponent types. Binary distillation columns handle two components, while multicomponent distillation columns handle more than two.

The McCabe-Thiele diagram, a graphical tool for analyzing binary distillation, is employed to determine the theoretical number of stages necessary for the separation process. This method utilizes the equilibrium curve to specify the actual number of stages. Several assumptions are made, including constant molal overflow, implying that:

- i) The heats of vaporization for components are uniform.
- ii) The column operates adiabatically.
- iii) Changes in specific heat are negligible.

Given values include:

- Feed rate (F) of 4200 kg/hr (Saturated Liquid)
- Feed pressure of 8 bar
- Feed components: C3 - C5
- A maximum of 3% iC5 in the distillate product
- A maximum of 1% C4 in the bottom product.

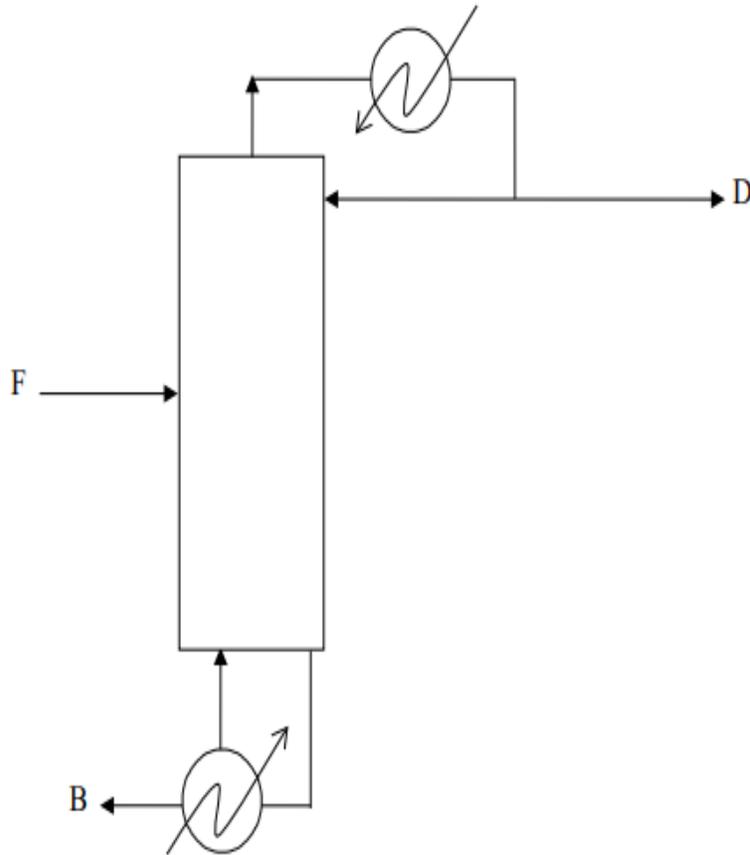
Steps needed to solve:

- 1) Determining the average relative volatility.
 - Step 1: identifying the given information.
 - Step 2: Calculating the Fractional Recovery of the Components.
- 2) Determine the minimum number of equilibrium stages using the Fenske equation.
- 3) Compare the separation specifications given to see whether they are satisfied by estimating the degree of separation of the non-key components using the Fenske equation.
- 4) Adjust the separation process using the Fenske equation to align with the specified requirements.
- 5) Calculate the minimum reflux using Underwood equation.
- 6) Calculate the actual number of stages using Gilliland correlation.
- 7) Find the compositions of the top and bottom products.
- 8) Evaluate the location of the feed tray.
- 9) Utilize the McCabe-Thiele method to ascertain the precise number of stages needed for a binary mixture.
- 10) Prove analytical procedure be improved to achieve better results.

Results:

Part A: Determining the average relative volatility.

Step 1: identifying the given information.



Component	Keys	Weight %
Propane C3	LNK	5
Iso-Butane iC4	LNK	15
n-Butane C4	LK	25
n-Pentane C5	HK	35
Iso-Pentane iC5	HNK	20

Assumptions:

- Non-Distribution of the non-keys components.
- Constant Molal Overflow
- The reflux ratio $R=1.5R_{min}$.
- specifying the LK and HK.
- Each stage considered as an equilibrium stage.
- $R=1.5 \cdot R_{min}$

Step 2: Calculating the Fractional Recovery of the Components (FR,i):

Why is the non-distribution assumption taken into consideration?

- In **simple distillation**, the process assumes ideal distribution (i.e., complete vapor-liquid equilibrium at each stage). However, it is not efficient for separating liquids with closely spaced boiling points.^[3]
- **Fractional distillation** is an improved method that accounts for non-ideal behavior. It involves using a fractionating column (packed or trayed) to enhance separation efficiency.

Using the mass balance since given the feed mass flow rate:

Table 1: mass balance equations

LNK	$zF = xD * D$
LK	$z_B F = x_{B,D} D + x_{B,B} B$
HK	$z_{IP} F = x_{IP,D} D + x_{IP,B} B$
HNK	$z_P F = x_B B$
Total balance	$F=B+D$

Table 2: Identifying the variables.

Component	MW	Wt%	Feed Kg/hr	Distillate Kg/hr	Bottom Kg/hr	$X_{i,D}$	$X_{i,B}$
Propane	44.10	5.00	210.00	210.00	0.00		0.00
Isobutane	58.10	15.00	630.00	630.00	0.00		0.00
Butane	58.10	25.00	1050.00	$1050-x$	x		0.01
Isopentane	72.10	20.00	840.00	y	$840-y$	0.03	
Pentane	72.10	35.00	1470.00	0.00	1470.00	0.00	

By solving *table 1* mass balances obtaining these values:

Table 3: Solving table 1 mass balances.

Component	MW	Wt%	Feed Kg/hr	Distillate Kg/hr	Bottom Kg/hr	X _{i,D}	X _{i,B}
Propane	44.10	5.00	210.00	210.00	0.00	0.11	0.00
Isobutane	58.10	15.00	630.00	630.00	0.00	0.33	0.00
Butane	58.10	25.00	1050.00	1027.25	22.75	0.53	0.01
Isopentane	72.10	20.00	840.00	57.75	782.25	0.03	0.34
Pentane	72.10	35.00	1470.00	0.00	1470.00	0.00	0.65
Total			4200	1925	2275	1	1

Part B: Determine the minimum number of equilibrium stages using the Fenske equation.

$$N_{min} = \frac{\ln \frac{\left(\frac{x_{LK}}{x_{HK}}\right)_{dist}}{\left(\frac{x_{LK}}{x_{HK}}\right)_{bots}}}{\ln \alpha_{LK, HK}} \quad (1)$$

$N_{min} = 9.04$.

Where:

LK: Butane.

HK: Isopentane.

Part C: Estimate the degree of separation of the non-key components using the Fenske equation and compare this with the separation specifications given to see whether they are satisfied.

Calculating the new distillate and the bottom flow rates using the following equations:

$$(D \cdot x_A) = (F \cdot Z_A) * (FR_A)_{dist} \quad (2)$$

$$(B \cdot x_A) = (F \cdot Z_A) * [1 - (FR_A)_{dist}] \quad (3)$$

The new composition for all species in the distillate and bottom were calculated using the following equation:

$$x_{i,D} = \frac{D_i}{D_T} \quad (4)$$

$$x_{i,B} = \frac{B_i}{B_T} \quad (5)$$

Note that all the calculation taken as two decimal places.

Solving for the bottom and distillate flow rate:

- considering for this calculation **propane**

$$(D \cdot x_A) = (F \cdot Z_{c3}) * (FR_{c3})_{dist}$$

$$(B \cdot x_{c3}) = (F \cdot Z_{c3}) * [1 - (FR_{c3})_{dist}]$$

$$(B \cdot x_{c3}) = 4.76 * (1-0.99) = 1.96 * 10^{-5} \text{ kmol/hr}$$

The composition of propane in the bottom and the distillate:

$$x_{i,D} = \frac{D_i}{D_T} = \frac{4.76}{34.086} = 0.139698$$

$$x_{i,B} = \frac{B_i}{B_T} = \frac{1.963606 \times 10^{-5}}{31.62943} = 6.2064 \times 10^{-7}$$

Using equation one, computing the new Nmin as shown in the table below

Table 4: New Nmin.

Mass basis	Component	Feed Kmol/hr	Z i (Weight %)	Xi,D
LNK	Propane (C3)	4.761904762	7.25%	13.97%
LNK	Isobutane (iC4)	10.84337349	16.50%	31.81%
LK	Butane (C4)	18.07228916	27.50%	51.87%
HK	Isopentane (iC5)	11.65048544	17.73%	2.35%
HNK	Pentane (C5)	20.38834951	31.02%	0.00%
Total	-	65.71640236	1	1

$$N_{min} = \frac{\ln \frac{\left(\frac{x_{LK}}{x_{HK}}\right)_{dist}}{\left(\frac{x_{LK}}{x_{HK}}\right)_{bots}}}{\ln \alpha_{LK, HK}} \quad \text{calculating } N_{min} \text{ using equation 1.}$$

$$N_{min} = \frac{\left[\frac{\left(\frac{0.52}{0.02349}\right)}{\left(\frac{0.01237}{0.343}\right)} \right]}{\ln 2.05} = 9.04 \text{ stages (including partial reboiler)}$$

Calculate the new fractional recovery:

$$(FR_C)_{dist} = \frac{\alpha_{c,HK}^{N_{min}}}{\frac{(FR_{HK})_{Bot}}{1-(FR_{HK})_{Bot}} + \alpha_{c,HK}^{N_{min}}} \quad (6)$$

$$(FR_C)_{Bot} = 1 - (FR_C)_{dist} \quad (7)$$

Taking an example:

Calculating the distillate and the bottom FR for **propane**:

$$(FR_C)_{dist} = \frac{5.36^{9.06}}{\frac{0.93}{1-0.93} + 5.36^{9.06}} = 0.999999722$$

The table below shows FR for all the components in the bottom and the distillate.

The new fractional recovery:

Table 5: The new fractional recovery

Component	FRD	FR B
Propane (C3)	0.999999722	2.775E-07
Isobutane (iC4)	0.997889859	0.0021101
Butane (C4)	0.978333333	0.0216667
Isopentane (iC5)	0.06875	0.93125
Pentane (C5)	0.01624848	0.9837515

Since pentane which is the $FR_{HNK} = 0.01624848$ then the non-distributing assumption is not valid, and second trial should be obtained

Repeating the previous calculation until reaching a valid conclusion of $FR_{old} = FR_{new}$

Second trial

Table 6:Second trial.

Mass basis	Component	Feed Kmole/hr	Z i (Weight %)
LNK	Propane (C3)	4.761904762	7.25%
LNK	Isobutane (iC4)	10.84337349	16.50%
LK	Butane (C4)	18.07228916	27.50%
HK	Isopentane (iC5)	11.65048544	17.73%
HNK	Pentane (C5)	20.38834951	31.02%
Total	-	65.71640236	1

$$N_{min} = 9.036486311437590$$

Fractional recovery table

Table 7:Fractional recovery table for second trial

Component	FRD	FR B
Propane (C3)	0.999997307	2.693E-06
Isobutane (iC4)	0.998591286	0.0014087
Butane (C4)	0.985440658	0.0145593
Isopentane (iC5)	0.099636463	0.9003635
Pentane (C5)	0.024160163	0.9758398

$$(FR_C)_{dist} = \frac{\alpha_{c,HK} N_{min}}{\frac{(FR_{HK})_{Bot}}{1 - (FR_{HK})_{Bot}} + \alpha_{c,HK} N_{min}} \quad (8)$$

$FR_{old} = FR_{new}$, thus the calculation will be proceed.

Part D: Revise the separation to meet the required specifications using Fenske equation.

- Change the table from mole percent to weight percent, rounding to two decimal places.

Table 8: Change from mole % to weight %

Component	MW	wt%	feed Kg/hr	Distillate Kg/hr	Bottom Kg/hr	Xi,D	Xi,B
Propane	44.1	5	210	210.00	0.00	0.11	0.00
Isobutane	58.1	15	630	628.67	1.33	0.32	0.00
Butane	58.1	25	1050	1027.25	22.75	0.53	0.01
Isopentane	72.1	20	840	57.75	782.25	0.03	0.35
Pentane	72.1	35	1470	23.77	1446.23	0.01	0.64
Total	61.85	100	4200	1947.44	2252.56	1.00	1.00

Conditions:

- o A maximum of 3 wt% iC5 in the distillate product.
- o A maximum of 1 wt% C4 in the bottom product.

The obtained results confirm that the separation process has met the specified requirements.

Part E: Calculate the minimum reflux using Underwood equation.

The calculations for ΔV_{feed} are shown in the table below, considering case B and assuming Φ to be 1.299730101. This assumption results in ΔV_{feed} being approximately zero due to the feed being saturated liquid.

$$V_{\min} = \sum_1^C \frac{\alpha_{i,\text{HK}}(Dx_{i,\text{dist}})}{\alpha_{i,\text{HK}} - \Phi} \quad (9)$$

$$V_{\min,\text{propane}} = \frac{5.3624793125207 \times 4.76188513130458 \times 0.139698097218442}{5.3624793125207 - 1.299730101}$$

$$V_{\min,\text{propane}} = 4.92$$

V_{\min} for other components and the final V_{\min} were calculated using excel as shown in table below:

Table 9:Final V_{\min} .

component	Average alpha(α_{avg})	Distillate (Kmol/hr)	$x_{i,D}$	Φ	V_{\min}
Propane	5.27	4.76	0.11	1.30	4.92
Isobutane	2.64	10.82	0.32	1.30	21.85
n-Butane	2.03	17.68	0.53	1.30	50.09
Isopentane	1.00	0.80	0.03	1.30	-3.43
n-Pentane	0.85	0.33	0.01	1.30	-0.79
Sum	11.79	34.40	1.00		72.64

$$N_{\min} = V_{\min} - D \quad (10)$$

$$L_{\min} = 38.24$$

$$R_{\min} = \frac{L_{\min}}{D} \quad (11)$$

$$R_{\min} = 1.11$$

Part F: Calculate the actual number of stages using Gilliland correlation.

$R = 1.5 \times R_{min}$ was taken into consideration, given that R varies between $(1.05 - 1.5) R_{min}$.

$$R = 1.5 \times 1.11 = 1.67$$

By Liddle equation:

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

$$x = 0.21$$

Since x is between 0.01 and 0.90, then:

$$\frac{N - N_{min}}{N + 1} = 0.545827 - 0.591422 \times x = \frac{0.02743}{x} \quad (12)$$

$$\frac{N - N_{min}}{N + 1} = 16.78$$

Substituting $N_{min} = 9.04$ and calculating for (N) gives the actual number of stages (N) equal to $16.78 \cong 17$ stages including partial reboiler.

Part G: Calculate the compositions of the top and bottom products.

Using Equations (4) and (5), the composition of the top and bottom is calculated, as shown in the following table:

Table 10: Composition of top and bottom.

Component	$X_{i,D}$	$X_{i,B}$
Propane	1.36E-01	4.22E-08
Isobutane	3.09E-01	7.31E-04
Butane	5.08E-01	1.25E-02
Isopentane	3.31E-02	3.46E-01
Pentane	1.41E-02	6.40E-01
Total	1.00	1.00

Part H: Estimate the location of the feed tray.

$$N_{F,\min} = \frac{\ln\left[\left(\frac{x_{LK}}{x_{HL}}\right)_{\text{dist}} / \left(\frac{z_{LK}}{z_{HK}}\right)\right]}{\ln(\alpha_{LK-HK})} \quad (13)$$

$$N_{F,\min} = \frac{\ln\left[\left(\frac{0.53}{0.03}\right)_{\text{dist}} / \left(\frac{0.25}{0.2}\right)\right]}{\ln(2.034)}$$

$$N_{F,\min} = 3.74$$

So, the actual feed stage can be estimated from equation (11):

$$\frac{N_{F,\min}}{N_{\min}} = \frac{N_F}{N} \quad (14)$$

$$\frac{3.74}{9.04} = \frac{N_F}{16.78}$$

Then, the feed is on the top of stage 6.94:

Part I: Determine the actual number of stages of a binary mixture using McCabe-Thiele method.

There are several steps to find the actual number of stages.

1- Drawing equilibrium line

- Data to find the equilibrium line has been taken from the literature.

2- Drawing feed line

- $x = y = Z_F = 0.512468$ (first point)
- The feed has been given as a saturated liquid, $q = 1$. Thus, a vertical straight line will be taken.

3- Drawing the top operating line

- $x = y = X_D = 0.96$ (first point)
- $y = \frac{R}{R+1}x + \frac{1}{R+1}x_D$ (second point) (15)

4- Drawing the bottom operating line

- $x = y = X_B = 0.0131010896$ (first point)
- The intersection between the top operating line and feed line. (second point)

5- Drawing the stages

- Starting Point: You start either from the top (x_D) or bottom (x_B) of the column.
- Switching between Operating Lines: As moving up or down the column, switching between the top operating line and the bottom operating line when you reach the point where the feed line intersects.
- Ending Point: end at either $x = x_D$ (started from the top) or $x = x_B$ (started from the bottom).
- Number of Stages (N): The total number of stages, including the partial reboiler, is 19.3.
- Feed Stage (NF): The feed stage is at stage 9 from the top.

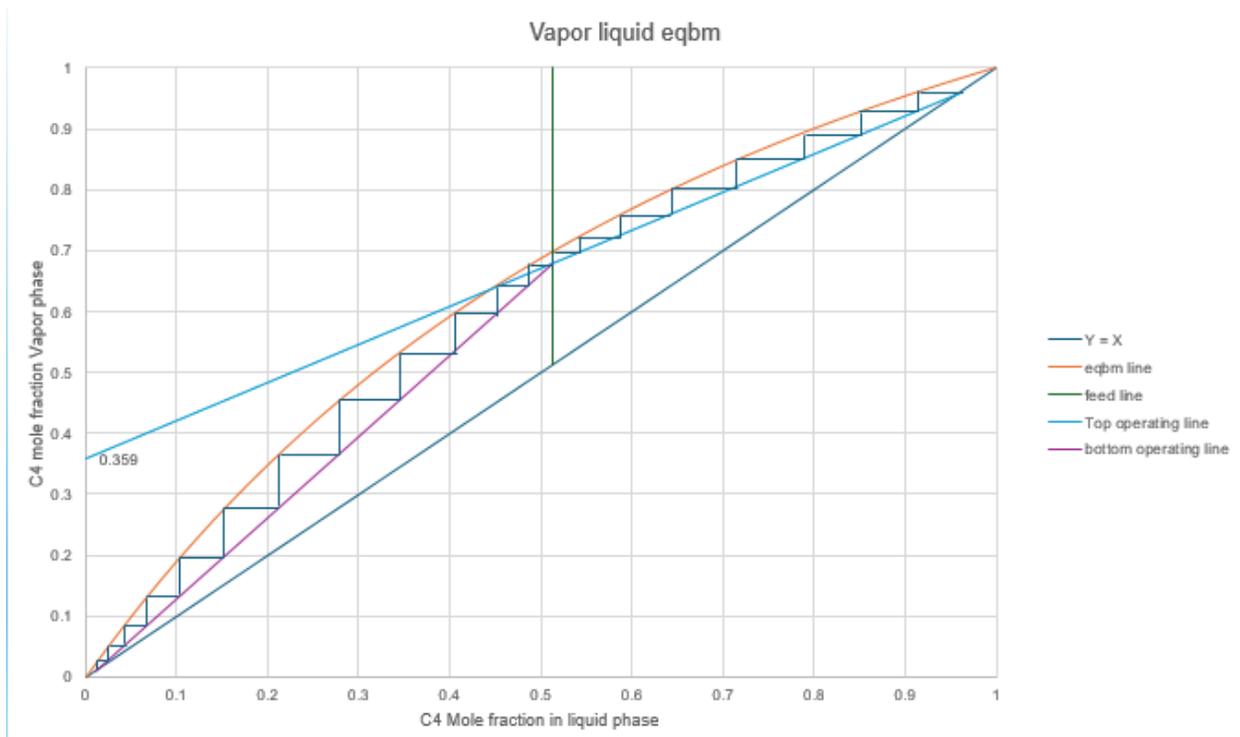


Figure 1: Calculate the number of stages.

- ❖ The actual number of stages is almost 19 stages.
- ❖ The theoretical number of stages is 17 stages.

The diagram below illustrates the solution achieved using the McCabe-Thiele method for the process: (Draw R_{min} Line) Second trial.

- First point: $x = y = x_D'' = 0.96$ where $(LK + LNK) = (0.11 + 0.32 + 0.53) = 0.96$
- Second point: using the top operating line equation to find it:

$$y = \frac{R_{min}}{R_{min}+1}x + \frac{1}{R_{min}+1}x_D \quad (16)$$

- Following the procedure of using pinch point to find R_{min} , followed by calculating R then draw the actual operating line, however, if the q-line which is between $y = x = x_D''$ and the intersection between the feed line and equilibrium line was beyond the equilibrium line, $R_{min}=1.11$.

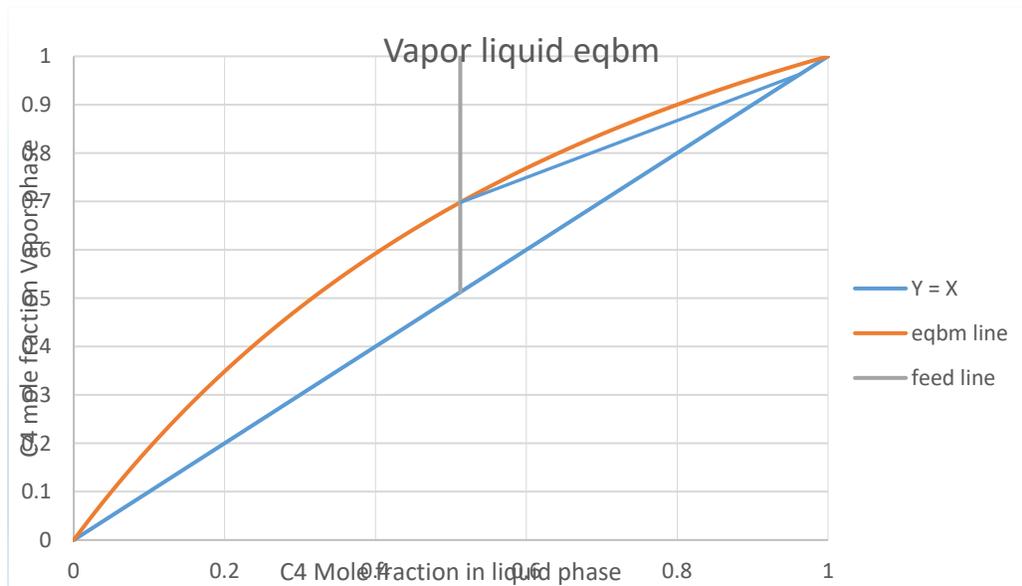


Figure 2: Calculating R_{min}

$$R_{min} = \frac{y_2 - y_1}{x_2 - x_1} = 1.386$$

$$R = R_{min} * 1.5 = 2.079$$

Why $R = 1.5$?

Higher reflux ratios generally result in higher product purities. This is because more liquid is returned to the column, providing additional opportunities for separation. However, there may be diminishing returns as the reflux ratio increases.^[4]

Therefore,

Finding the y intersection and bottom operating line with the new reflux ratios.

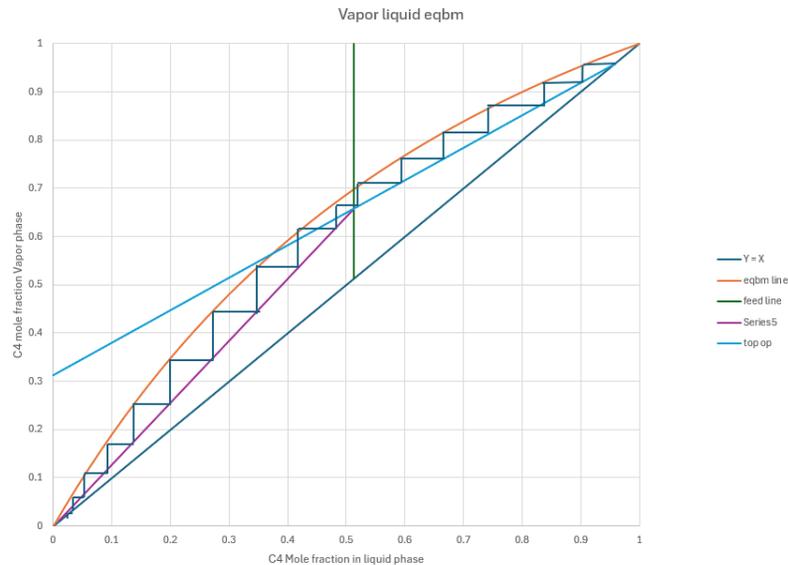


Figure 3: McCabe Thiele Method NO stages.

Part J: How can analytical procedure be improved to achieve better results.

- Relocate the feed stage to the middle of the column for better results.
- Use case (C) instead of case (B) to ensure more accurate solutions, particularly in calculating relative volatilities.
- Lower the temperature of the reboiler to prevent problems such as fouling.
- Maintain the column pressure above atmospheric pressure for optimal performance.

Discussion of results:

As shown in figure 3 the actual number of stages is 18 stages which is less than what has been shown previously in figure 1. That is because of the increase in reflux ratio in the second trial. Generally, increasing the reflux ratio in a distillation column tends to reduce the number of stages required for a given separation. This is because a higher reflux ratio provides more liquid for contact with the rising vapor, enhancing separation efficiency per stage. As a result, fewer stages are needed to achieve the same separation compared to a lower reflux ratio.

Conclusion:

The multicomponent mixture's results slightly deviate from those of a pseudo binary system due to the validity of such a system. In a pseudo binary system, the relative volatilities of two components remain unaffected by other non-key components. This is particularly applicable to hydrocarbon mixtures, which are nearly non-ideal and can be approximated as a pseudo binary system. Appendix 1 contains the data necessary for drawing the McCabe-Thiele diagram, which sums up these concepts and relationships.

Reference

[1] Szekely, L. C. G. (n.d.). Distillation. Distillation - an overview | ScienceDirect Topics.

<https://www.sciencedirect.com/topics/earth-and-planetary-sciences/distillation>.

[2] aspen plus, determining the relative volatilities to draw the equilibrium line.

[3] Alexander Sandtorv Distillation. - an overview | LibreTexts Chemistry.

https://chem.libretexts.org/Bookshelves/Organic_Chemistry/Book%3A_How_to_be_a_Successful_Organic_Chemist_%28Sandtorv%29/02%3A_COMMON_ORGANIC_CHEMISTRY_LABORATORY_TECHNIQUES/2.02%3A_Distillation

[4] Reshma Kadam optimum reflux ratio – How to calculate | Technologynews5

<https://technologynews5.com/distillation/optimum-reflux-ratio/>

Appendix 1

These values are taken from aspen plus.

liquid mole frac butane	vapor mole frac butane
0	0
0.02	0.0411501
0.04	0.0807119
0.06	0.118764
0.08	0.155381
0.1	0.190632
0.12	0.224582
0.14	0.257293
0.16	0.288823
0.18	0.319227
0.2	0.348557
0.22	0.376861
0.24	0.404185
0.26	0.430573
0.28	0.456066
0.3	0.480703
0.32	0.504521
0.34	0.527554
0.36	0.549837
0.38	0.5714
0.4	0.592273
0.42	0.612485

0.44	0.632062
0.46	0.65103
0.48	0.669414
0.5	0.687235
0.52	0.704518
0.54	0.721281
0.56	0.737546
0.58	0.753332
0.6	0.768655
0.62	0.783535
0.64	0.797986
0.66	0.812026
0.68	0.825669
0.7	0.838929
0.72	0.85182
0.74	0.864356
0.76	0.876549
0.78	0.88841
0.8	0.899952
0.82	0.911185
0.84	0.92212
0.86	0.932767
0.88	0.943136
0.9	0.953236
0.92	0.963076
0.94	0.972664
0.96	0.982009
0.98	0.991118
1	1