Continuous Distillation

Practical Chemical Engineering

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Story:

SwissBioFuel Co. wants to produce 90 wt% ethanol from fermentation process, and it was decided to use a plate distillation column for the purification of ethanol from an initial solution of 15 wt% ethanol/water. In order to maximize the efficiency of the process, the concentration of ethanol in the residue stream should not be higher than 5 wt%. SwissBioFuel has hired you to characterize and optimize a predesigned distillation column for the aforementioned ethanol separation.

OBJECTIVES

- Understand the basic concepts about binary mixture distillation.
- Use the McCabe-Thiele method for the distillation of a ethanol/water solution.
- Perform a detailed material and energy balance on a continuous distillation column.
- Combine theory and experimental results to characterize and optimize a distillation process.
- Compare plate distillation column and packed distillation column.

> Specifications should include: theoretical number of stages, overall efficiency of the trays, Murphree efficiencies, optimum feed stage, operational feed stage, minimum reflux ratio, optimum reflux ratio, operational reflux ratio and distillate to feed ratio.

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1. THEORETICAL BASIS

When a liquid mixture of volatile components boils, the composition of the vapor is not necessarily the same as the concentration of the liquid. This aspect can be leveraged to separate components of a mixture and is the basis of distillation. Distillation can be applied to solutions where all components are appreciably volatile, such as in ammonia-water or ethanol-water solutions, where both components will be in the vapor phase. In continuous distillation, a liquid mixture is continuously (without interruption) fed into the process and separated fractions are removed continuously as output streams.

The most widely used industrial applications of continuous, steady-state fractional distillation are in petroleum refineries, natural gas processing plants, petrochemical and chemical plants. In order to design a continuous distillation column with optimized operational conditions, it is imperative to understand basic vapor-liquid equilibrium relations.

1.1. *Vapor-Liquid Equilibrium Relations, T-x-y and VLE diagrams*

Often the vapor-liquid equilibrium relations for a binary mixture of A and B are given as a boiling-point diagram, also called a T-x-y diagram. **Figure 1** shows an example of a binary system, benzene (A) - toluene (B), at a total pressure of 101.32 kPa. The upper line is the saturated vapor line (*dew-point line*) and the lower line is the saturated liquid line (*bubble-point line*). The two-phase region is the area between the dew-point line and the bubble-point line. The mass fraction of the components in the liquid phase is represented by x and the mass fraction in the vapor phase is represented by y.

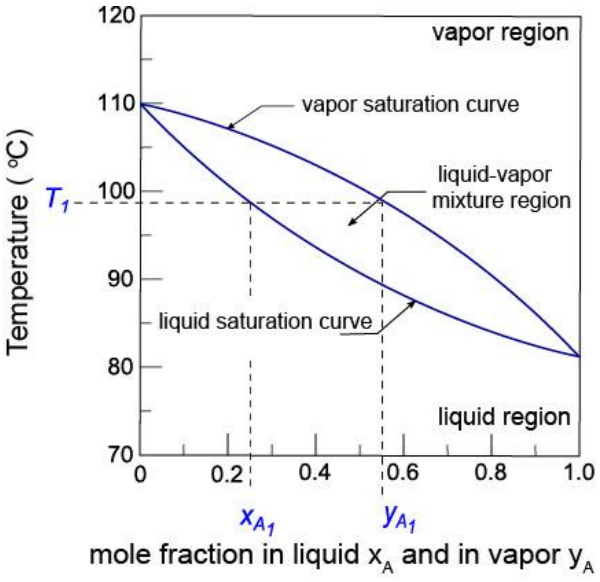


Figure 1. Boiling-point diagram for the system of benzene (*A*) - toluene (*B*) at a total pressure of 101.32 kPa

As presented in **Figure 1**, if a liquid mixture of x_{A1} = 0.25 is heated up to T_1 (98°C, 371.2 K) it will reach a liquid-vapor equilibrium, and the mixture will start to boil with a minimal increase in temperature. The composition of the first vapor in equilibrium will be given by the vapor saturation curve, which in this example is y_{A1} = 0.55. If the temperature continues to increase, the composition of x_A will decrease according to the liquid saturation curve and the composition of y_A will be lower according to the vapor saturation curve.

The boiling-point diagram in **Figure 1** is a typical ideal system following Raoult's law. Non-ideal systems differ considerably. Given that the benzene-toluene system follows Raoult's law, the boiling-point diagram can be calculated from the pure vapor-pressure data and the following equations:

$$p_A + p_B = P \qquad \text{(Eq. 1)}$$

$$P_A x_A + P_B(1 - x_A) = P \qquad \text{(Eq. 2)}$$

$$y_A = \frac{p_A}{P} = \frac{P_A x_A}{P} \qquad \text{(Eq. 3)}$$

Where p_i is the partial pressure of component *i* in the gas phase mixture, and P_i is the vapor pressure of pure *i*. **Figure 2** shows the equilibrium data of the benzene-toluene system where y_A is plotted versus x_A. The 45° line is given as reference, $y_A = x_A$. This type of diagram is called VLE (Vapor-Liquid Equilibrium)

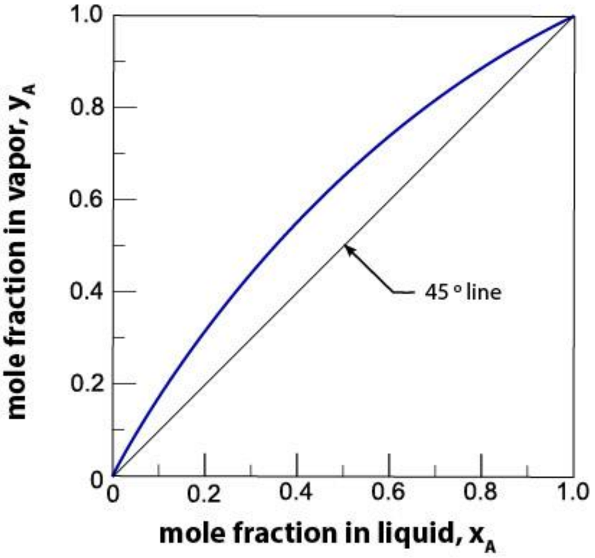


Figure 2. VLE diagram of the benzene (A)-toluene (B) system at a total pressure of 101.32 kPa

1.2. *Single-Stage Equilibrium Contact for Vapor-Liquid System*

Based on **Figure 2** if a mixture of benzene and toluene reaches its boiling point, the produced vapor would be abundant in benzene but it will not be pure benzene. The complete separation of the two components is achieved by employing multiple boiling and condensing stages. In a single equilibrium stage two different phases (liquid and vapor) are in counter-current contact during a certain time, called contact time. During the contact time the phases are mixed and the solution components diffuse within both phases creating an equilibrium in composition. It is important that the contact time between the phases is sufficiently long to reach the equilibrium. **Figure 3** shows an example of a single equilibrium stage where L_0 and V_2, of known amount and composition, are the inlet streams to the stage and L_1 and V_1 are the outlet stream of the stage. Total mass balance is described as:

$$L_0 + V_2 = L_1 + V_1 = M \qquad \text{(Eq. 4)}$$

where L is the liquid stream in kg, V is the vapor phase in kg and M is the total mass

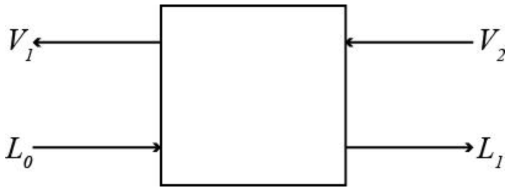


Figure 3. Representation of a single equilibrium stage

For a system where three components (A, B and C) are present in the streams, the total mass balance is described as:

$$L_0 x_{A0} + V_2 y_{A2} = L_1 x_{A1} + V_1 y_{A1} = M x_{AM} \quad \text{(Eq. 5)}$$

$$L_0 x_{C0} + V_2 y_{C2} = L_1 x_{C1} + V_1 y_{C1} = M x_{CM} \quad \text{(Eq. 6)}$$

$$x_A + x_B + x_C = y_A + y_B + y_C = 1 \quad \text{(Eq. 7)}$$

where the mass fraction of A in the M stream is defined as x_{AM}

1.3. *Multiple-Stage Equilibrium Contact for Vapor-Liquid System*

Continuous distillation or fractionation is a multistage operation at countercurrent stream flows. In each of the stages the vapor stream V and the liquid stream L mix and reach an equilibrium before leaving the stage. A representation of a general countercurrent multistage operation is shown in **Figure 4**.

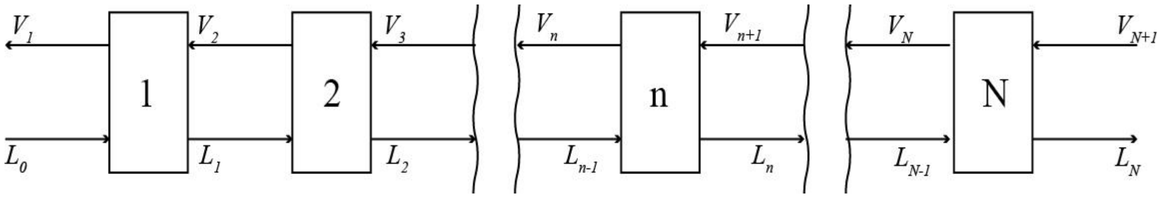


Figure 4. Countercurrent cascade operation of idea stages.

The general mass balance of the system for the component A in **Figure 4** is described as follows:

$$V_{n+1} y_{n+1} + L_0 x_0 = L_n x_n + V_1 y_1 \quad \text{(Eq. 8)}$$

where n is the nth stage in the multistage operation

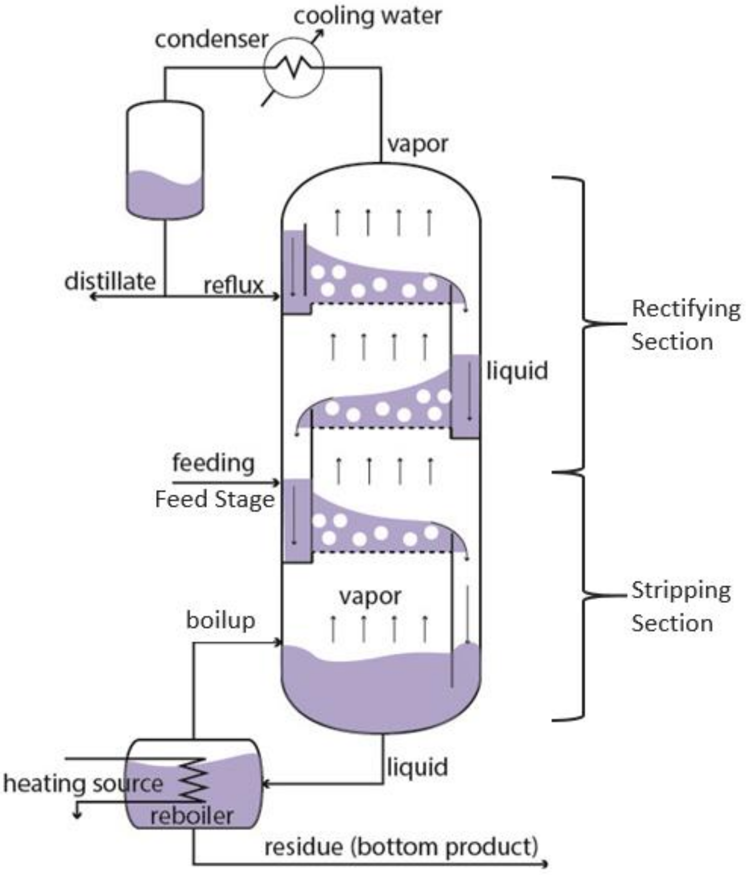


Figure 5. Process flow of a continuous distillation tower containing sieve trays.

In a distillation tower, the stages (referred to as sieve plates or trays) are arranged vertically, as shown schematically in **Figure 5.** The *feed (F)*, which contain a more volatile component – the *light key*, *LK* – and a less volatile component – the *heavy key, HK* – enters the column at a certain stage called *feed stage (f)*. The sections below and above the *feed stage* are called the *stripping section* and the *rectifying section*, respectively. The *stripping section* has a partial reboiler, which can be considered as a stage, at the bottom of the column where the liquid mixture is partially vaporized and split into two streams: the *boilup (B)* and the *residue (W)*. The *boilup* is recycled back to the distillation column at the bottom stage with a high concentration of the *heavy key*. The *residue* is considered bottom product or waste. At the top of the *rectifying section* the vapor mixture is condensed by a total condenser and it is split into two streams. These streams are the *distillate (D),* with high purity of the *light key,* and the *reflux (L)*, which is recycled back to the distillation column at the top stage. An overall material balance around the entire column in **Figure 5** states that the *feed* [mol/h] must be equal to the sum of the *distillate* [mol/h] and the *residue* [mol/h].

$$F = D + W \tag{Eq. 9}$$

At the *feed stage*, the feed might be liquid, vapor or a mixture of liquid and vapor. When the feed is liquid, the mixture flows down through the stages against the up-flow of the *boilup* at the *stripping section.* In the case of vaporous feed, the solution bubbles through the stages against the down-flow of the *reflux* at the *rectifying section.* Inside the tower, the liquids and vapors are always at their bubble points and dew points, respectively, so the highest temperature is at the bottom and the lowest is at the top. As both streams equilibrate at the stages, the *heavy key* is removed from the vapor by the falling liquid and the liquid is stripped of the *light key* by the rising vapor.

1.4. *Continuous Distillation: Material balance, Constant Molal Overflow, Number of Ideal Plates and McCabe-Thiele Method*

1.4.1. Introduction

McCabe and Thiele developed an approximate graphical method to estimate the number of equilibrium stages and the amount of reflux required for a desired degree of separation. This method combines the equilibrium-distribution curve for a binary system (VLE curve) and the operating-line curves of the rectifying and stripping sections of the column.

The operating lines are defined by simplified mass balance equations in each of the column section (rectifying and stripping), using the approximation of *constant molal overflow.* This approximation assumes constant molar enthalpies and negligible heat of mixing, heat loss within the stage and pressure drop through the column. In case of constant pressure along the column, the *constant molal overflow* could be applied if the heat balances in each of the trays (using the temperature in each tray (T_n)) show that the sensible heat differences in the four streams are negligible and only the molar latent heats between streams are significant. Given that the latent heats between chemically similar streams are comparable, for every mole of vapor or A condensed; 1 mole of liquid or B is vaporized, i.e. $V_{n+1} = V_n$ and $L_n = L_{n-1}$. Following the representation of a tray in **Figure 6**, a total mass balance gives:

$$V_{n+1} + L_{n-1} = V_n + L_n \qquad \text{(Eq. 10)}$$

and a component balance gives:

$$V_{n+1}y_{n+1} + L_{n-1}x_{n-1} = V_n y_n + L_n x_n \qquad \text{(Eq. 11)}$$

where n is the tray number, V_{n+1} is mol/h of vapor leaving the tray $n+1$, V_n is mol/h of vapor leaving the tray n, L_{n-1} is mol/h of liquid leaving the tray $n-1$, L_n is mol/h of liquid leaving the tray n, y is the mole fraction of A in the vapor streams and x is the mole fraction of A in the liquid streams.

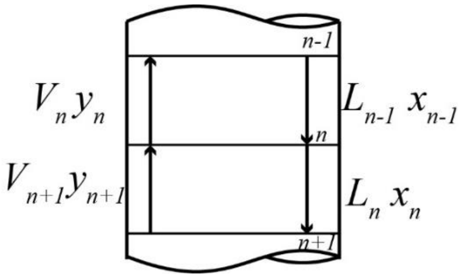


Figure 6. Vapor and liquid streams entering and leaving a tray.

1.4.2. Rectifying Section Equations

As shown in **Figure 5**, the rectifying section covers from the top stage (including the total condenser) to just above the *feed stage.* A material balance for the *light key* over the *rectifying section* (dashed-lines) shown in **Figure 7a,** is as following:

$$V_{n+1}y_{n+1} = L_n x_n + Dx_D \qquad \text{(Eq. 12)}$$

$$y_{n+1} = \frac{L_n}{V_{n+1}}x_n + \frac{Dx_D}{V_{n+1}} \qquad \text{(Eq. 13)}$$

Since $y_1 = x_D = x_0$ and the *constant molal overflow* is assumed, $L_n = L = constant$ and $V_{n+1} = V = constant$, then Eq. 14 could be rewritten as:

$$y_{n+1} = \frac{L}{V} x_n + \frac{D}{V} x_D \qquad \textbf{(Eq. 14)}$$

Eq. 14 is the operation line of the *rectifying section* of the distillation column.

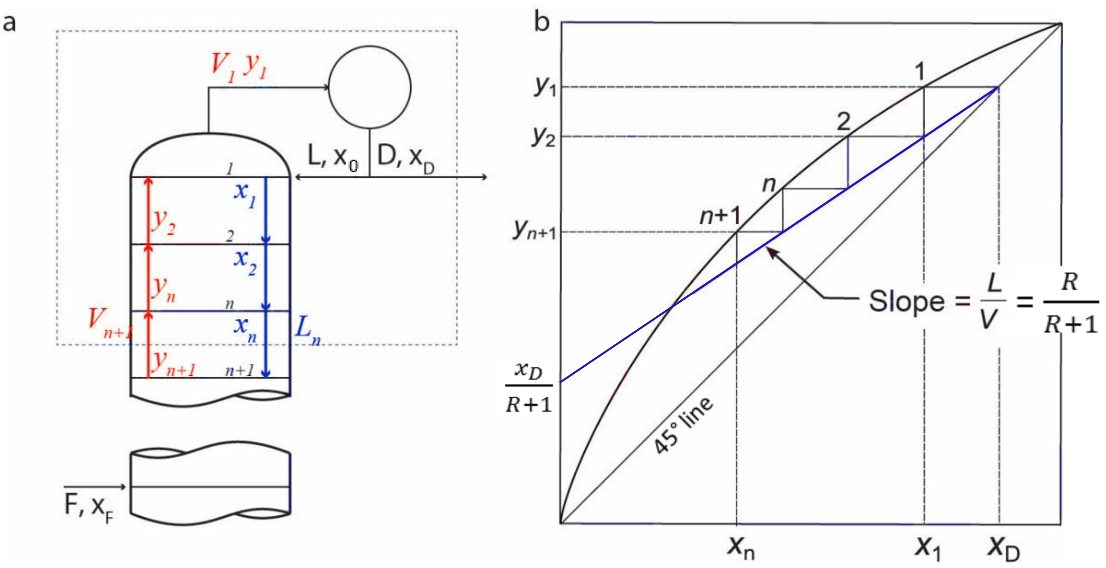


Figure 7. (a) Schematic representation of the rectifying section of a tower. (b) McCabe-Thiele operating line for the rectifying section (blue), the VLE curve and the 45°line (black).

The liquid entering at the stop stage is the external *reflux* rate and its ratio to the *distillate* rate, $^{\boldsymbol{L}}/_{\boldsymbol{D}}$, is called the *reflux ratio, R.* Since $\boldsymbol{V = L + D}$ and $\boldsymbol{R} = {}^{\boldsymbol{L}}/_{\boldsymbol{D}}$, then Eq. 14 can be written as:

$$y_{n+1} = \frac{R}{R+1} x_n + \frac{1}{R+1} x_D \qquad \text{(Eq. 15)}$$

If values of R and x_D are specified, Eq. 15 plots as a straight line on the xy diagram with intersection at $y_1 = x_D$ on the 45° line, slope$= L/V = R/(R+1)$, and intersection at $y = x_D/(R+1)$ for $x = 0$ as shown in **Figure 7b**.

1.4.3. Stripping Section Equations

As shown in **Figure 8a**, the stripping section convers from the *feed stage* to the bottom stage including the partial reboiler. A material balance for the *heavy key* over the stripping section (dashed-line) shown in **Figure 8a,** is as follows:

$$V_{m+1}y_{m+1} = L_m x_m - W x_w \qquad \text{(Eq. 16)}$$

$$y_{m+1} = \frac{L_m}{V_{m+1}} x_m - \frac{W}{V_{m+1}} x_w \qquad \text{(Eq. 17)}$$

where L_m and V_{m+1} are the total molar flows in the stripping sections, which by the *constant molal overflow* assumption, remain constant stage to stage ($L_m = L_N = L_{st}$ and $V_{m+1} = B = V_{st}$). Therefore, Eq. 17 could be rewritten as:

$$y_{m+1} = \frac{L_{st}}{V_{st}} x_m - \frac{W}{V_{st}} x_w \qquad \text{(Eq. 18)}$$

the subscript *st* is used as reference for the stripping section.

The vapor leaving the partial reboiler, *boilup*, is assumed to be in equilibrium with the liquid *residue* products. Thus, the partial reboiler acts as an extra equilibrium stage. The ratio between the *boilup* rate and the *residue* rate, $^{B}/_{W}$, is the *boilup ratio,* V_B. Since $L_{st} = V_{st} + W$, $B = V_{st}$ and $V_B = {}^{B}/_{W}$, then Eq. 18 could be rewritten as:

$$y_{m+1} = \frac{V_B+1}{V_B} x_m - \frac{1}{V_B} x_w \qquad \text{(Eq. 19)}$$

If values of V_B and x_W are known, Eq. 19 could be plotted as a straight line on the xy diagram with intersection at $y = x_W$ on the 45° line, slope $= L/V = (V_B + 1)/V_B$ and intercept at $y = -x_W/V_B$ for $x = 0$ as shown **Fig. 8b**.

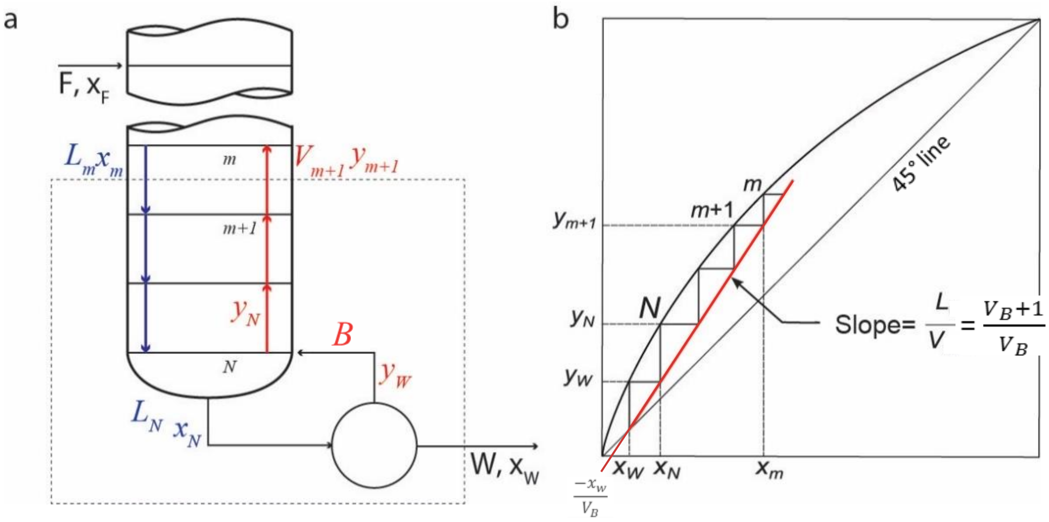


Figure 8. (a) Schematic representation of the stripping section of a tower. (b) McCabe-Thiele operating line for the stripping section (red), the VLE curve and the 45°line (black).

The theoretical stages are determined by drawing the stairlike construction $TQBNC\ldots S$ and counting the number of steps, seen in **Figure 9**. In a VLE curve with McCabe-Thiele operating lines, a theoretical stage is drawn starting at x_D or x_W, then it intercepts with the VLE curve (y_1 or y_W) and consequently with the operation line (x_1 or x_N). Subsequence stages are drawn following the same order.

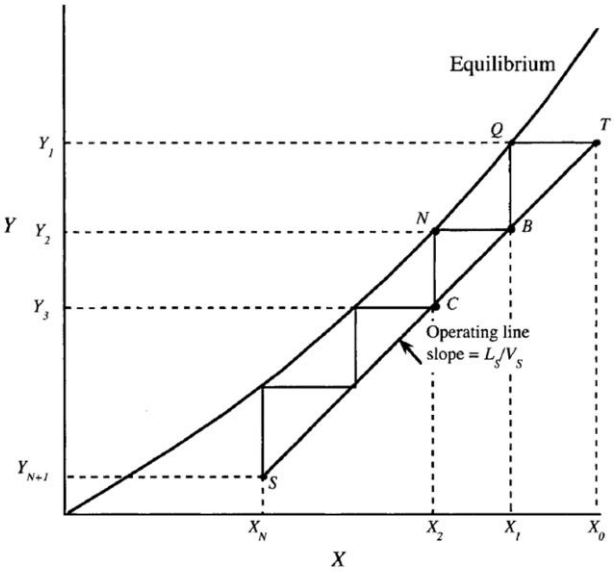


Figure 9. The stairlike construction TQBNC...S of theoretical stages.

1.4.4. Feed stage

Given that the *feed*, *F*, may consist of liquid, vapor or a mixture of both, the quantities of the vapor and liquid streams inside the column change dramatically at the *feed stage, f*. If the *feed* is a saturated liquid, L_{st} will exceed L. If for the contrary the *feed* is a saturated vapor, V will exceed V_{st}. The following equations are the material and energy balances around the *feed stage* to establish a general relation between streams:

$$F + L + V_{st} = V + L_{st} \qquad \text{(Eq. 20)}$$

$$FH_F + LH_{L,f-1} + V_{st}H_{V,f+1} = VH_{V,f} + L_{st}H_{L,f} \qquad \text{(Eq. 21)}$$

The vapor and liquid streams inside the tower are saturated, and the molal enthalpies of the saturated vapors and liquids at this section are identical because the differences in temperature and composition between trays are negligible. Therefore, $H_{V,f+1} = H_{V,f}$ and $H_{L,f-1} = H_{L,f}$. Eq. 21 then becomes:

$$(L_{st} - L)H_{L,f} = (V_{st} - V)H_{V,f} + FH_F \qquad \text{(Eq. 22)}$$

By combining Eq. 20, the condition of the *feed* could be described using the term *quantity, q*.

$$\frac{L_{st}-L}{F} = \frac{H_{V,f}-H_F}{H_{V,f}-H_{L,f}} = q \qquad \text{(Eq. 23)}$$

The *quantity* is defined as the energy required to convert 1 mol of *feed* from its condition H_F to a saturated vapor, divided by the molal latent heat of evaporation $(H_{V,f} - H_{L,f})$. There are five different thermal conditions in which the *feed* could be introduced to the tower. For each of these conditions, the value of q will be different. The **Table 1** shows values of q according to the *feed* condition.

Feed Condition	**q-value**
Subcooled liquid	> 1
Bubble-point (saturated) liquid	1
Partially vaporized	$L_F/F = 1 - V_F/F$
Dew-point (saturated) vapor	0
Superheated vapor	< 0

Table 1. Quantity values according to the feed condition.

Quantity values provide a convenient method to determine V_{st} by combining Eq. 20 and Eq. 23:

$$V_{st} = V + F(q+1) \qquad \text{(Eq. 24)}$$

and the q-line equation. The q-line equation represents the locus of the intersection of the two operating lines, is a straight line on the xy diagram of slope $= q/(q-1)$ and it passes through the point $x = y = z_F$ on the 45°line, where x_F is the overall composition of the *feed* (see **Figure 10**). This equation could be found as following:

- Subtracting the rectifying and stripping operational lines (Eq. 14 and Eq. 18):

$$y(V - V_{st}) = (L - L_{st})x + Dx_D + Wx_w \qquad \text{(Eq. 25)}$$

- Combining an overall material balance for the component A, Eq. 26, and the Eq. 23:

$$Fz_F = Dx_D + Wx_w \qquad \text{(Eq. 26)}$$

$$y = \frac{q}{q-1}x - \frac{1}{q-1}z_F \qquad \text{(Eq. 27)}$$

Figure 10 shows a graphical interpretation of the q-line for the five *feed* conditions discussed in **Table 1**. After drawing the q-line and the rectifying-section operating line, the stripping-section operating line is located by drawing a straight line from the point $x = y = x_W$ on the 45°line to the intercept of the q-line and the rectifying-section operating line. The intercept must lie somewhere between the VLE curve and the 45°line. This analysis shows that for a given *feed* condition and a *reflux ratio*, the liquid/vapor ratio in the stripping section and the reboiler heat load is automatically established.

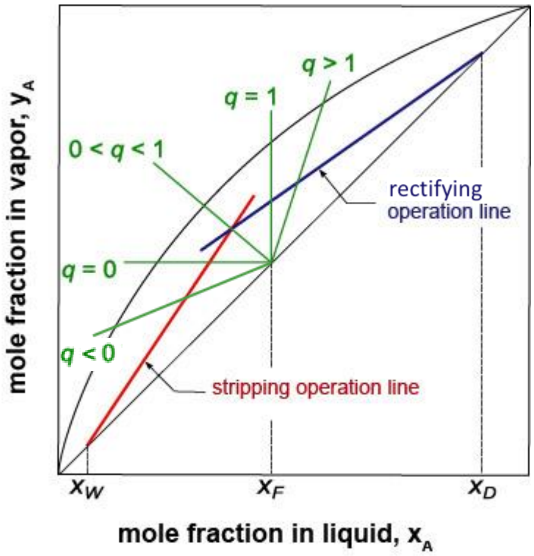


Figure 10. Location of the *q*-line for typical feed conditions.

1.4.5. Number of Equilibrium Stages and Feed-Stage Location.

For a given *feed* condition, x_F and the q-line are fixed. For particular *distillate* and *residue* products, x_D and x_W are fixed. With a specification of the *reflux ratio*, the location of the rectifying and stripping line are fixed. The q-line simplifies the graphical location of the stripping-section line, but the intercept of the two operating lines does not necessarily determine the separation between the stripping and rectifying section of the column. Rather, it is the introduction of the *feed* which governs the change from one operating line to the other. The intercept of the operating lines shows the optimal location where the *feed* should be introduced into the column to achieve the least number of ideal stages.

Following the example in **Figure 11**, if the *feed* is introduced at the fourth stage, the rectifying line is used to draw the stages 1 through 3, and beginning with stage 4, the stripping line must be used. The total number of ideal stages required in this example is approximately 6.5, including the partial reboiler.

The number of theoretical trays in a tower should not include the reboiler stage.

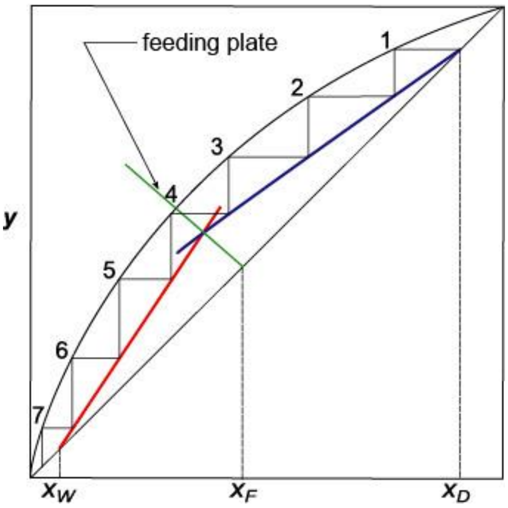


Figure 11. Optimum location of feed stage with the least number of ideal stages.

Figure 12 shows three examples where the *feed stage* location is at the optimal stage, below or above. In the design of a new column, the optimum location is used for the introduction of the *feed*.

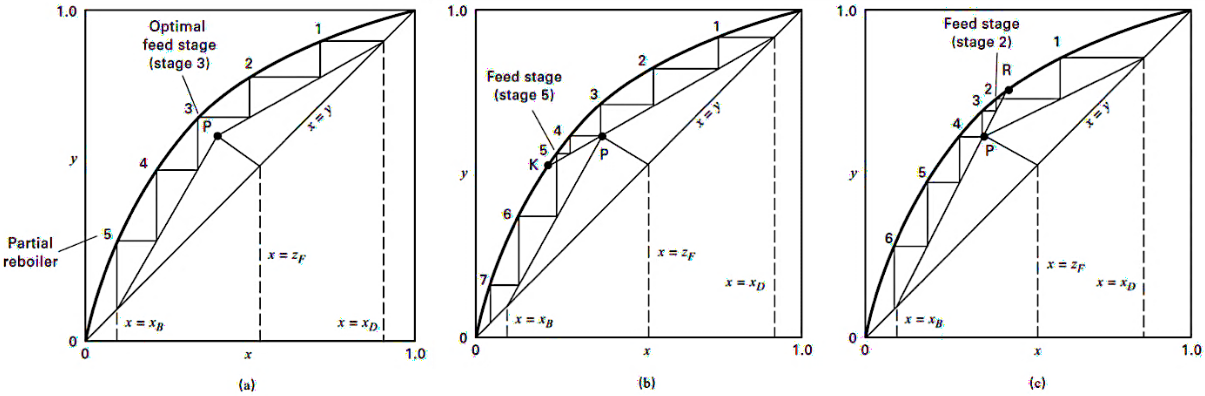


Figure 12. Optimal and non-optimal locations of feed stage: (a) optimal feed-stage location; (b) feed-stage location below optimal stage; (c) feed-stage location above optimal stage.

1.4.6. Reflux limiting conditions

The *reflux ratio* could be specified between a minimum, R_{min}, to an infinite value (*total reflux*). The R_{min} corresponds to an infinite number of stages, **Figure 13-a**, while a *total reflux* corresponds to the minimum number of stages, **Figure 13-b**. As the *reflux ratio* increases, both operating lines move toward the 45° line until they coincide and the *total reflux* limiting condition is reached. At this condition, all the overhead vapor is condensed and returned to the top stage. Thus, no distillate is withdrawn. When the *reflux ratio* decreases from the limiting case of *total reflux*, the interception between the operating lines and the q-line moves from the 45° line towards the equilibrium curve. When the interception reaches the equilibrium line, the R_{min} limiting condition occurs. This interception is called the *pinch point*, P, because the operating lines pinch the equilibrium line.

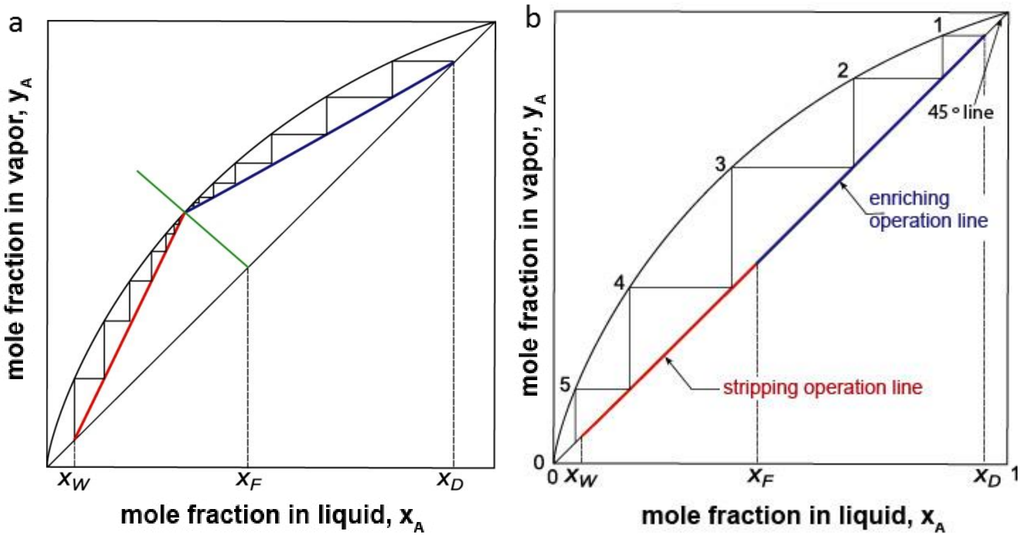


Figure 13. a) Minimum reflux ratio and infinite number of stages by McCabe-Thiele method. b) Total reflux and minimum number of stages by McCabe-Thiele method.

In case of a nearly ideal binary solution in **Figure 14-a**, the *pinch point* is at the *feed stage*. To reach that stage from the rectifying or stripping section, an infinite number of stage is required. For a highly non-ideal binary system, the *pinch point* can occur above or below the *feed stage*. In the case illustrated in **Figure 14-b**, the rectifying operating line intersects the equilibrium curve above the *feed stage* where an infinite number of stages exists.

The slope of the rectifying operating line cannot be reduced further because the line would cross the equilibrium curve and violate the second law of thermodynamics. In other words, a spontaneous mass transfer would be required from a region of low concentration to a region of high concentration which is impossible in a binary system. This is analogous to a second law violation by a temperature crossover in a heat exchanger. Therefore, the column cannot operate at minimum reflux with the given system.

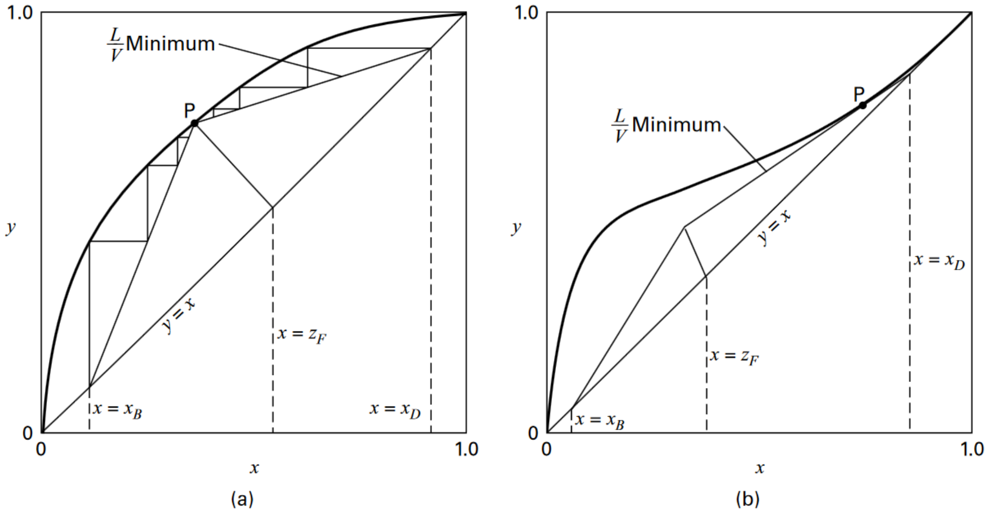


Figure 14. Construction for minimum reflux at infinite stages: (a) near-ideal system, P at the feed stage; (b) highly non-ideal system, P above the feed stage.

1.4.1. Optimum Reflux Ratio

Any *reflux ratio* between the limiting conditions will provide the desired separation specifications. However, for new designs, the optimum *reflux ratio* should be used to achieve the least total cost of operation. At R_{min} the column requires an infinite number of stages and, consequently, the fixed cost is infinite, but the operating costs are the lowest possible. As R increases, the number of trays decreases, but the column diameter increases due to larger quantities of recycled liquid and vapor per unit quantity of *feed*. The condenser, reboiler and reflux pump must also be larger. The fixed costs reach a minimum and rise to infinity again at *total reflux* and the operating costs increase directly with the *reflux ratio*, as shown in **Figure 15**. The total cost, the sum of the fixed and operating costs, must pass through a minimum at the optimum *reflux ratio*. The accepted range of optimum *reflux ratio* is from $1.2R_{min}$ to $1.5R_{min}$, with the lower value applying to a difficult separation (e.g., $\alpha = 1.2$). However, because the optimum *reflux ratio* is broad, columns are often designed for *reflux ratios* greater than the optimum.

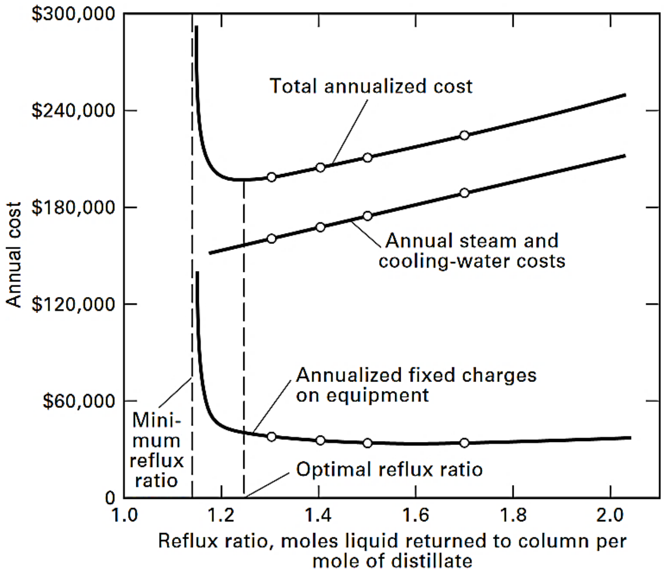


Figure 15. Optimum reflux ratio for a representative distillation process.

1.5. *Plate Efficiencies*

To convert ideal plates to real plates, the plate efficiency must be known. There are three kinds of plate efficiency: (1) *overall efficiency*, which concern the entire column; (2) *Murphree efficiency*, which has to do with a single plate; and (3) *local efficiency*, which pertains to a specific location on a single plate. The *overall efficiency*, E_o, it is defined as the ratio of the number of ideal plates needed in an entire column to the number of real plates, see Equation 28.

$$E_o = \frac{Number\ of\ theoretical\ stages}{Number\ of\ real\ stages} \qquad \text{(Eq. 28)}$$

The *Murphree efficiency*, η_M, is defined by:

$$\eta_M = \frac{x_n - x_{n-1}}{x_n^* - x_{n-1}} \qquad \text{(Eq. 29)}$$

where x_n : actual (real) concentration of liquid leaving plate n

x_{n-1} : actual (real) concentration of liquid entering plate n

x_n^* : theoretical concentration of liquid in equilibrium with the leaving vapor from plate n

The *Murphree efficiency* is therefore the change in liquid composition of the previous plate to the plate divided by the change that would have occurred if the liquid leaving were in equilibrium with the leaving vapor. The *Murphree efficiency* is defined in terms of vapor concentrations, but the measured efficiencies are rarely based on vapor phase analysis due to unreliable sampling. Instead, liquid samples are taken on each plate, and the vapor compositions are determined from a McCabe-Thiele diagram. Columns that operated at high velocities will have significant entrainment, and this reduces the plate efficiency, given that the drops of entrained liquid are less rich in the *light-key* than the vapor.

The *local efficiency, η',* is defined by:

$$\eta' = \frac{x'_n - x'_{n-1}}{x'_{en} - x'_{n-1}} \quad \text{(Eq. 30)}$$

where x'_n : concentration of liquid leaving a specific location on plate n

x'_{n-1} : concentration of liquid entering plate n at the same location

x'_{en} : concentration of liquid in equilibrium with vapor at the same location on plate n

1.5.1. Relation between Murphree and overall efficiencies

The *overall efficiency* is not the same as the average *Murphree efficiency* of the individual plates. These values could be very different specifically for stripping columns and at the top of the rectifying section. The relation between these efficiencies depends on the relatives slopes of the equilibrium line and the operating line. For columns with both sections, stripping and rectifying, the *overall efficiency* may be relatively close to the average value of *Murphree efficiency*. This is due to that the values in the stripping section, where E_o is greater than η_M, tends to compensate the values in the rectifying section, where E_o is smaller than η_M. When analyzing the performance of a real column, the correct value of η_M should be determined rather than just determining E_o and assume $E_o = \eta_M$. In cases where the equilibrium and operating lines are straight, the following equation can be applied:

$$E_o = \frac{Ln\left(1+\eta_M\left(m\cdot\frac{V}{L}-1\right)\right)}{Ln\left(m\cdot\frac{V}{L}\right)} \qquad \text{(Eq. 31)}$$

where m is the slope of the VLE line. Note that when $m\cdot\frac{V}{L} = 1.0$ or when $\eta_M \approx 1.0$, $E_o = \eta_M$.

1.5.2. Relation between Murphree and local efficiencies

The *local efficiency* and the *Murphree efficiency* are equal in small columns where the liquid is sufficiently agitated by the vapor flow and no measurable concentration gradient in the liquid is found. Given that the concentration of the liquid leaving the plate is the same on the entire plate, Equation 30 will become the same as Equation 29. The change in concentration between x_n and x_{n+1} occurs right at the downcomer exit when the liquid on plate *n+1* and the downcomer liquid are vigorously mixed. In larger columns, liquid mixing in the direction of flow is not complete and a concentration gradient does exist in the liquid on the plate.

1.5.3. Use of Murphree efficiency

When Murphree efficiency is known, it can readily be used in the McCabe-Thiele diagram to compare theoretical plates to real plates, see **Figure 16**. Triangle *acd* represents an ideal plate and triangle *abe* the real plate. The actual plate, instead of enriching the vapor from y_{n+1} to y_n^*, shown by the segment *ac*, reached less enrichment $y_n - y_{n+1}$, shown by the segment *ab*. In order to apply the known *Murphree efficiency*, the VLE curve should be replaced by the *pseudo-equilibrium line* by the following equation:

$$y_{pseudo} = x + \eta_M(y^*(x) - x) \qquad \text{(Eq. 32)}$$

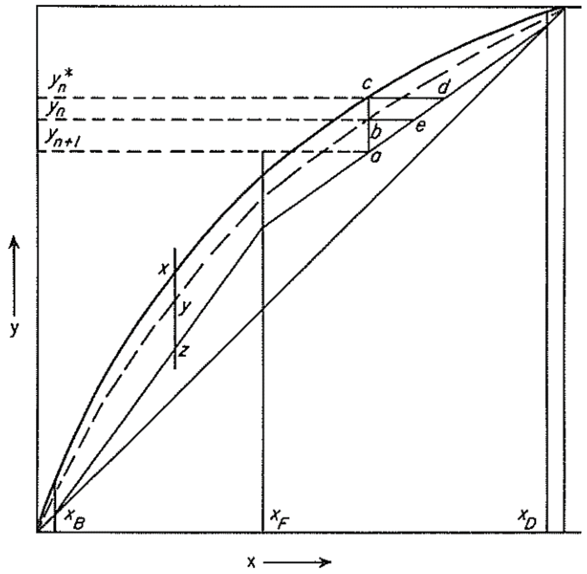


Figure 16. Use of Murphree efficiency on *xy* diagram. Dashed line is the pseudo-equilibrium curve for $\eta_M = 0.60$.

Once the *pseudo-equilibrium curve* has been created, the usual stairlike construction is made and the number of actual plates is determined. The reboiler is not subject to a discount for plate efficiency, and the VLE curve is used for the last step in the stripping section.

2. EXPERIMENTAL DETAILS

2.1. *Equipment Setup*

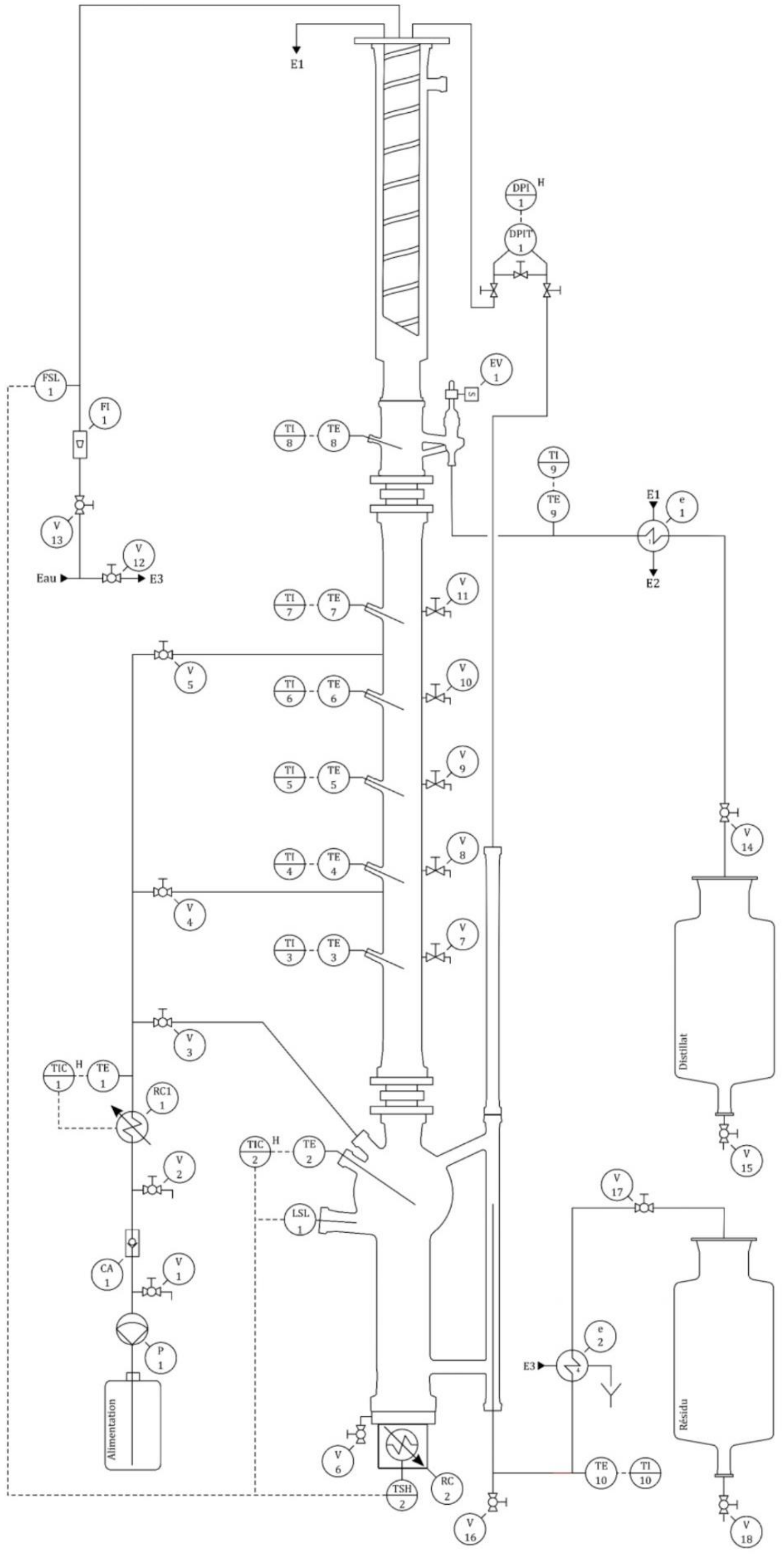


Figure 17. Scheme of the distillation unit.

2.2. ***Warnings***

1. Before starting the distillation, make sure the cooling water circulates in the condenser, with adequate flow (200 L/h minimum on flow meter FI1).
2. Make sure the valve of the feed plate or boiler is open before turning on pump P1.
3. To collect the distillate, ensure there is a steady liquid flow in the goose neck before activating solenoid valve EV1. Activating EV1 while the goose neck is dry may cause the glass to break.
4. Regularly check that the pressure drop DPIT1 is stable. If it increases excessively, indicating column flooding (liquid accumulation in the column), reduce the reboiler heat duty.
5. At the end of the day, ensure that the heaters, pump, cooling water, and interface box are turned off. Before leaving, empty the unit and keep valves V6, V15, V16 and V18 open.

3. EXPERIMENTAL PLAN

3.1. *Software*

a) Click TIC1 (resp. TIC2) to adjust the heat duty or setpoint temperature of the feed heater RC1 (resp. reboiler RC2).
b) Click TIMER to modify the reflux ratio.
c) Insert a USB drive and click EXPORT to save the temperature data and pressure drop at the end of the experiment.

3.2. *Recommended experiments*

a) Batch mode with total reflux.
b) Continuous feeding mode with finite reflux. Evaluate the influence of reflux ratio and feed stage.
c) Distillation using the packed column.

3.3. *McCabe-Thiele diagrams*

- Label all parts in the diagrams

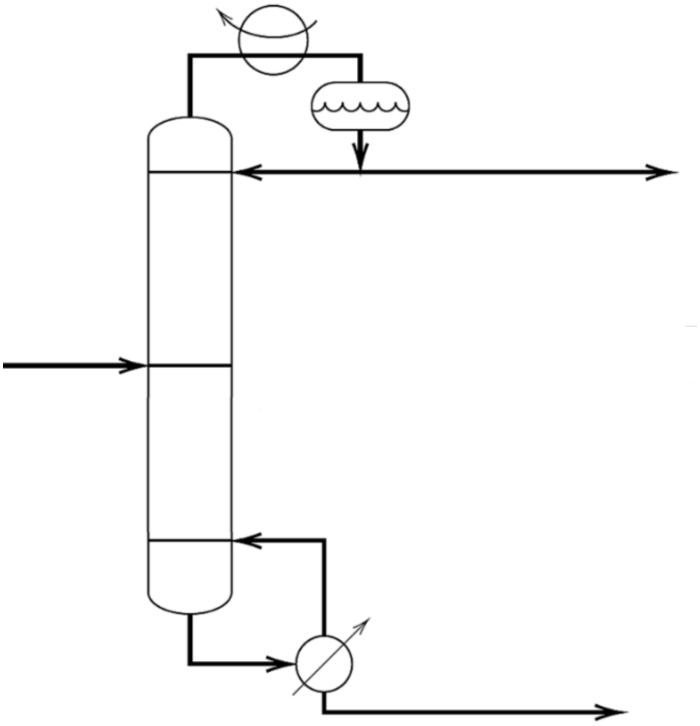
3.4. *Detailed material and energy balance of the system*

- Present detailed material and energy balance of all the components of the system.
- Explain assumptions and list thermodynamic data.
- It is recommended to use a scheme of the system and tables to summarise all the values.

Example:

Stream/Equipment	**Flow**		**xA**	**Density**	**Temperature**	**Energy**
	[mol/min]	**[ml/min]**	**[mol/mol]**	**[g/ml]**	**[°C]**	**[W*s]**
Feed (F)						
Distillate (D)						
Residues (W)						
Rectifying Vapor (V)						
Rectifying Liquid (L)						
Stripping Vapor (Vst)						
Stripping Liquid (Lst)						
Condenser						
Reboiler						

Temperature	$\Delta H_{vap,Water}$	$\Delta H_{vap,MeOH}$	$\Delta h_{l,Water}$	$\Delta h_{l,MeOH}$	$Cp_{,l,Water}$	$Cp_{,l,MeOH}$	$Cp_{,g,Water}$	$Cp_{,g,MeOH}$
[°C]	**[kJ/mol]**		**[kJ/mol]**		**[J/mol-K]**		**[J/mol-K]**	



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4. APPENDIX

I. Properties of water and ethanol

Properties	Water	Ethanol
Molar mass [g/mol]	18.016	46.069
Density at 20 °C [g/cm³]	0.998	0.789
Boiling point at 1 atm [°C]	100	78.24
Molar heat capacity at 25°C [J/mol K]	75.3	112.3
Enthalpy of vaporization [kJ/mol]	40.65	38.56

W.M. Haynes, *CRC Handbook of Chemistry and Physics,* 97th ed., CRC Press (2016).

II. Vapor-liquid equilibrium data for ethanol-water binary system

T [°C]	x_{EtOH}	y_{EtOH}
100	0	0
95.5	0.019	0.17
89.0	0.0721	0.3891
86.7	0.0966	0.4375
85.3	0.1238	0.4704
84.1	0.1661	0.5089
82.7	0.2337	0.548
82.3	0.2608	0.558
81.5	0.3273	0.583
80.7	0.3965	0.6122
79.8	0.5079	0.6564
79.7	0.5198	0.6599
79.3	0.5732	0.6841
78.74	0.6763	0.7385
78.41	0.7472	0.7815
78.15	0.8943	0.8943
-	0.93	0.92
-	0.97	0.96
78.24	1	1

x_{EtOH} and y_{EtOH} in mole fraction

R.H. Perry, C.H. Chilton and S.O. Kirkpatrick, *Chemical Engineering Handbook*, 7th ed., McGraw-Hill (1997).

III. Density of ethanol-water mixture as a function of ethanol mass fraction

%	10°C	15°C	20°C	25°C	30°C	35°C	40°C	%	10°C	15°C	20°C	25°C	30°C	35°C	40°C
0	0.99973	0.99913	0.99823	0.99708	0.99568	0.99406	0.99225	50	0.92126	0.91776	0.91384	0.90985	0.90580	0.90168	0.89750
1	785	725	636	520	379	217	034	51	.91943	555	160	760	353	.89940	519
2	602	542	453	336	194	031	.98846	52	723	333	.90936	534	125	710	288
3	426	365	275	157	014	.98849	663	53	502	110	711	307	.89896	479	056
4	258	195	103	.98984	.98839	672	485	54	279	.90885	485	079	667	248	.88823
5	098	032	.98938	817	670	501	311	55	055	659	258	.89850	437	016	589
6	.98946	.98877	780	656	507	335	142	56	.90831	433	031	621	206	.88784	356
7	801	729	627	500	347	172	.97975	57	607	207	.89803	392	.88975	552	122
8	660	584	478	346	189	009	808	58	381	.89980	574	162	744	319	.87888
9	524	442	331	193	031	.97846	641	59	154	752	344	.88931	512	085	653
10	393	304	187	043	.97875	685	475	60	.89927	523	113	699	278	.87851	417
11	267	171	047	.97897	723	527	312	61	698	293	.88882	446	044	615	180
12	145	041	.97910	753	573	371	150	62	468	062	650	233	.87809	379	.86943
13	026	.97914	775	611	424	216	.96989	63	237	.88830	417	.87998	574	142	705
14	.97911	790	643	472	278	063	829	64	006	597	183	763	337	.86905	466
15	800	669	514	334	133	.96911	670	65	.88774	364	.87948	527	100	667	227
16	692	552	387	199	.96990	760	512	66	541	130	713	291	.86863	429	.85987
17	583	433	259	062	844	607	352	67	308	.87895	477	054	625	190	747
18	473	313	129	.96923	697	452	189	68	074	660	241	.86817	387	.85950	407
19	363	191	.96997	782	547	294	023	69	.87839	424	004	579	148	710	266
20	252	068	864	639	395	134	.95856	70	602	187	.86766	340	.85908	470	025
21	139	.96944	729	495	242	.95973	687	71	365	.86949	527	100	667	228	.84783
22	024	818	592	348	087	809	516	72	127	710	287	.85859	426	.84986	540
23	.96907	689	453	199	.95929	643	343	73	.86888	470	047	618	184	743	297
24	787	558	312	048	769	476	168	74	648	229	.85806	376	.84941	500	053
25	665	424	168	.95895	607	306	.94991	75	408	.85988	564	134	698	257	.83809
26	539	287	020	738	442	133	810	76	168	747	322	.84891	455	013	564
27	406	144	.95867	576	272	.94955	625	77	.85927	505	079	647	211	.83768	319
28	268	.95996	710	410	098	774	438	78	685	262	.84835	403	.83966	523	074
29	125	844	548	241	.94922	590	248	79	442	018	590	158	720	277	.82827
30	.95977	686	382	067	741	403	055	80	197	.84772	344	.83911	473	029	578
31	823	524	212	.94890	557	214	.93860	81	.84950	525	096	664	224	.82780	329
32	665	357	038	709	370	021	662	82	702	277	.83848	415	.82974	530	079
33	502	186	.94860	525	180	.93825	461	83	453	028	599	164	724	279	.81828
34	334	011	679	337	.93986	626	257	84	203	.83777	348	.82913	473	027	576
35	162	.94832	494	146	790	425	051	85	.83951	525	095	660	220	.81774	322
36	.94986	650	306	.93952	591	221	.92843	86	697	271	.82840	405	.81965	519	067
37	805	464	114	756	390	016	634	87	441	014	583	148	708	262	.80811
38	620	273	.93919	556	186	.92808	422	88	181	.82754	323	.81888	448	003	552
39	431	079	720	353	.92979	597	208	89	.82919	492	062	626	186	.80742	291
40	238	.93882	518	148	770	385	.91992	90	654	227	.81797	362	.80922	478	028
41	042	682	314	.92940	558	170	774	91	386	.81959	529	094	655	211	.79761
42	.93842	478	107	729	344	.91952	554	92	114	688	257	.80823	384	.79941	491
43	639	271	.92897	516	128	733	332	93	.81839	413	.80983	549	111	669	220
44	433	062	685	301	.91910	513	108	94	561	134	705	272	.79835	393	.78947
45	226	.92852	472	085	692	291	.90884	95	278	.80852	424	.79991	555	114	670
46	017	640	257	.91868	472	069	660	96	.80991	566	138	706	271	.78831	388
47	.92806	426	041	649	250	.90845	434	97	698	274	.79846	415	.78981	542	100
48	593	211	.91823	429	028	621	207	98	399	.79975	547	117	684	247	.77806
49	379	.91995	604	208	.90805	396	.89979	99	094	670	243	.78814	382	.77946	507
								100	.79784	360	.78934	506	075	641	203

R.H. Perry, C.H. Chilton and S.O. Kirkpatrick, *Chemical Engineering Handbook*, 7th ed., McGraw-Hill (1997).

IV. List of chemicals and safety considerations

Name	**Formula**	**Risk description**	**Danger**	**PPE**
Ethanol	CH_3CH_2OH	***Hazard statement(s)*** H225: Highly flammable liquid and vapour H319: Causes serious eye irritation ***Precautionary statement(s)*** P210: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking P233: Keep container tightly closed P305+P351+P338 if in eyes: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.