

MODULE 5: DISTILLATION

LECTURE NO. 1

5.1. Introduction

Distillation is method of separation of components from a liquid mixture which depends on the differences in boiling points of the individual components and the distributions of the components between a liquid and gas phase in the mixture. The liquid mixture may have different boiling point characteristics depending on the concentrations of the components present in it. Therefore, distillation processes depends on the vapor pressure characteristics of liquid mixtures. The vapor pressure is created by supplying heat as separating agent. In the distillation, the new phases differ from the original by their heat content. During most of the century, distillation was by far the most widely used method for separating liquid mixtures of chemical components (Seader and Henley, 1998). This is a very energy intensive technique, especially when the relative volatility of the components is low. It is mostly carried out in multi tray columns. Packed column with efficient structured packing has also led to increased use in distillation.

5.1.1. Vapor Pressure

Vapor Pressure: The vaporization process changes liquid to gaseous state. The opposite process of this vaporization is called condensation. At equilibrium, the rates of these two processes are same. The pressure exerted by the vapor at this equilibrium state is termed as the vapor pressure of the liquid. It depends on the temperature and the quantity of the liquid and vapor. From the following Clausius-Clapeyron Equation or by using Antoine Equation, the vapor pressure can be calculated.

Clausius-Clapeyron Equation:

$$\ln\left(\frac{p^v}{p_1^v}\right) = \left(\frac{\lambda}{R}\right)\left(\frac{1}{T_1} - \frac{1}{T}\right) \quad (5.1)$$

where p^v and p_1^v are the vapor pressures in Pascal at absolute temperature T and T_1 in K. λ is the molar latent heat of vaporization which is independent of temperature.

Antoine Equation:

$$\ln p^v (\text{Pascal}) = A - \frac{B}{T + C} \quad (5.2)$$

Typical representative values of the constants A, B and C are given in the following Table 5.1 (Ghosal et al., 1993)

Table 5.1: Typical representative values of the constants A, B and C

Components	Range of Temperature (T), K	A	B	C
Acetone	241-350	21.5439	2940.46	-35.93
Ammonia	179-261	21.8407	2132.50	-32.98
Benzene	280-377	20.7934	2788.51	-52.36
Ethanol	270-369	21.8045	3803.98	-41.68
Methanol	257-364	23.4801	3626.55	-34.29
Toluene	280-410	20.9063	3096.52	-53.67
Water	284-441	23.1962	3816.44	-46.13

Readers are suggested to revise the thermodynamics for more about vapor pressure and boiling point.

5.1.2. Phase Diagram

For binary mixture phase diagram only two-component mixture, (e.g. A (more volatile) and B (less volatile)) are considered. There are two types of phase diagram: constant pressure and constant temperature.

5.1.3. Constant Pressure Phase Diagram

The Figure 5.1 shows a constant pressure phase diagram for an ideal solution (one that obeys Raoult's Law). At constant pressure, depending on relative

concentrations of each component in the liquid, many boiling point temperatures are possible for mixture of liquids (solutions) as shown in phase diagram diagram (Figure 5.1). For mixture, the temperature is called bubble point temperature when the liquid starts to boil and dew point when the vapor starts to condense. Boiling of a liquid mixture takes place over a range of boiling points. Likewise, condensation of a vapor mixture takes place over a range of condensation points. The upper curve in the boiling point diagram is called the dew-point curve (DPC) while the lower one is called the bubble-point curve (BPC). At each temperature, the vapor and the liquid are in equilibrium. The constant pressure phase diagram is more commonly used in the analysis of vapor-liquid equilibrium.

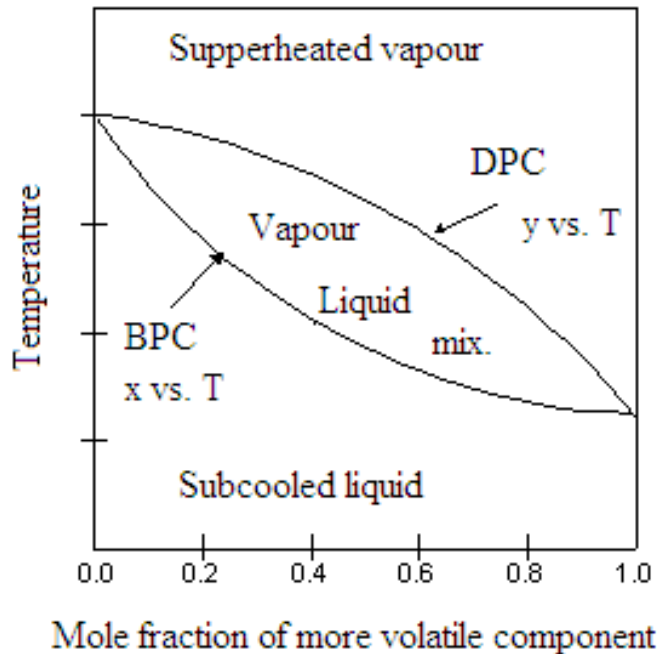


Figure 5.1: Phase diagram of binary system at constant pressure

5.1.4. Constant temperature (isothermal) phase diagram

The constant temperature phase diagram is shown in Figure 5.2. The constant temperature phase diagram is useful in the analysis of solution behaviour. The more volatile liquid will have a higher vapor pressure (i.e. p_A at $x_A = 1.0$)

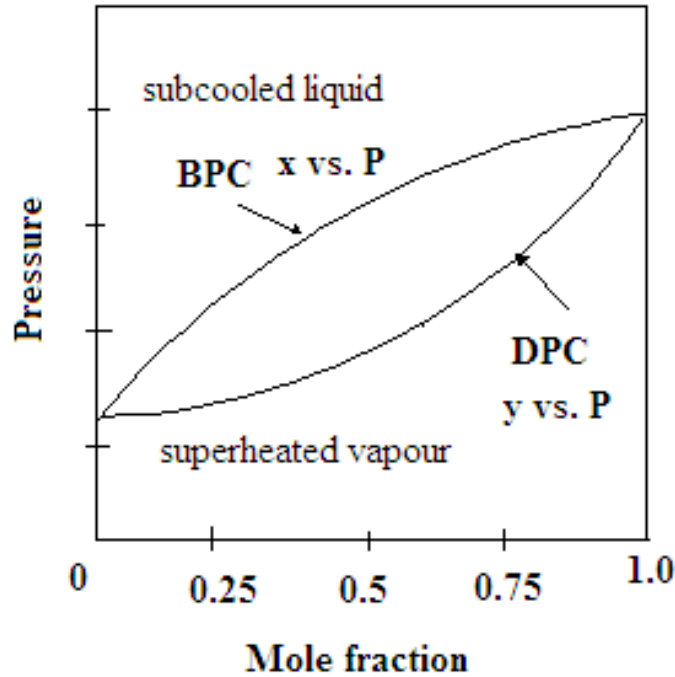


Figure 5.2: Binary Phase diagram at constant temperature

5.1.5. Relative volatility

Relative volatility is a measure of the differences in volatility between two components, and hence their boiling points. It indicates how easy or difficult a particular separation will be. The relative volatility of component 'A' with respect to component 'B' in a binary mixture is defined as

$$\alpha_{AB} = \frac{y_A/x_A}{y_B/x_B} \quad (5.3)$$

where, y_A = mole fraction of component 'A' in the vapor, x_A = mole fraction of component 'A' in the liquid. In general, relative volatility of a mixture changes with the mixture composition. For binary mixture, $x_B = 1-x_A$. So Equation (5.3) can be rearranged, simplifying and expressed by dropping subscript 'A' for more volatile component as:

$$y = \frac{\alpha_{ave}x}{1 - (\alpha_{ave} - 1)x} \quad (5.4)$$

The Equation (5.4) is a non-linear relationships between x and y . This Equation can be used to determine the equilibrium relationship (y vs. x) provided the

average relative volatility, α_{ave} is known. If the system obeys Raoult's law, i.e. $p_A = Py_A$, $p_B = Py_B$, the relative volatility can be expressed as:

$$\alpha_{AB} = \frac{p_A/x_A}{p_B/x_B} \quad (5.5)$$

where p_A is the partial pressure of component A in the vapor, p_B is the partial pressure of component B in the vapor and P is the total pressure of the system. Thus if the relative volatility between two components is equal to one, separation is not possible by distillation. The larger the value of α , above 1.0, the greater the degree of separability, i.e. the easier the separation. Some examples of optimal relative volatility that are used for distillation process design are given in Table 5.2.

Table 5.2: Some optimal relative volatility that are used for distillation process design

More volatile component (normal boiling point °C)	Less boiling component (normal boiling point, °C)	Optimal Relative volatility
Benzene (80.1)	Toluene (110.6)	2.34
Toluene (110.6)	p-Xylene (138.3)	2.31
Benzene (80.6)	p-Xylene (138.3)	4.82
m-Xylene (139.1)	p-Xylene (138.3)	1.02
Pentane (36.0)	Hexane (68.7)	2.59
Hexane (68.7)	Heptane (98.5)	2.45
Hexane (68.7)	p-Xylene (138.3)	7.0
Ethanol (78.4)	iso-Propanol (82.3)	1.17
iso-Propanol (82.3)	n-Propanol (97.3)	1.78
Ethanol (78.4)	n-Propanol (97.3)	2.10
Methanol (64.6)	Ethanol (78.4)	1.56
Methanol (64.6)	iso-Propanol (82.3)	2.26
Chloroform (61.2)	Acetic acid (118.1)	6.15

5.1.6. Vapor-Liquid Equilibrium (VLE)

It is useful for graphical design in determining the number of theoretical stages required for a distillation column. A typical equilibrium curve for a binary mixture on x-y plot is shown in Figure 5.3. It can be plotted by the Equations (5.4) or (5.5) as discussed earlier section. It contains less information than the phase diagram (i.e. temperature is not included), but it is most commonly used. The VLE plot

expresses the bubble-point and the dew-point of a binary mixture at constant pressure. The curved line in Figure 5.3 is called the equilibrium line and it describes the compositions of the liquid and vapor in equilibrium at some fixed pressure. The equilibrium line can be obtained from the Equation (5.4) once the relative volatility is known.

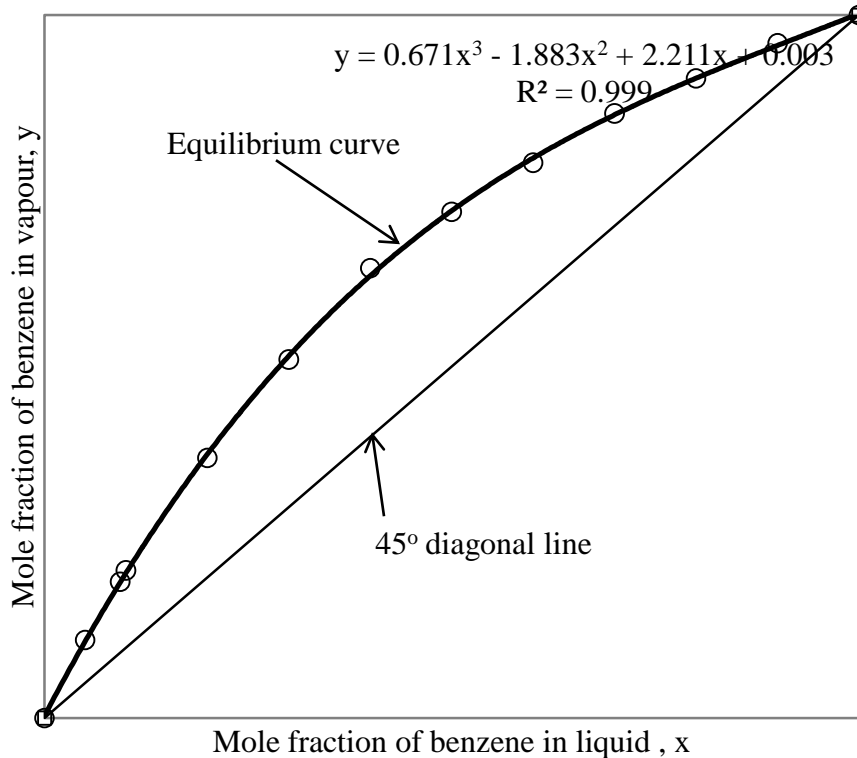


Figure 5.3: Equilibrium diagram for a benzene-toluene mixture at 1 atmosphere

This particular VLE plot (Figure 5.3) shows a binary ideal mixture that has a uniform vapor-liquid equilibrium that is relatively easy to separate. On the other hand, the VLE plots shown in Figure 5.4 represented for non-ideal systems. These non-ideal VLE systems will present more difficult separation.

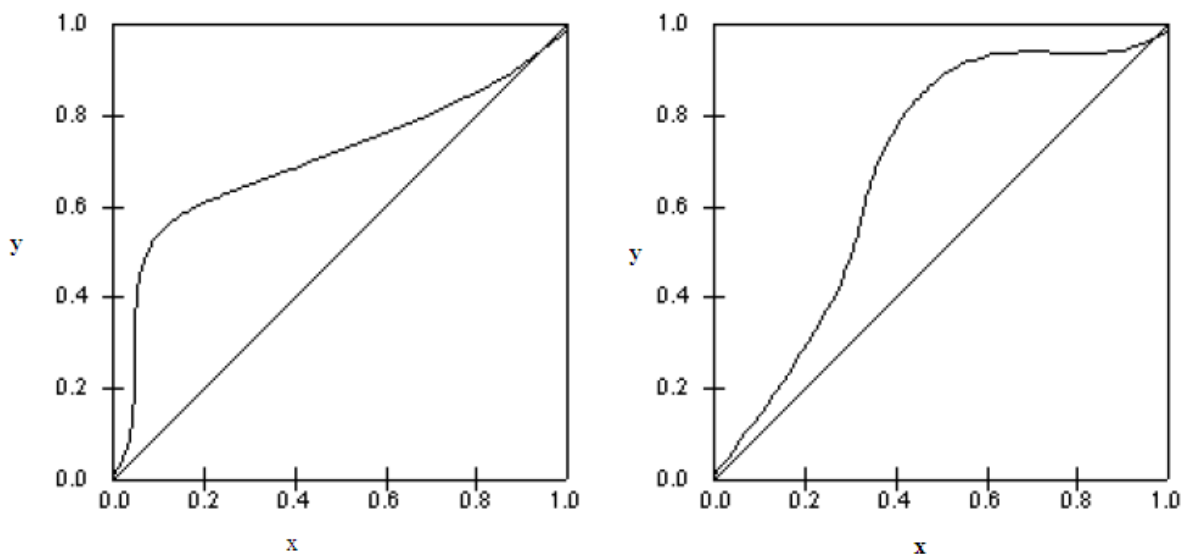


Figure 5.4: Vapor-liquid equilibrium curve for non-ideal systems

The most challenging VLE curves are generated by azeotropic systems. An azeotrope is a liquid mixture which when vaporized, produces the same composition as the liquid. Two types of azeotropes are known: minimum-boiling and maximum-boiling (less common). Ethanol-water system (at 1 atm, 89.4 mole %, 78.2 °C; Carbon-disulfide - acetone (61.0 mole% CS₂, 39.25 °C, 1 atm) and Benzene - water (29.6 mole% H₂O, 69.25 °C, 1 atm) are minimum-boiling azeotropes. Hydrochloric acid - water (11.1 mole% HCl, 110 °C, 1 atm); Acetone - chloroform (65.5 mole% chloroform, 64.5 °C, 1 atm) are the examples of Maximum-boiling azeotropes. The Figure 5.5 shows two different azeotropic systems, one with a minimum boiling point (Figure 5.5a) and one with a maximum boiling point (Figure 5.5b). The points of intersections of the equilibrium curves with the diagonal lines are called azeotropic points. An azeotrope cannot be separated by conventional distillation. However, vacuum distillation may be used as the lower pressures which can shift the azeotropic point.

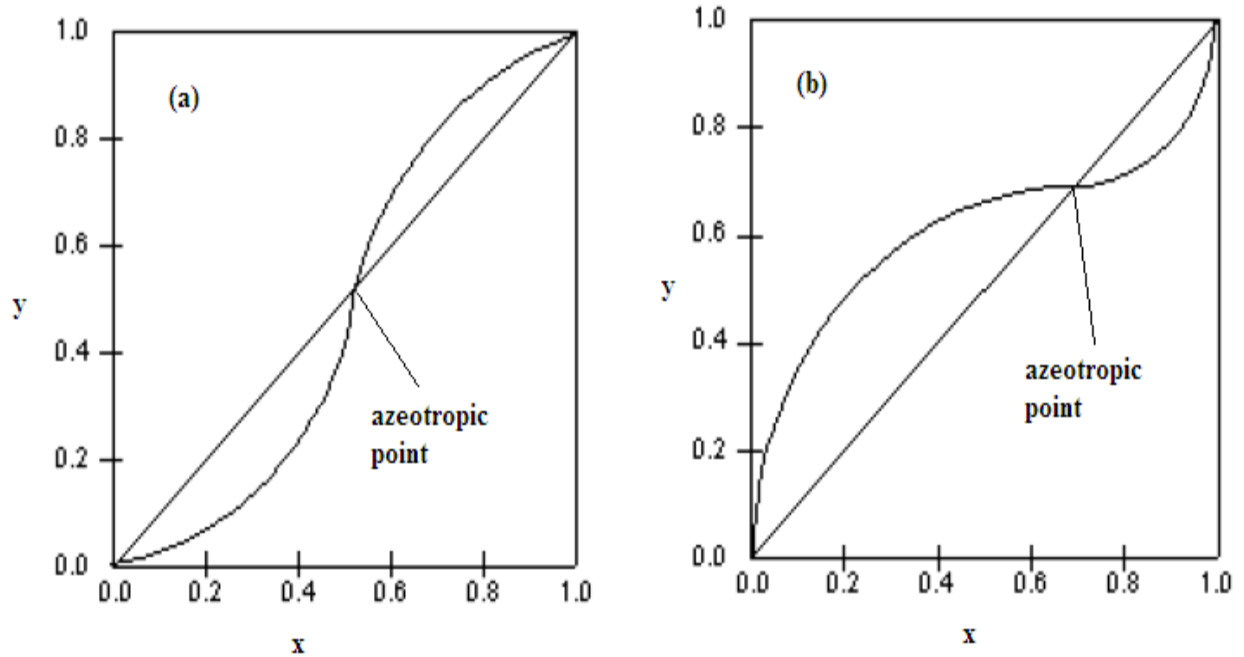


Figure 5.5: VLE curves for azeotropic systems: (a) for maximum boiling point, (b) for minimum boiling point

Although most distillations are carried out at atmospheric or near atmospheric pressure, it is not uncommon to distill at other pressures. High pressure distillation (typically 3 - 20 atm) usually occurs in thermally integrated processes. In those cases the equilibrium curve becomes narrower at higher pressures as

shown in Figure 5.6. Separability becomes less at higher pressures.

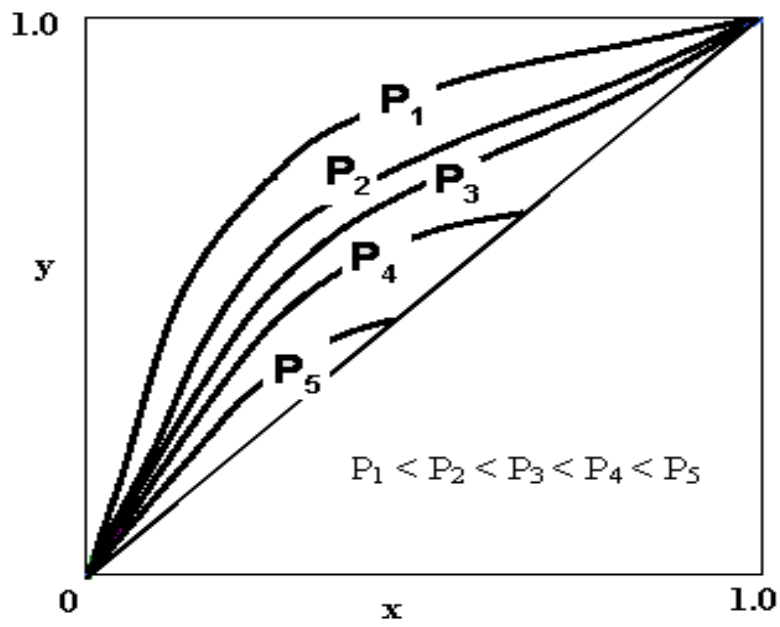


Figure 5.6: Variation of equilibrium curve with pressure

MODULE 5: DISTILLATION

LECTURE NO. 2

5.2. Distillation columns and their process calculations

There are many types of distillation columns each of which is designed to perform specific types of separations. One way of classifying distillation column type is to look at how they are operated. Based on operation, they are of two types: batch or differential and continuous columns.

5.2.1. Batch or differential distillation columns and their process calculation

In batch operation, the feed is introduced batch-wise to the column. That is, the column is charged with a 'batch' and then the distillation process is carried out. When the desired task is achieved, a next batch of feed is introduced. Consider a binary mixture of components A (more volatile) and B (less volatile). The system consists of a batch of liquid (fixed quantity) inside a kettle (or still) fitted with heating element and a condenser to condense the vapor produced as shown in Figure 5.7. The condensed vapor is known as the distillate. The distillate is collected in a condensate receiver. The liquid remaining in the still is known as the residual. The process is unsteady state. The concentration changes can be analyzed using the phase diagram, and detailed mathematical calculations carried out using the Rayleigh Equation. Readers are suggested to follow chapter 9 (Page 369) of text book “Mass-transfer operations” by R. E Treybal, third edition. As the process is unsteady state, the derivation is based on a differential approach to changes in concentration with time. Let L_1 = initial moles of liquid

originally in still, L_2 = final moles of liquid remained in still, x_1 = initial liquid composition in still (mole fraction of A), x_2 = final liquid composition in still (mole fraction A). At any time t , the amount of liquid in the still is L , with mole fraction of A in the liquid being x . After a small differential time ($t + dt$), a small amount of vapor dL is produced, and the composition of A in the vapor is y (mole fraction). The vapor is assumed to be in equilibrium with the residue liquid. The amount of liquid in the still is thus reduced from L to $(L - dL)$, while the liquid composition changed from x to $(x - dx)$. Then the material balance on A can be written as:

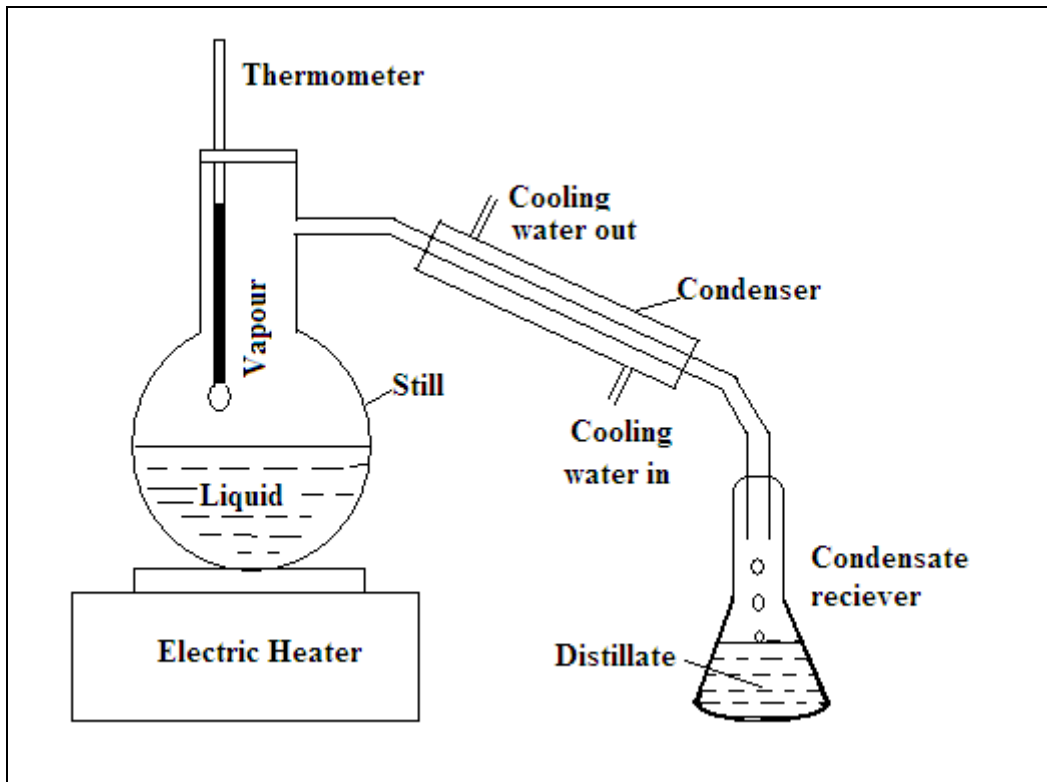


Figure 5.7: Simple batch or differential distillation process

Initial amount in still = Amount left in still + Amount vaporized

$$xL = (x - dx)(L - dL) + ydL \tag{5.6}$$

or

$$xL = xL - xdL - Ldx + dxL + ydL \quad (5.7)$$

Neglecting the term $(dx)(dL)$, the Equation (5.7) may be written as:

$$Ldx = ydL - xdL \quad (5.8)$$

Re-arranging and Integrating from L_1 to L_2 , and from x_1 to x_2 , one can obtain the following Equation which is called Rayleigh Equation:

$$\ln\left(\frac{L_1}{L_2}\right) = \int_{x_2}^{x_1} \frac{1}{(y-x)} dx \quad (5.9)$$

The integration of Equation (5.9) can be obtained graphically from the equilibrium curve, by plotting $1/(y-x)$ versus x .

Example problem 5.1

A mixture of 40 mole % isopropanol in water is to be batch-distilled at 1 atm until 70 mole % of the charge has been vaporized. Calculate the composition of the liquid residue remaining in the still pot, and the average composition of the collected distillate. VLE data for this system, in mole fraction of isopropanol, at 1 atm are (Seader and Henley, 1998):

Temp.(K)	366	357	355.1	354.3	353.6	353.2	353.3	354.5
y	0.220	0.462	0.524	0.569	0.593	0.682	0.742	0.916
x	0.012	0.084	0.198	0.350	0.453	0.679	0.769	0.944

Solution 5.1:

Calculate $1/(y-x)$

As per Rayleigh Equation

$$\ln\left(\frac{L_1}{L_2}\right) = \int_{x_2}^{x_1} \frac{1}{(y-x)} dx$$

$$x_1 = 0.4$$

$$\text{Feed } L_1 = 100$$

$$\text{Distillate (D)} = 70$$

$$\text{Liquid residue as } L_2 = L_1 - D = 30$$

$$\text{Find } x_2 \text{ by equating } \ln\left(\frac{L_1}{L_2}\right) = \int_{x_2}^{x_1} \frac{1}{(y-x)} dx$$

$$\text{The value of } x_2 = 0.067$$

$$y_D = \frac{L_1 x_1 - L_2 x_2}{D} = 0.543$$

MODULE 5: DISTILLATION

LECTURE NO. 3

5.2.2. Continuous distillation columns

In contrast, continuous columns process a continuous feed stream. No interruptions occur unless there is a problem with the column or surrounding process units. They are capable of handling high throughputs. Continuous column is the more common of the two types.

Types of Continuous Columns

Continuous columns can be further classified according to the nature of the feed that they are processing:

Binary distillation column: feed contains only two components

Multi-component distillation column: feed contains more than two components the number of product streams they have

Multi-product distillation column: column has more than two product streams where the extra feed exits when it is used to help with the separation,

Extractive distillation: where the extra feed appears in the bottom product stream

Azeotropic distillation: where the extra feed appears at the top product stream the type of column internals.

Tray distillation column: where trays of various designs are used to hold up the liquid to provide better contact between vapor and liquid, hence better separation. The details of the tray column are given in Module 4.

Packed distillation column: where instead of trays, 'packings' are used to enhance contact between vapor and liquid. The details of the packed column are given in Module 4.

4.2.2.1. A single-stage continuous distillation (Flash distillation):

A single-stage continuous operation occurs where a liquid mixture is partially vaporized. The vapor produced and the residual liquids are in equilibrium in the process are separated and removed as shown in Figure 5.8. Consider a binary mixture of A (more volatile component) and B (less volatile component). The feed is preheated before entering the separator. As such, part of the feed may be vaporized. The heated mixture then flows through a pressure-reducing valve to the separator. In the separator, separation between the vapor and liquid takes place. The amount of vaporization affects the concentration (distribution) of A in vapor phase and liquid phase. The relationship between the scale of vaporization and mole fraction of A in vapor and liquid (y and x) is known as the Operating Line Equation. Define f as molal fraction of the feed that is vaporized and withdrawn continuously as vapor. Therefore, for 1 mole of binary feed mixture, $(1-f)$ is the molal fraction of the feed that leaves continuously as liquid. Assume, y_D = mole fraction of A in vapor leaving, x_B = mole fraction of A in liquid leaving, x_F = mole fraction of A in feed entering. Based on the definition for f , the greater the heating is, the larger the value of f . If the feed is completely vaporized, then $f = 1.0$. Thus, the value of f can vary from 0 (no vaporization) to 1 (total vaporization). From material balance for the more volatile component (A) one can write

$$1.x_F = fy_D + (1-f)x_B \quad (5.10)$$

Or,

$$fy_D = x_F - (1-f)x_B \quad (5.11)$$

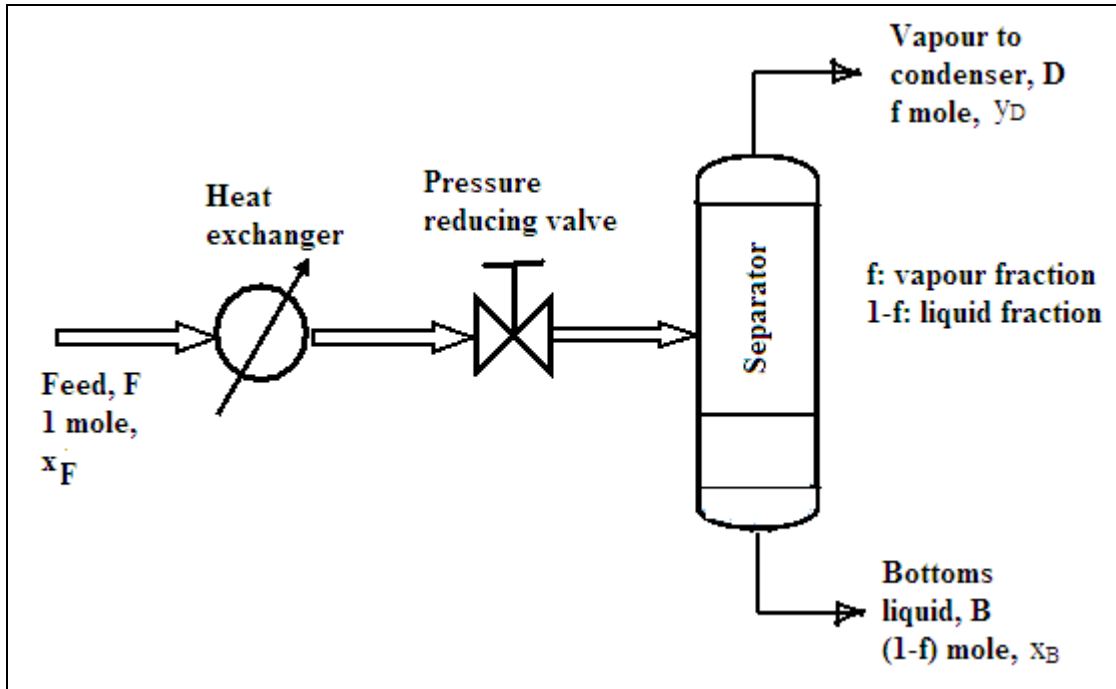


Figure 5.8: Flash distillation process

The Equation (5.11) on rearranging becomes:

$$y_D = -\left(\frac{1-f}{f}\right)x_B + \frac{x_F}{f} \quad (5.12)$$

The fraction f depends on the enthalpy of the liquid feed, the enthalpies of the vapor and liquid leaving the separator. For a given feed condition, and hence the known value of f and x_F , the Equation (5.12) is a straight line Equation with slope $-(1-f)/f$ and intercept x_F/f as shown in Figure 5.9. It will intersect the equilibrium line at the point (x_B, y_D) . From this value, the composition of the vapor and liquid leaving the separator can be obtained.

before it enters the distillation column. The feed enters the column at feed stage contains more volatile components (called light key, LK) and less volatile components (called heavy key, HK). At the feed stage feed may be liquid, vapor or mixture of liquid and vapor. The section above the feed where vapor is washed with the reflux to remove or absorb the heavy key is called enriching or rectifying section. The section below the feed stage where liquid is stripped of the light key by the rising vapor is called stripping section.

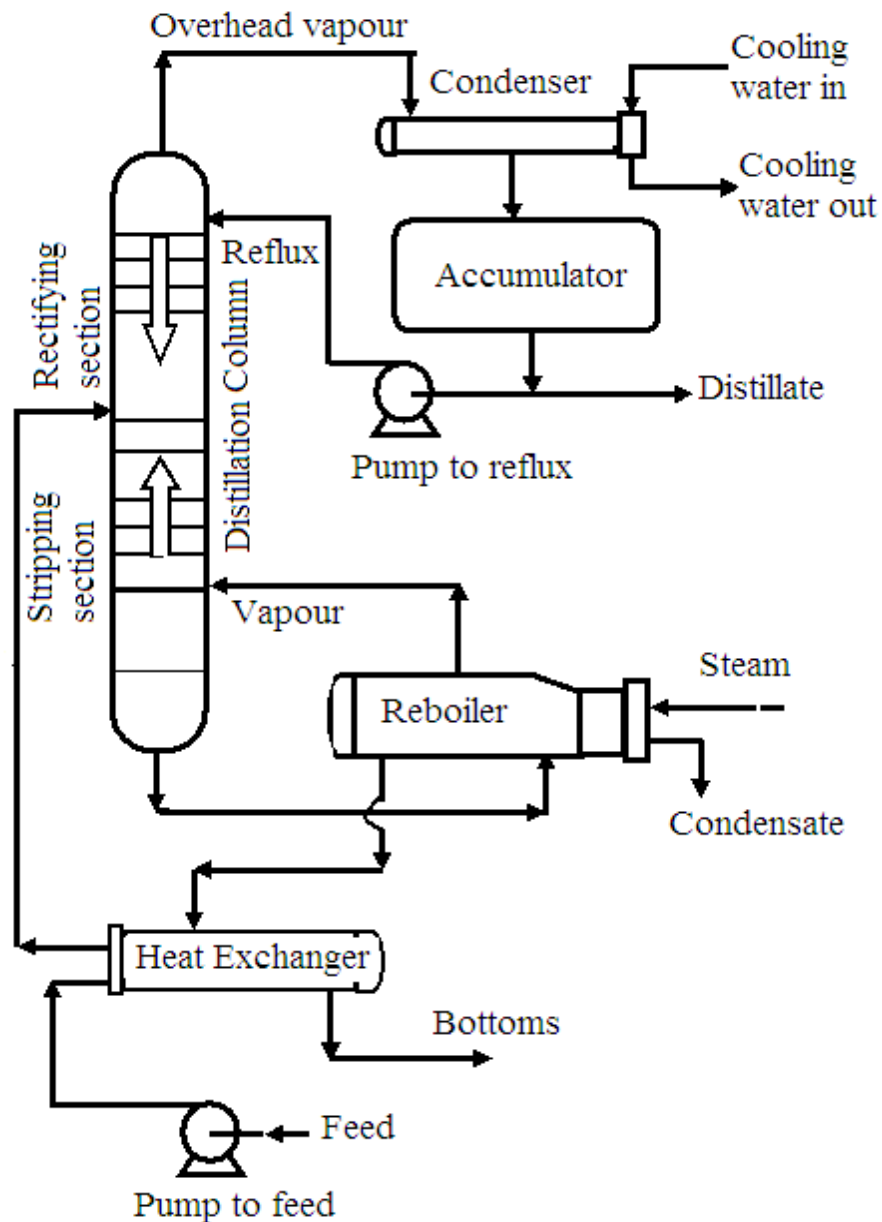


Figure 5.10: Multi-stage binary distillation column

MODULE 5: DISTILLATION

LECTURE NO. 4

5.2.2.3. Analysis of binary distillation in tray towers: McCabe-Thiele Method

McCabe and Thiele (1925) developed a graphical method to determine the theoretical number of stages required to effect the separation of a binary mixture (McCabe and Smith, 1976). This method uses the equilibrium curve diagram to determine the number of theoretical stages (trays) required to achieve a desired degree of separation. It assumes constant molar overflow and this implies that: (i) molal heats of vaporization of the components are roughly the same; (ii) heat effects are negligible. The information required for the systematic calculation are the VLE data, feed condition (temperature, composition), distillate and bottom compositions; and the reflux ratio, which is defined as the ratio of reflux liquid over the distillate product. For example, a column is to be designed for the separation of a binary mixture as shown in Figure 5.11.

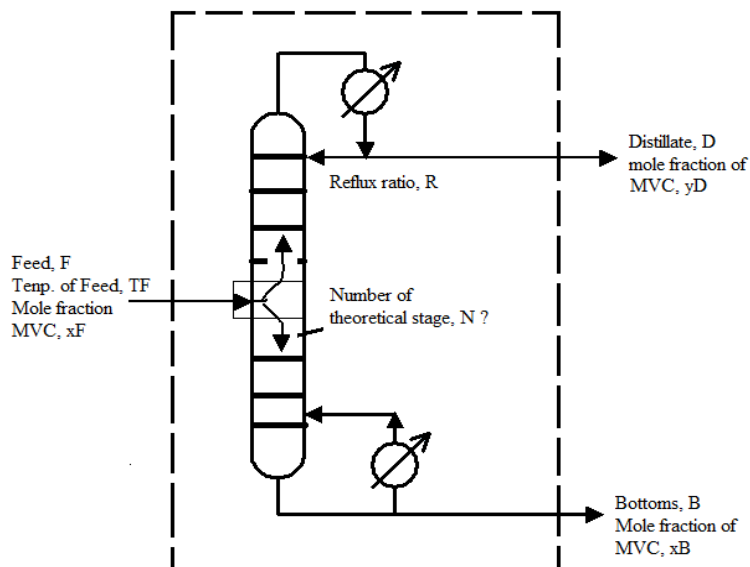


Figure 5.11: Schematic of column for separation of binary mixture

The feed has a concentration of x_F (mole fraction) of the more volatile component, and a distillate having a concentration of x_D of the more volatile component and a bottoms having a concentration of x_B is desired. In its essence, the method involves the plotting on the equilibrium diagram three straight lines: the rectifying section operating line (ROL), the feed line (also known as the q -line) and the stripping section operating line (SOL). An important parameter in the analysis of continuous distillation is the Reflux Ratio, defined as the quantity of liquid returned to the distillation column over the quantity of liquid withdrawn as product from the column, i.e. $R = L / D$. The reflux ratio R is important because the concentration of the more volatile component in the distillate (in mole fraction x_D) can be changed by changing the value of R . The steps to be followed to determine the number of theoretical stages by McCabe-Thiele Method:

- Determination of the Rectifying section operating line (ROL).
- Determination the feed condition (q).
- Determination of the feed section operating line (q -line).
- Determination of required reflux ratio (R).
- Determination of the stripping section operating line (SOL).

- Determination of number of theoretical stage.

Determination of the Rectifying section operating line (ROL)

Consider the rectifying section as shown in the Figure 5.12. Material balance can be written around the envelope shown in Figure 5.12:

Overall or total balance:

$$V_{n+1} = L_n + D \quad (5.13)$$

Component balance for more volatile component:

$$V_{n+1}y_{n+1} = L_nx_n + Dx_D \quad (5.14)$$

From Equations (4.13) and (4.14), it can be written as

$$(L_n + D)y_{n+1} = L_nx_n + Dx_D \quad (5.15)$$

Consider the constant molal flow in the column, and then one can write: $L_1 = L_2 = \dots \dots \dots L_{n-1} = L_n = L_{n+1} = L = \text{constant}$ and $V_1 = V_2 = \dots \dots \dots V_{n-1} = V_n = V_{n+1} = V = \text{constant}$. Thus, the Equation (5.15) becomes:

$$(L + D)y_{n+1} = Lx_n + Dx_D \quad (5.16)$$

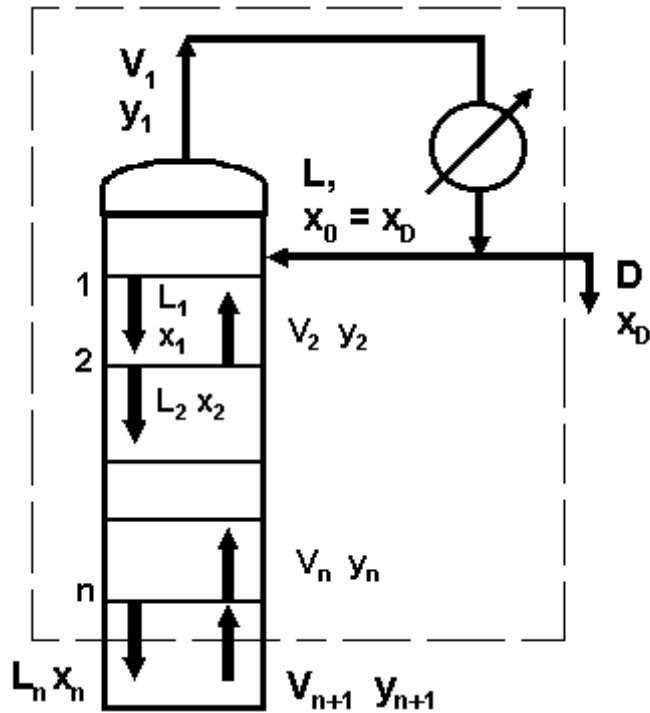


Figure 5.12: Outline graph of rectifying section

After rearranging, one gets from Equation (5.16) as:

$$y_{n+1} = \left(\frac{L}{L+D} \right) x_n + \left(\frac{D}{L+D} \right) x_D \quad (5.17)$$

Introducing reflux ratio defined as: $R = L/D$, the Equation (5.17) can be expressed as:

$$y_{n+1} = \left(\frac{R}{R+1} \right) x_n + \left(\frac{1}{R+1} \right) x_D \quad (5.18)$$

The Equation (5.18) is the rectifying section operating line (ROL) Equation having slope $R/(R+1)$ and intercept, $x_D/(R+1)$ as shown in Figure 5.13. If $x_n = x_D$, then $y_{n+1} = x_D$, the operating line passed through the point (x_D, x_D) on the 45° diagonal line. When the reflux ratio R changed, the ROL will change. Generally the

rectifying operating line is expressed without subscript of n or n+1. Without subscript the ROL is expressed as:

$$y = \left(\frac{R}{R+1}\right)x + \left(\frac{1}{R+1}\right)x_D \quad (5.19)$$

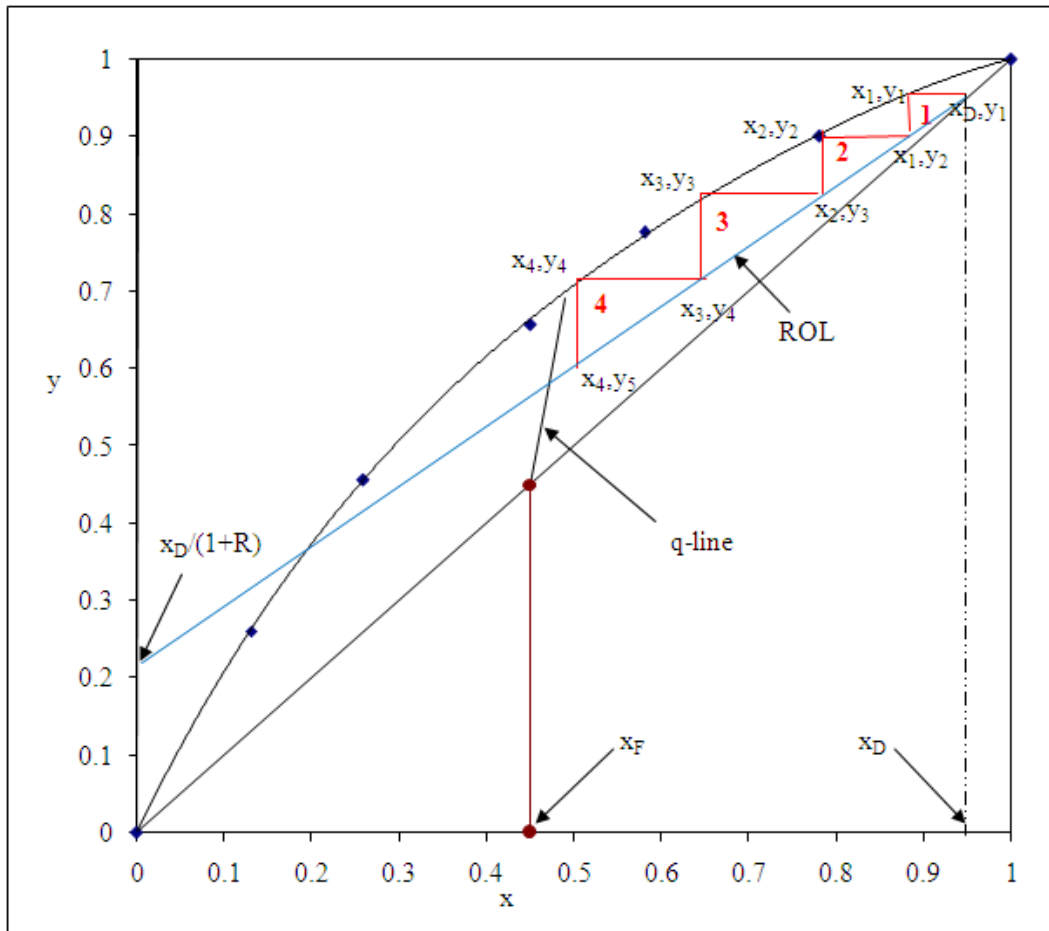


Figure 5.13: Representation of the rectifying operating line

Determination the feed condition (q):

The feed enters the distillation column may consists of liquid, vapor or a mixture of both. Some portions of the feed go as the liquid and vapor stream to the rectifying and stripping sections. The moles of liquid flow in the stripping section that result from the introduction of each mole of feed, denoted as 'q'. The limitations of the q-value as per feed conditions are shown in Table 5.3.

Table 5.3: Limitations of q-value as per feed conditions

Feed condition	Limit of q-value
cold feed (below bubble point)	$q > 1$
feed at bubble point (saturated liquid)	$q = 1$
feed as partially vaporized	$0 < q < 1$
feed at dew point (saturated vapor)	$q = 0$
feed as superheated vapor	$q < 0$
feed is a mixture of liquid and vapor	q is the fraction of the feed that is liquid

Calculation of q-value

When feed is partially vaporized:

Other than saturated liquid ($q = 1$) and saturated vapor ($q = 0$), the feed condition is uncertain. In that case one must calculate the value of q . The q -value can be obtained from enthalpy balance around the feed plate. By enthalpy balance one can obtain the q -value from the following form of Equation:

$$q = \frac{H_V - H_F}{H_V - H_L} \quad (5.20)$$

where H_F , H_V and H_L are enthalpies of feed, vapor and liquid respectively which can be obtained from enthalpy-concentration diagram for the mixture.

When feed is cold liquid or superheated vapor:

q can be alternatively defined as the heat required to convert 1 mole of feed from its entering condition to a saturated vapor; divided by the molal latent heat of vaporization. Based on this definition, one can calculate the q -value from the following Equations for the case whereby $q > 1$ (cold liquid feed) and $q < 0$ (superheated vapor feed) as:

For cold liquid feed:

$$q = \frac{C_{p,L}(T_{bp} - T_F) + \lambda}{\lambda} \quad (5.21)$$

For superheated vapor feed:

$$q = \frac{C_{p,V}(T_{dp} - T_F)}{\lambda} \quad (5.22)$$

where T_{bp} is the bubble point, λ is the latent heat of vaporization and T_{dp} is the dew point of the feed respectively.

Determination of the feed section operating line (q-line):

Consider the section of the distillation column (as shown in Figure 5.11) at the tray (called feed tray) where the feed is introduced. In the feed tray the feed is introduced at F moles/hr with liquid of q fraction of feed and vapor of $(1-q)$ fraction of feed as shown in Figure 5.14. Overall material balance around the feed tray:

$$L' = L + qF \quad \text{and} \quad V = V' + (1-q)F \quad (5.23)$$

Component balances for the more volatile component in the rectifying and stripping sections are:

For rectifying section:

$$Vy = Lx + Dx_D \quad (5.24)$$

For stripping section:

$$V'y = L'x - Bx_B \quad (5.25)$$

At the feed point where the two operating lines (Equations (5.24) and (5.25)) intersect can be written as:

$$(V - V')y = (L - L')x + Dx_D + Bx_B \quad (5.26)$$

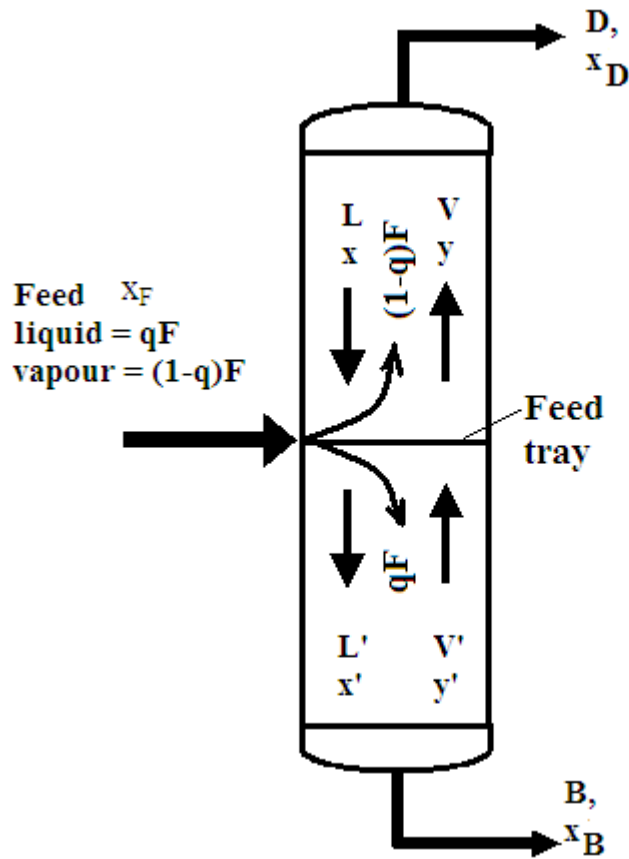


Figure 5.14: Feed tray with fraction of liquid and vapor of feed

From component balance around the entire column, it can be written as

$$Fx_F = Dx_D + Bx_B \quad (5.27)$$

Substituting L-L' and V-V' from Equations (5.23) and (5.24) into Equation (5.26) and with Equation (5.27) one can get the q-line Equation after rearranging as:

$$y = -\left(\frac{q}{1-q}\right)x + \left(\frac{1}{1-q}\right)x_F \quad (5.28)$$

For a given feed condition, x_F and q are fixed, therefore the q-line is a straight line with slope $-q / (1-q)$ and intercept $x_F/(1-q)$. If $x = x_F$, then from Equation

(5.28) $y = x_F$. At this condition the q-line passes through the point (x_F, x_F) on the 45° diagonal. Different values of q will result in different slope of the q-line. Different q-lines for different feed conditions are shown in Figure 5.15.

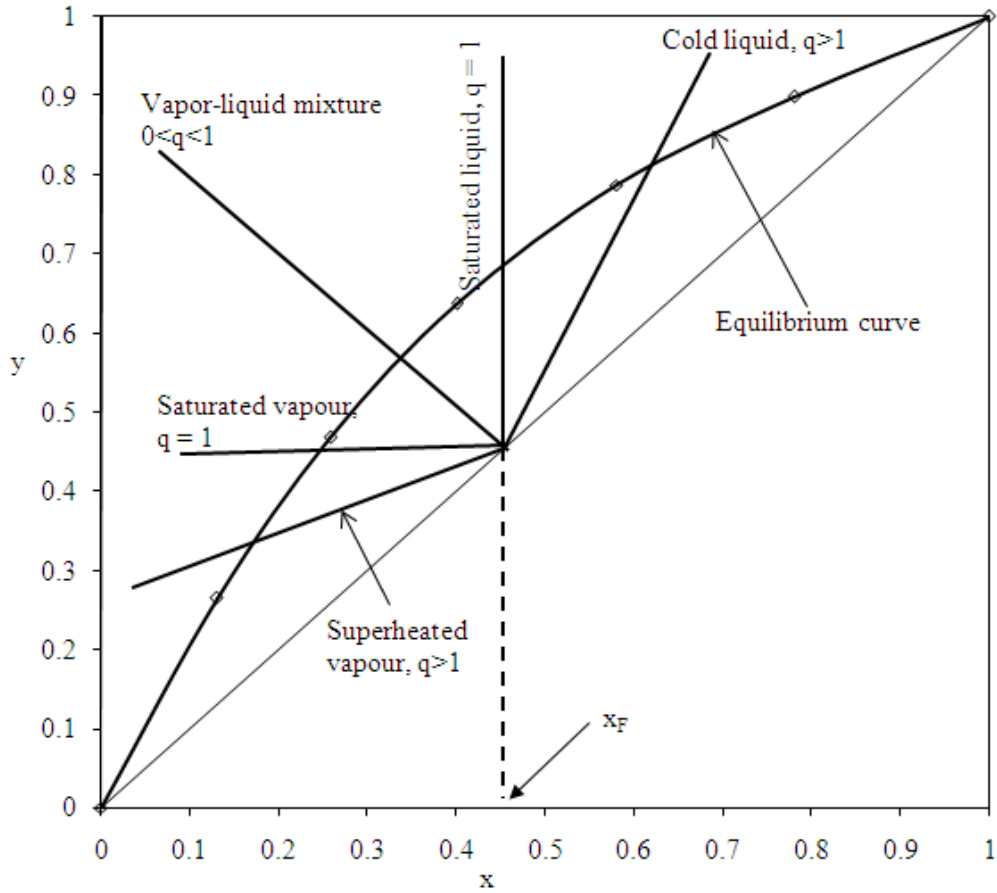


Figure 5.15: Different q-lines for different feed conditions

MODULE 5: DISTILLATION

LECTURE NO. 5

Determination of the stripping section operating line (SOL):

The stripping section operating line (SOL) can be obtained from the ROL and q -line without doing any material balance. The SOL can be drawn by connecting point x_B on the diagonal to the point of intersection between the ROL and q -line. The SOL will change if q -line is changed at fixed ROL. The change of SOL with different q -lines for a given ROL at constant x_D is shown in Figure 5.16.

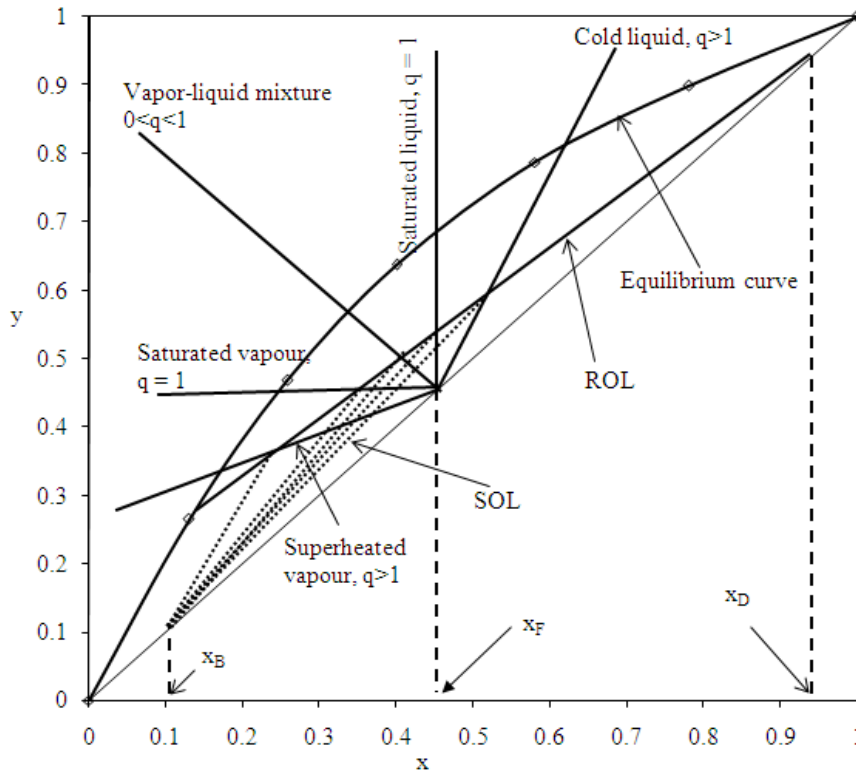


Figure 5.16: Stripping section operating line with different q -lines

The stripping section operating line can be derived from the material balance around the stripping section of the distillation column. The stripping section of a distillation column is shown in Figure 5.17. The reboiled vapor is in equilibrium with bottoms liquid which is leaving the column.

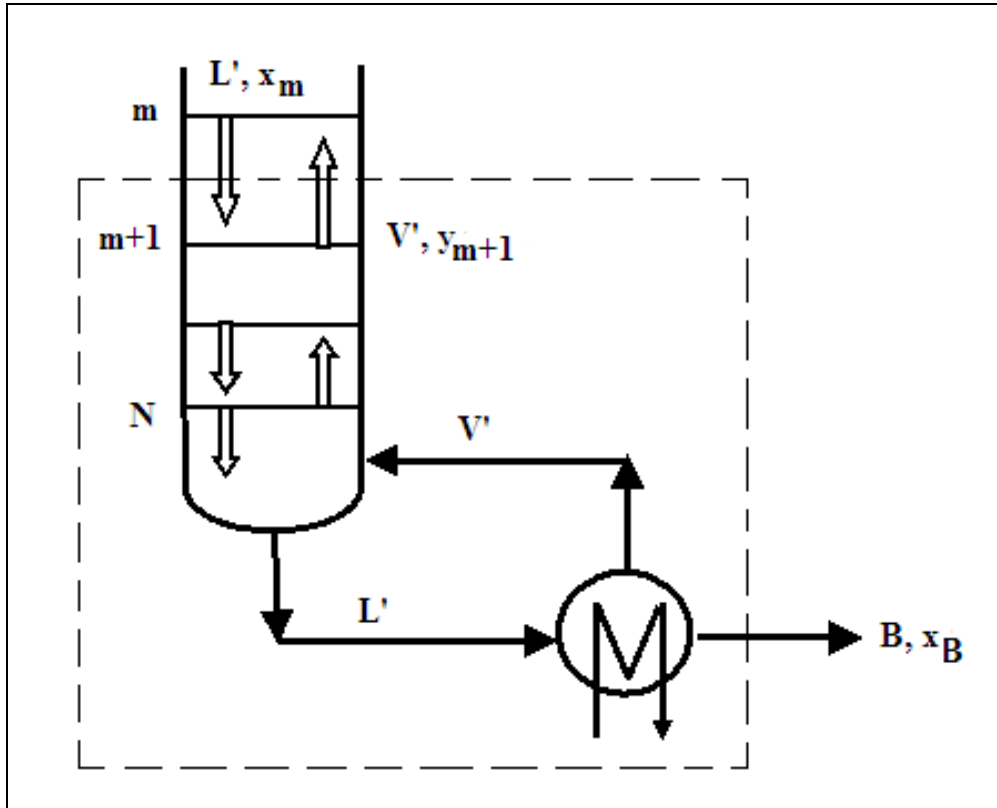


Figure 5.17: Schematic of the stripping section

Consider the constant molal overflow in the column. Thus $L'_m = L'_{m+1} = \dots = L' =$ constant and $V'_m = V'_{m+1} = \dots = V' =$ constant.

Overall material balance gives

$$L' = V' + B \quad (5.29)$$

More volatile component balance gives:

$$L'x_m = V'y_{m+1} + Bx_B \quad (5.30)$$

Substituting and re-arranging the Equation (5.30) yields

$$y_{m+1} = \left(\frac{L'}{V'}\right)x_m - \left(\frac{B}{V'}\right)x_B \quad (5.31)$$

Dropping the subscripts "m+1" and "m" it becomes:

$$y = \left(\frac{L'}{V'}\right)x - \left(\frac{B}{V'}\right)x_B \quad (5.32)$$

Substituting $V' = L' - B$ from Equation (5.29), the Equation (5.32) can be written as:

$$y = \left(\frac{L'}{L' - B}\right)x - \left(\frac{B}{L' - B}\right)x_B \quad (5.33)$$

The Equation (5.33) is called the stripping operating line (SOL) which is a straight line with slope $(L' / L' - B)$ and intercept $(Bx_B / L' - B)$. When $x = x_B$, $y = x_B$, the SOL passes through (x_B, x_B) on the 45° diagonal line.

Determination of number of theoretical stage

Suppose a column is to be designed for the separation of a binary mixture where the feed has a concentration of x_F (mole fraction) of the more volatile component and a distillate having a concentration of x_D of the more volatile component whereas the bottoms having a desired concentration of x_B . Once the three lines (ROL, SOL and q-line) are drawn, the number of theoretical stages required for a given separation is then the number of triangles that can be drawn between these operating lines and the equilibrium curve. The last triangle on the diagram represents the reboiler. A typical representation is given in Figure 5.18.

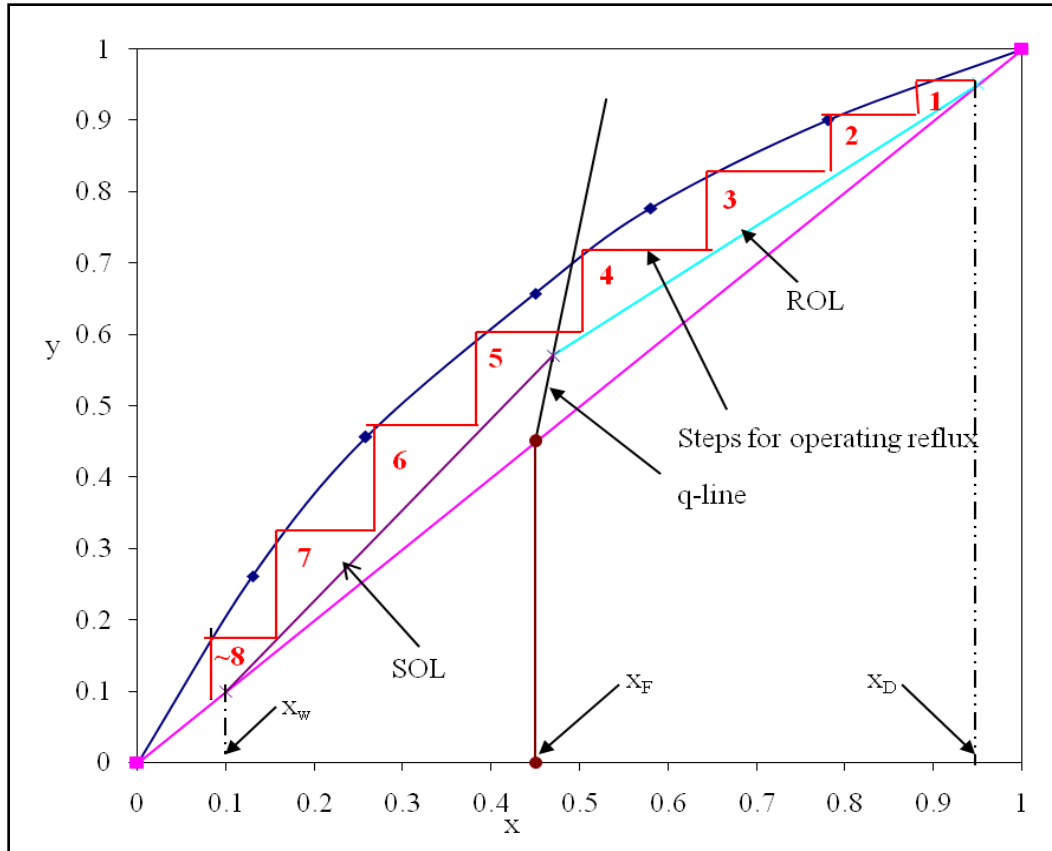


Figure 5.18: A typical representation of identifying number of theoretical stages

Reflux Ratio, R

The separation efficiency by distillation depends on the reflux ratio. For a given separation (i.e. constant x_D and x_B) from a given feed condition (x_F and q), higher reflux ratio (R) results in lesser number of required theoretical trays (N) and vice versa. So there is an inverse relationship between the reflux ratio and the number of theoretical stages. At a specified distillate concentration, x_D , when R changes, the slope and intercept of the ROL changes (Equation (5.19)). From the Equation (5.19), when R increases (with x_D constant), the slope of ROL becomes steeper, i.e. $(R/R+1)$ and the intercept $(x_D/R+1)$ decreases. The ROL therefore rotates around the point (x_D, x_D) . The reflux ratio may be any value between a minimum value and an infinite value. The limit is the minimum reflux ratio (result in infinite stages) and the total reflux or infinite reflux ratio (result in minimum stages). With x_D constant, as R decreases, the slope $(R/R+1)$ of ROL (Equation

(5.19)) decreases, while its intercept ($x_D/R+1$) increases and rotates upwards around (x_D, x_D) as shown in Figure 5.19. The ROL moves closer to the equilibrium curve as R decreases until point Q is reached. Point Q is the point of intersection between the q -line and the equilibrium curve.

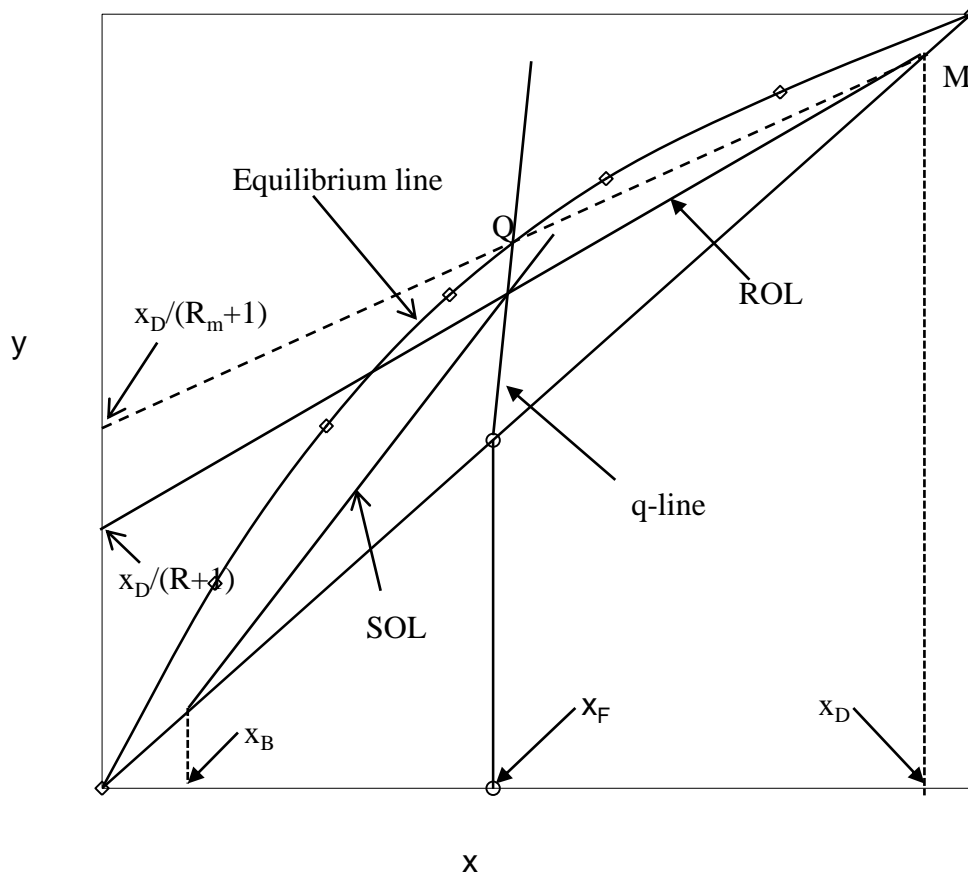


Figure 5.19: Representation of minimum reflux ratio

At this point of intersection the driving force for mass transfer is zero. This is also called as Pinch Point. At this point separation is not possible. The R cannot be reduced beyond this point. The value of R at this point is known as the minimum reflux ratio and is denoted by R_{min} . For non-ideal mixture it is quite common to exhibit inflections in their equilibrium curves as shown in Figure 5.20 (a, b). In those cases, the operating lines where it becomes tangent to the equilibrium curve (called tangent pinch) is the condition for minimum reflux. The ROL cannot

move beyond point P, e.g. to point K. The condition for zero driving force first occurs at point P, before point K which is the intersection point between the q-line and equilibrium curve. Similarly it is the condition SOL also. At the total reflux ratio, the ROL and SOL coincide with the 45 degree diagonal line. At this condition, total number of triangles formed with the equilibrium curve is equal minimum number of theoretical stages. The reflux ratio will be infinite.

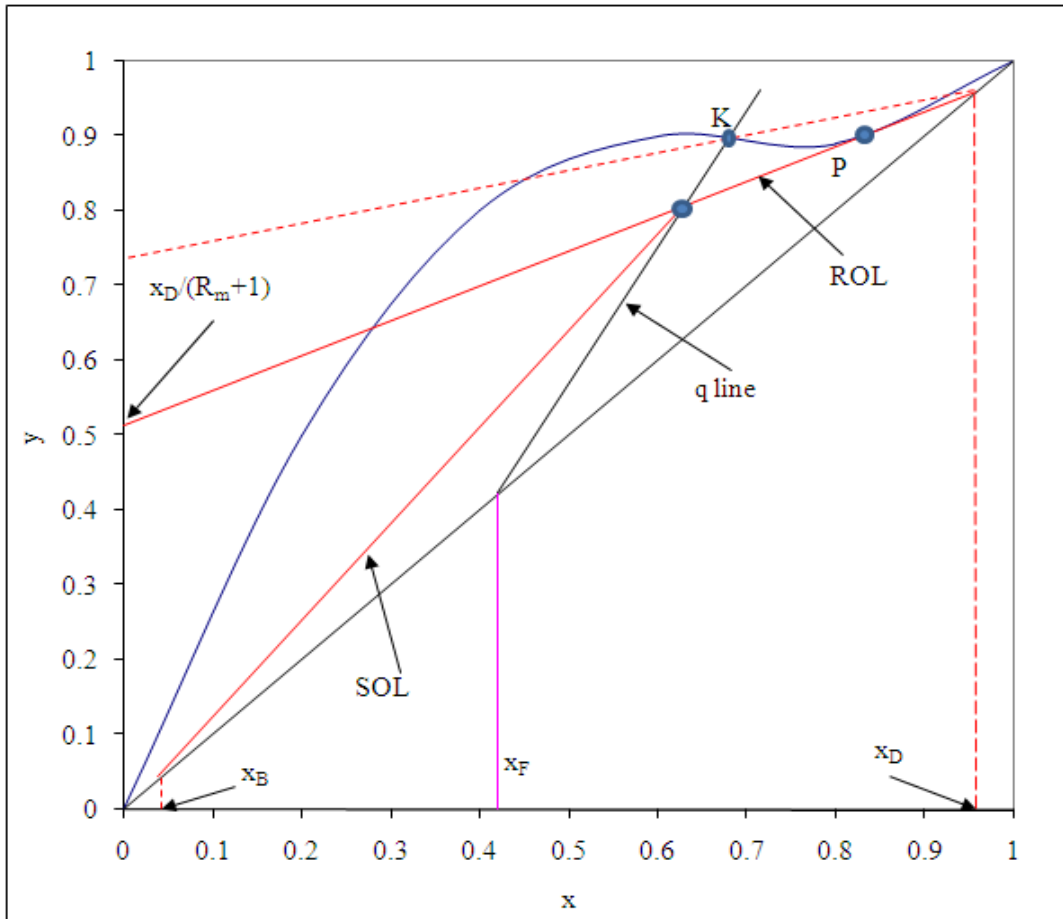


Figure 5.20 (a): Representation of minimum reflux ratio for non-ideal mixture

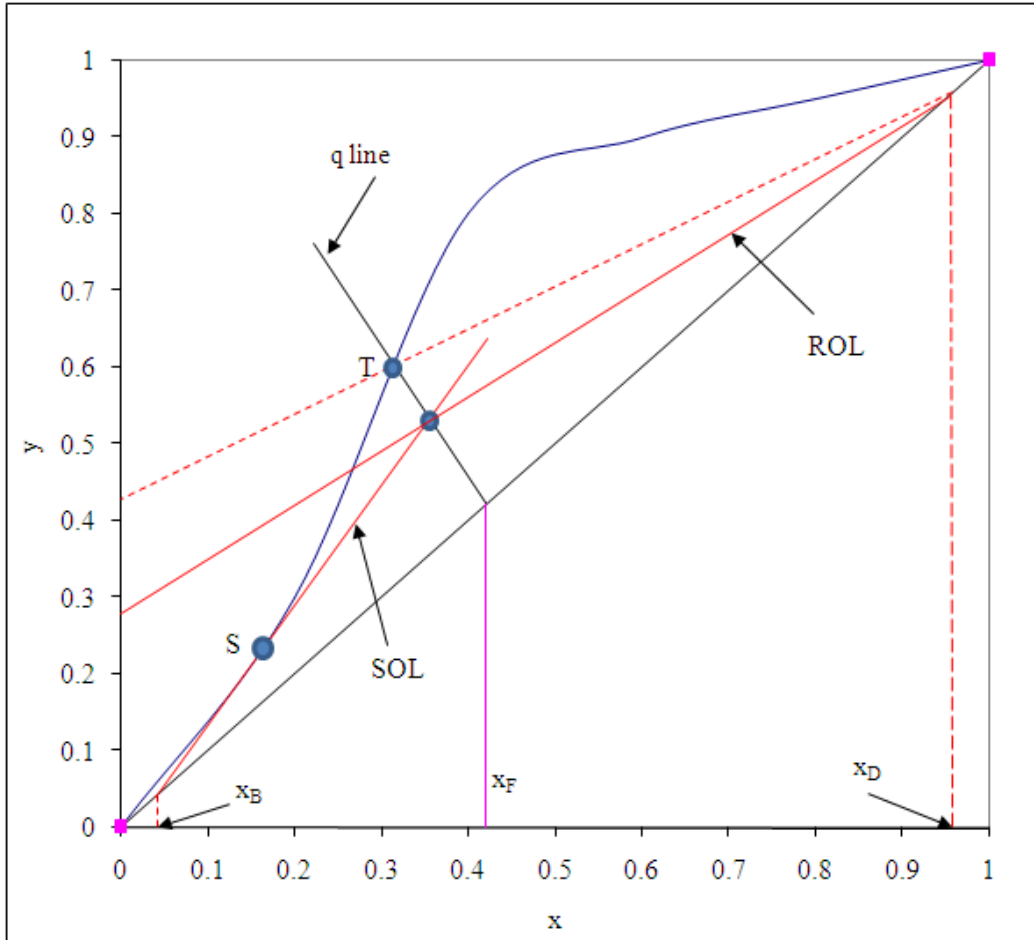


Figure 5.20 (b): Representation of minimum reflux ratio for non-ideal mixture

Tray Efficiency

For the analysis of theoretical stage required for the distillation, it is assumed that the the vapor leaving each tray is in equilibrium with the liquid leaving the same tray and the trays are operating at 100% efficiency. In practice, the trays are not perfect. There are deviations from ideal conditions. The equilibrium with temperature is sometimes reasonable for exothermic chemical reaction but the equilibrium with respect to mass transfer is not often valid. The deviation from the ideal condition is due to: (1) Insufficient time of contact (2) Insufficient degree of mixing. To achieve the same degree of desired separation, more trays will have to be added to compensate for the lack of

perfect separability. The concept of tray efficiency may be used to adjust the the actual number of trays required.

Overall Efficiency

The overall tray efficiency, E_o is defined as:

$$E_o = \frac{\text{No. of theoretical trays}}{\text{No. of actual trays}} \quad (5.34)$$

It is applied for the whole column. Every tray is assumed to have the same efficiency. The overall efficiency depends on the (i) geometry and design of the contacting trays, (ii) flow rates and flow paths of vapor and liquid streams, (iii) Compositions and properties of vapor and liquid streams (Treybal, 1981; Seader and Henley, 1998). The overall efficiency can be calculated from the following correlations:

The Drickamer-Bradford empirical correlation:

$$E_o = 13.3 - 66.8 \log(\mu) \quad (5.35)$$

The correlation is valid for hydrocarbon mixtures in the range of $342 \text{ K} < T < 488.5 \text{ K}$, $1 \text{ atm} < P < 25 \text{ atm}$ and $0.066 < \mu < 0.355 \text{ cP}$

The O'Connell correlation:

$$E_o = 50.3(\alpha\mu)^{-0.226} \quad (5.36)$$

Murphree Efficiency

The efficiency of the tray can also be calculated based on semi-theoretical models which can be interpreted by the Murphree Tray Efficiency E_M . In this case it is assumed that the vapor and liquid between trays are well-mixed and have uniform composition. It is defined for each tray according to the separation

achieved on each tray based on either the liquid phase or the vapor phase. For a given component, it can be expressed as:

Based on vapor phase:

$$E_{MV} = \frac{y_n - y_{n+1}}{y_n^* - y_{n+1}} \quad (5.37)$$

Based on liquid phase:

$$E_{ML} = \frac{x_n - x_{n-1}}{x_n^* - x_{n-1}} \quad (5.38)$$

Example problem 5.2:

A liquid mixture of benzene toluene is being distilled in a fractionating column at 101.3 k Pa pressure. The feed of 100 kmole/h is liquid and it contains 45 mole% benzene (A) and 55 mole% toluene (B) and enters at 327.6 K. A distillate containing 95 mole% benzene and 5 mole% toluene and a bottoms containing 10 mole% benzene and 90 mole% toluene are to be obtained. The amount of liquid is fed back to the column at the top is 4 times the distillate product. The average heat capacity of the feed is 159 KJ/kg mole. K and the average latent heat 32099 kJ/kg moles.

Calculate

- i. The kg moles per hour distillate, kg mole per hour bottoms
- ii. No. of theoretical stages at the operating reflux.
- iii. The minimum no. of theoretical stages required at total reflux
- iv. If the actual no. of stage is 10, what is the overall efficiency increased at operating condition compared to the condition of total reflux?

The equilibrium data:

Temp.(K)	353.3	358.2	363.2	366.7	373.2	378.2	383.8
X _A (mole fraction)	1.000	0.780	0.580	0.450	0.258	0.13	0
Y _A (mole fraction)	1.000	0.900	0.777	0.657	0.456	0.261	0

Solution 5.2:

$$F = D + B$$

$$100 = D + B$$

$$F x_F = D x_D + B x_B$$

Therefore, $D = 41.2$ kg mole/h, $B = 58.8$ kg mole/h

$$y = [R/(R+1)] x + x_D/(R+1) = 0.8 x + 0.190$$

$$q = 1 + cpL (T_B - T_F)/\text{Latent heat of vaporization}$$

$T_B = 366.7$ K from boiling point of feed, $T_F = 327.6$ K (inlet feed temp)

$$\text{Therefore } q = 1.195$$

Slope of q line = 6.12

From the graph (Figure E1), Total no of theoretical stages is 8 at operating reflux (Red color)

From the graph (Figure E1), Total no of theoretical stages is 6 at total reflux (Black color)

Overall efficiency at operating conditions: E_o (Operating) = No of ideal stage/ No of actual trays = $7.9/10 = 0.79$

Overall efficiency at total reflux conditions: E_o (total reflux) = No of ideal stage/ No of actual trays = $5.9/10 = 0.59$

Overall efficiency increased: $0.79 - 0.59 = 0.20$

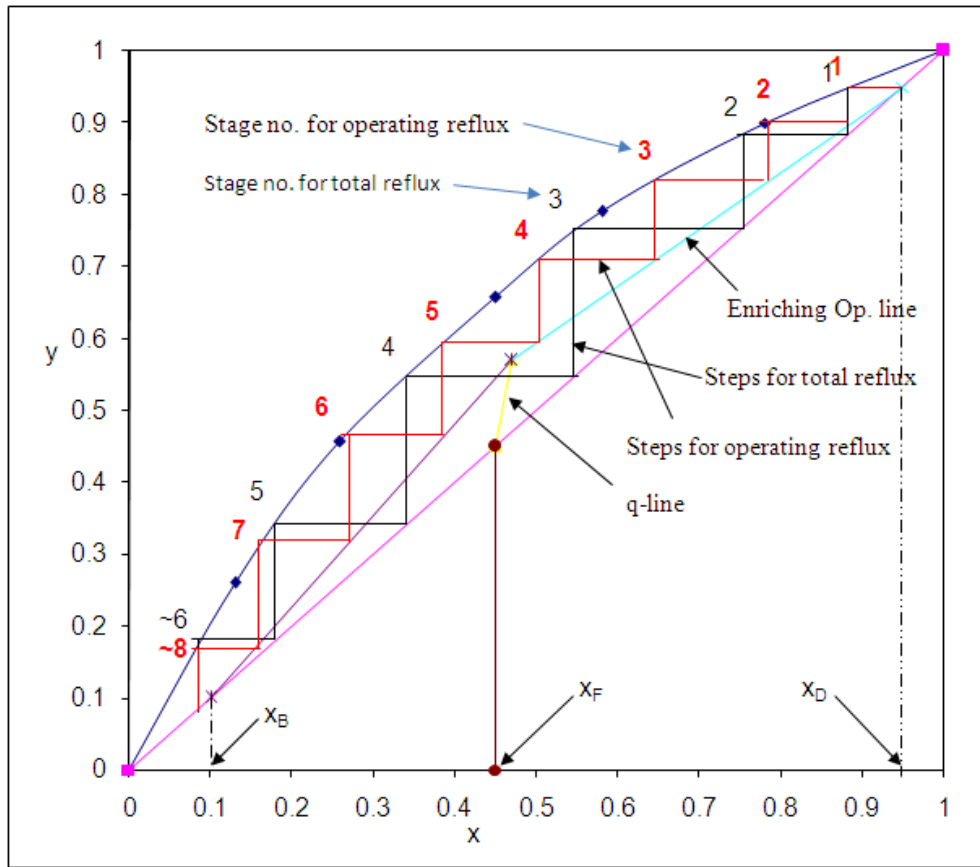


Figure E1: Graph representing the example problem 5.2.

MODULE 5: DISTILLATION

LECTURE NO. 6

5.2.2.4. Analysis of binary distillation by Ponchon-Savarit Method

Background Principle:

The method is concerned with the graphical analysis of calculating the theoretical stages by enthalpy balance required for desired separation by distillation process (Hines and Maddox, 1984). In this method, the enthalpy balances are incorporated as an integral part of the calculation however it is not considered in the analysis separation by distillation process by McCabe-Thele method. This procedure combines the material balance calculations with enthalpy balance calculations. This method also provides the information on the condenser and reboiler duties. The overall material balance for the distillation column is as shown in Figure 5.11:

$$F = D + B \quad (5.39)$$

For any component the material balance around the column can be written as:

$$Fx_F = Dx_D + Bx_B \quad (5.40)$$

Overall enthalpy balance for the column yields

$$FH_F + Q_R = DH_D + BH_B + Q_C \quad (5.41)$$

where H is the enthalpy of the liquid stream, energy/mol, Q_R is the heat input to the reboiler, J/s and Q_C is the heat removed from condenser, J/s. The heat balance on rearrangement of Equation (5.41) gives

$$FH_F = D\left(H_D + \frac{Q_C}{D}\right) + B\left(H_B - \frac{Q_R}{R}\right) \quad (5.42)$$

The Equation (5.41) is rearranged as Equation (5.42) for the convenience to plot it on the enthalpy-concentration diagram. The points represented by the feed, distillate and bottom streams can be plotted on the enthalpy-concentration diagram as shown in Figure 5.21.

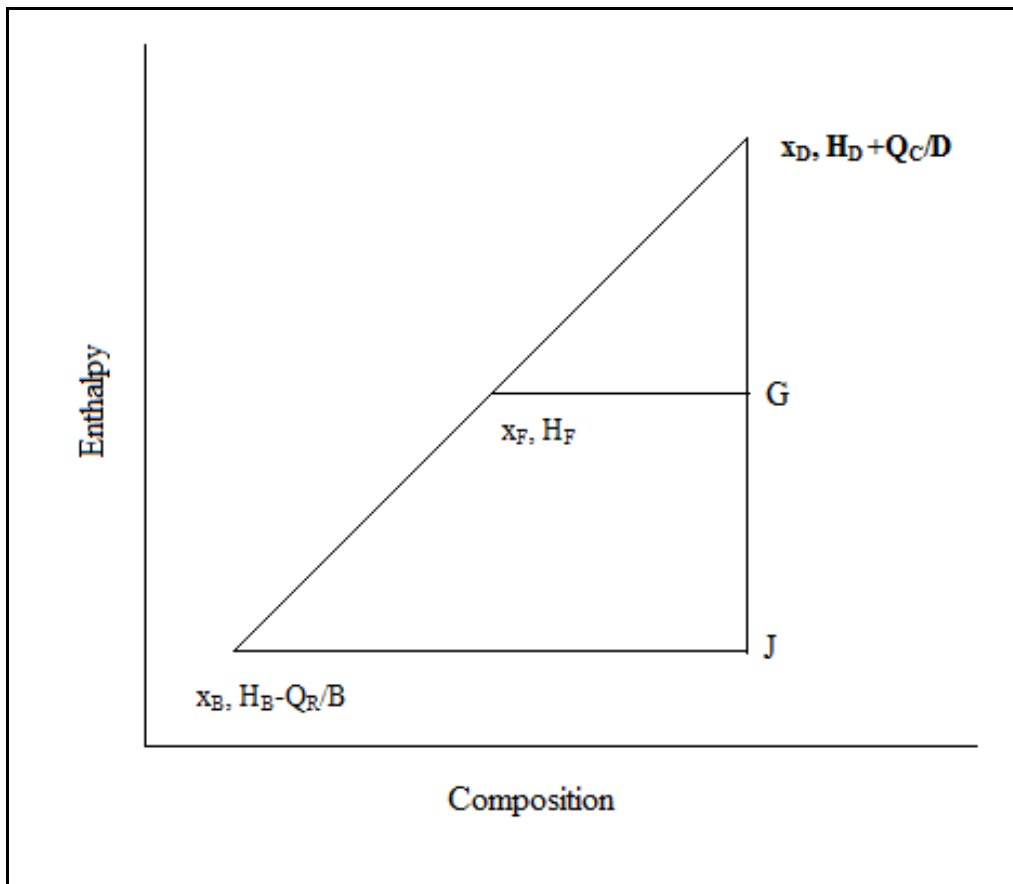


Figure 5.21: Representation of feed, distillate and bottom streams on the enthalpy-concentration

Substituting the Equation (5.39) into the Equation (5.40) and (5.42) we get

$$(D + B)x_F = Dx_D + Bx_B \quad (5.43)$$

$$(D + B)H_F = D\left(H_D + \frac{Q_C}{D}\right) + B\left(H_B - \frac{Q_R}{R}\right) \quad (5.44)$$

From the Equations (5.43) and (5.44) one can write

$$\frac{D}{B} = \frac{x_F - x_B}{x_D - x_F} = \frac{H_F - \left(H_B - \frac{Q_R}{R}\right)}{\left(H_D + \frac{Q_C}{D}\right) - H_F} \quad (5.45)$$

The Equation (5.45) can also be written on rearrangement as:

$$\frac{x_F - x_B}{H_F - \left(H_B - \frac{Q_R}{R}\right)} = \frac{x_D - x_F}{\left(H_D + \frac{Q_C}{D}\right) - H_F} \quad (5.46)$$

Comparing the Equation (5.46) with the points plotted on Figure (5.21), it is found that the left hand side of the Equation (5.46) represents the slope of the straight line between the points $(x_B, H_B - Q_R/B)$ and (x_F, H_F) . The right hand side of the Equation (5.46) represents the slope of the straight line passing through the points (x_F, H_F) and $(x_D, H_D - Q_C/D)$. From this it can be said that all three points are on a same straight line. The amount of distillate as per Equation (5.45) is proportional to the horizontal distance $\overline{x_F x_B}$ and the amount of bottoms is proportional to the horizontal distance $\overline{x_D x_F}$, then from the overall material balance it can be interpreted that the amount of feed is proportional to the horizontal distance $\overline{x_D x_B}$. This leads to the inverse lever rule. Based on this principle, the analysis of distillation column is called Enthalpy-composition analysis or Ponchon-Savarit analysis of distillation column.

Analysis of tray column

Consider the theoretical stage shown in Figure 5.12 and the principle by which it operates is described in the enthalpy-composition diagram as shown in Figure 5.22.

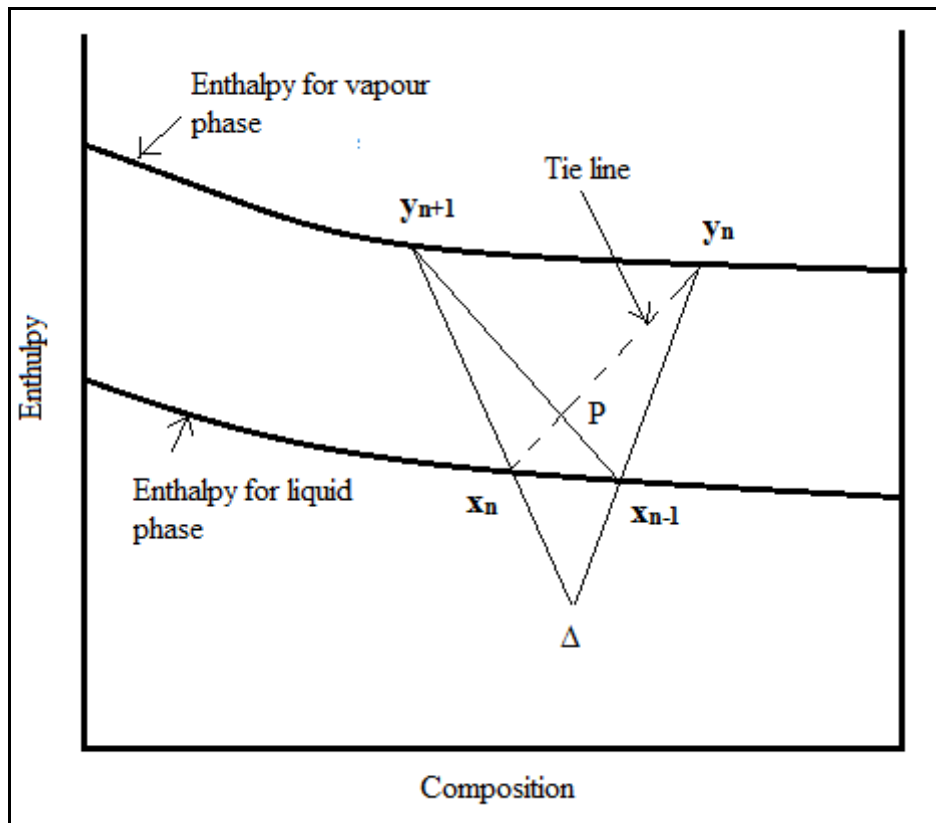


Figure 5.22: Operation principle of stage-wise binary distillation on enthalpy-concentration diagram

The vapor entering to the tray (n), V_{n+1} is a saturated vapor of composition y_{n+1} whereas the liquid entering to the tray is L_{n-1} of composition x_{n-1} . The point P in the Figure 5.22 represents the total flow to the tray. The point P lies in a straight line joining x_{n-1} and y_{n+1} . The distance $\overline{y_{n+1}P}$ is proportional to the quantity L_{n-1} and the distance $\overline{x_{n-1}P}$ is proportional to the quantity V_{n+1} as per lever rule. So

$$\frac{L_{n-1}}{V_{n+1}} = \frac{y_{n+1}P}{x_{n-1}P} \quad (5.47)$$

The sum of the liquid and vapor leaving the plate must equal the total flow to the plate. So the tie line must pass through the point P which represents the addition point of the vapor V_n and liquid L_n leaving the tray. The intersection of the tie line with the enthalpy-composition curves will represent the compositions of these streams.

Around the tray n, the material balances can be written as:

$$V_{n+1} - L_n = V_n - L_{n-1} \quad (5.48)$$

$$V_{n+1}y_{n+1} - L_nx_n = V_ny_n - L_{n-1}x_{n-1} \quad (5.49)$$

The enthalpy balance around the tray gives

$$V_{n+1}H_{V,n+1} - L_nH_{L,n} = V_nH_{V,n} - L_{n-1}H_{L,n-1} \quad (5.50)$$

Where H_V is the enthalpy of the vapor and H_L is the enthalpy of the liquid. From the material and enthalpy balance Equations above mean that the stream that added to V_{n+1} to generate L_n is same to the stream that must be added to V_n to generate L_{n-1} . In the Figure 5.22, Δ represents the difference point above and below the tray. This point of a common difference point can be extended to a section of a column that contains any number of theoretical trays.

MODULE 5: DISTILLATION

LECTURE NO. 7

Stepwise procedure to determine the number of theoretical trays:

Step 1: Draw the equilibrium curve and the enthalpy concentration diagram for the mixture to be separated

Step 2: Calculate the compositions of the feed, distillate and bottom products. Locate these compositions on the enthalpy-concentration diagram.

Step 3: Estimate the reflux rate for the separation and locate the rectifying section difference point as Δ_R as shown in Figure 5.23. Point y_1 is the intersection point of line joining point x_D and Δ_R and H_{V-y} curve.

Step 4: Locate the stripping section difference point Δ_S . The point Δ_S is to be located at a point where the line from Δ_R through x_F intersects the x_B composition coordinate as shown in Figure 4.23.

Step 5: Step off the trays graphically for the rectifying section. Then the point of composition x_1 of liquid of top tray is to be determined from the equilibrium relation with y_1 of vapor which is leaving the tray and locate it to the H_{L-x} curve. Then the composition y_2 is to be located at the point where the line of points Δ_R and x_1 intersects H_{V-y} curve. This procedure is to be continued until the feed plate is reached.

Step 6: Similarly follow the same rule for stripping section. In the stripping section, the vapor composition y_B leaving the reboiler is to be estimated from the equilibrium relation. Then join the y_B and Δ_S to find the x_N . The vapor composition

y_N is to be determined by extending a tie line to saturated vapor curve H_{V-y} . The procedure is to be continued until the feed tray is attained.

Determination of the reflux rate:

The reflux rate can be calculated from the energy balance around the condenser as:

$$V_1 H_{V,1} = L_D H_D + D H_D + Q_C \quad (5.51)$$

By substituting $V_1 = L_D + D$ into Equation (5.51) and rearranging, it can be written as:

$$\frac{L_D}{D} = \frac{(H_D + Q_C / D) - H_{V,1}}{H_{V,1} - H_D} = \frac{\overline{\Delta_R H_{V,1}}}{\overline{H_{V,1} H_D}} \quad (5.52)$$

Where $\overline{\Delta_R H_{V,1}}$ and $\overline{H_{V,1} H_D}$ are the lengths of lines between points Δ_R and $H_{V,1}$ and $H_{V,1}$ and H_D . The internal reflux ratio between any two stages in rectifying section can be expressed as:

$$\frac{L_n}{V_{n+1}} = \frac{(H_D + Q_C / D) - H_{V,n+1}}{(H_D + Q_C / D) - H_{L,n}} = \frac{\overline{\Delta_R H_{V,n+1}}}{\overline{\Delta_R H_{L,n}}} \quad (5.53)$$

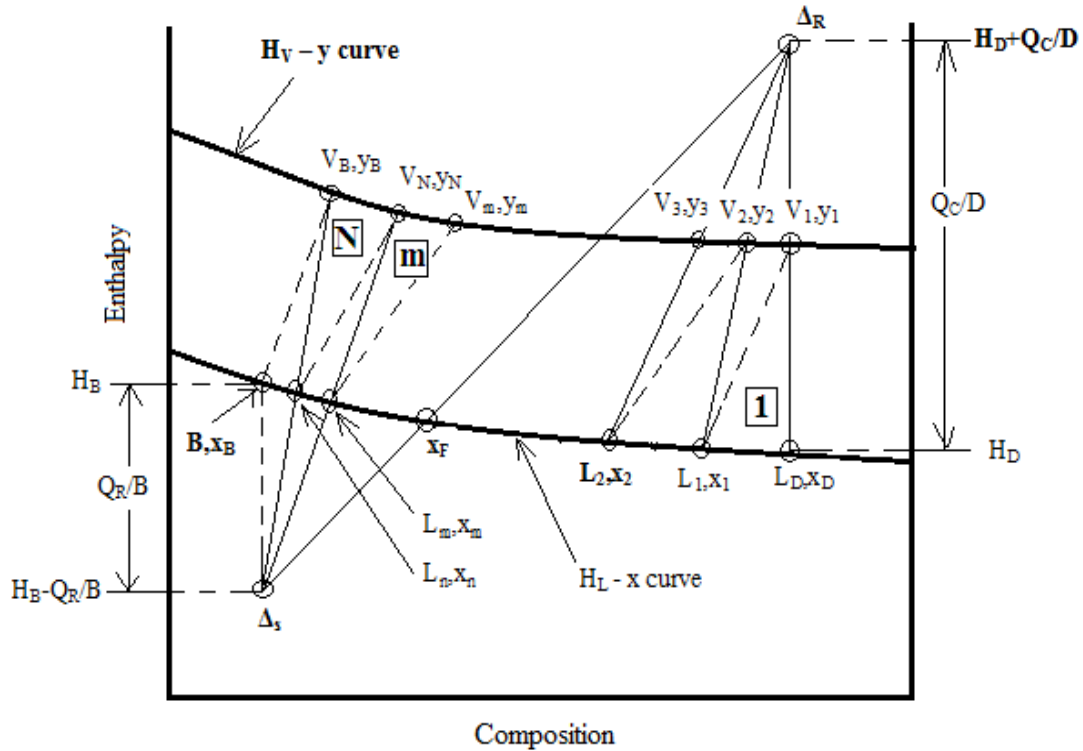


Figure 5.23: Representation of estimation of no of stages by Ponchon-Savarit Method

Whereas in the stripping section it can be expressed as:

$$\frac{L_{m-1}}{V_m} = \frac{H_{V,m} - (H_B - Q_R/B)}{H_{L,m-1} - (H_B - Q_R/B)} \quad (5.54)$$

The relationship between the distillate and bottom products in terms of compositions and enthalpies can be made from the material balance around the overall column which can be written as:

$$FH_F + Q_R = DH_D + BH_B + Q_C \quad (5.55)$$

Overall material balance $F = D + B$ combining with Equation (5.55) yields

$$\frac{D}{B} = \frac{H_F - (H_B - Q_R/B)}{(H_D + Q_C/D) - H_F} = \frac{x_F \Delta_S}{x_F \Delta_R} \quad (5.56)$$

Minimum number of trays

In this method, if D approaches zero, the enthalpy coordinate ($H_D + Q_C/D$) of the difference point approaches infinity. Other way it can be said that Q_C becomes large if L becomes very large with respect to D . Similarly enthalpy coordinate for stripping section becomes negative infinity as B approaches zero or liquid loading in the column becomes very large with respect to B . Then the difference points will locate at infinity. In such conditions, the trays required for the desired separation is referred as minimum number of trays. The thermal state of the feed has no effect on the minimum number of trays required for desired separation.

Minimum reflux

The minimum reflux for the process normally occurs at the feed tray. The minimum reflux rate for a specified separation can be obtained by extending the tie line through the feed composition to intersect a vertical line drawn through x_D . Extend the line to intersect the x_B composition line determines the boilup rate and the reboiler heat duty at minimum reflux.

Example problem 5.3:

A total of 100 gm-mol feed containing 40 mole percent n-hexane and 60 percent n-octane is fed per hour to be separated at one atm to give a distillate that contains 92 percent hexane and the bottoms 7 percent hexane. A total condenser is to be used and the reflux will be returned to the column as a saturated liquid at its bubble point. A reflux ratio of 1.5 is maintained. The feed is introduced into the column as a saturated liquid at its bubble point. Use the Ponchon-Savarit method and determine the following:

- (i) Minimum number of theoretical stages
- (ii) The minimum reflux ratio
- (iii) The heat loads of the condenser and reboiler for the condition of minimum reflux.

- (iv) The quantities of the distillate and bottom streams using the actual reflux ratio.
- (v) Actual number of theoretical stages
- (vi) The heat load of the condenser for the actual reflux ratio
- (vii) The internal reflux ratio between the first and second stages from the top of tower.

VLE Data, Mole Fraction Hexane, 1 atm

x	0	0.1	0.3	0.5	0.55	0.7	1
y	0	0.36	0.7	0.85	0.9	0.95	1

Enthalpy-Concentration Data

Mole fraction, Hexane		0	0.1	0.3	0.5	0.7	0.9	1
Enthalpy, Cal/gm-mol	Sat. Liquid	7000	6300	5000	4100	3400	3100	3000
	Sat. Vapor	15,700	15,400	14,700	13,900	12,900	11,600	10,000

Solution 5.3:

(i) The minimum number of stages can be obtained by drawing vertical operating lines that intersects as Δ_R tends to infinity. As shown in Figure E2, three stages required.

(ii) Using Equation (5.52), the minimum reflux is

$$\frac{L_D}{D} = \frac{(H_D + Q_C / D) - H_{V,1}}{H_{V,1} - H_D} = \frac{\Delta_R H_{V,1}}{H_{V,1} H_D} = \frac{19000 - 11500}{11500 - 3000} = 0.88$$

(iii) From the Figure E2, $H_B - Q_R / B = -5000$

$$Q_R / B = 5000 + 7000 = 12000$$

$$H_D + Q_C / D = 19000$$

$$Q_C / D = 19000 - 11500 = 7500$$

(iv) The distillate and bottom flowrates are obtained by solving the overall and component material balances simultaneously

$$100 = D + B$$

$$0.4 (1000) = 0.92 D + 0.04 B$$

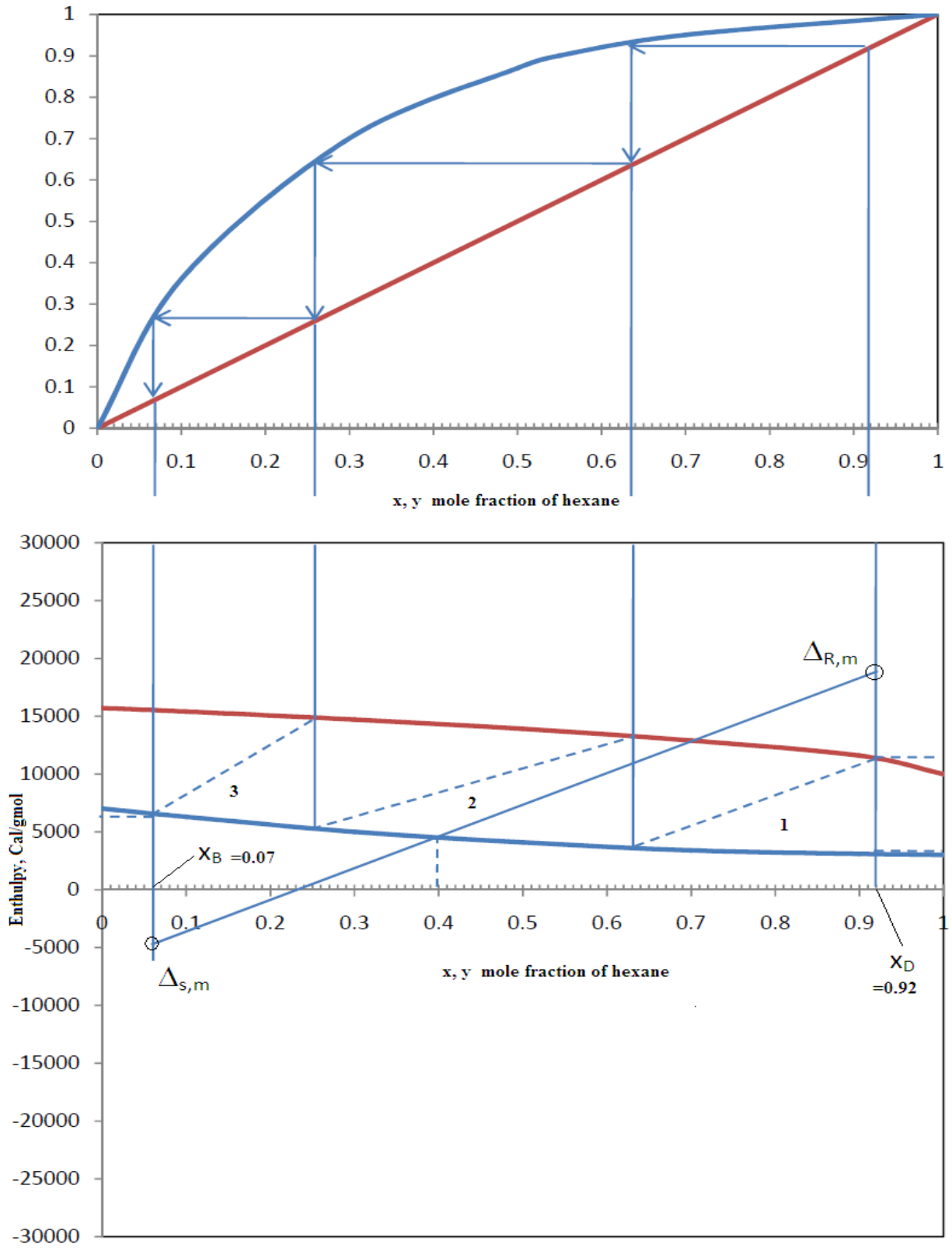


Figure E2: Minimum reflux and minimum stages for example problem 4.3

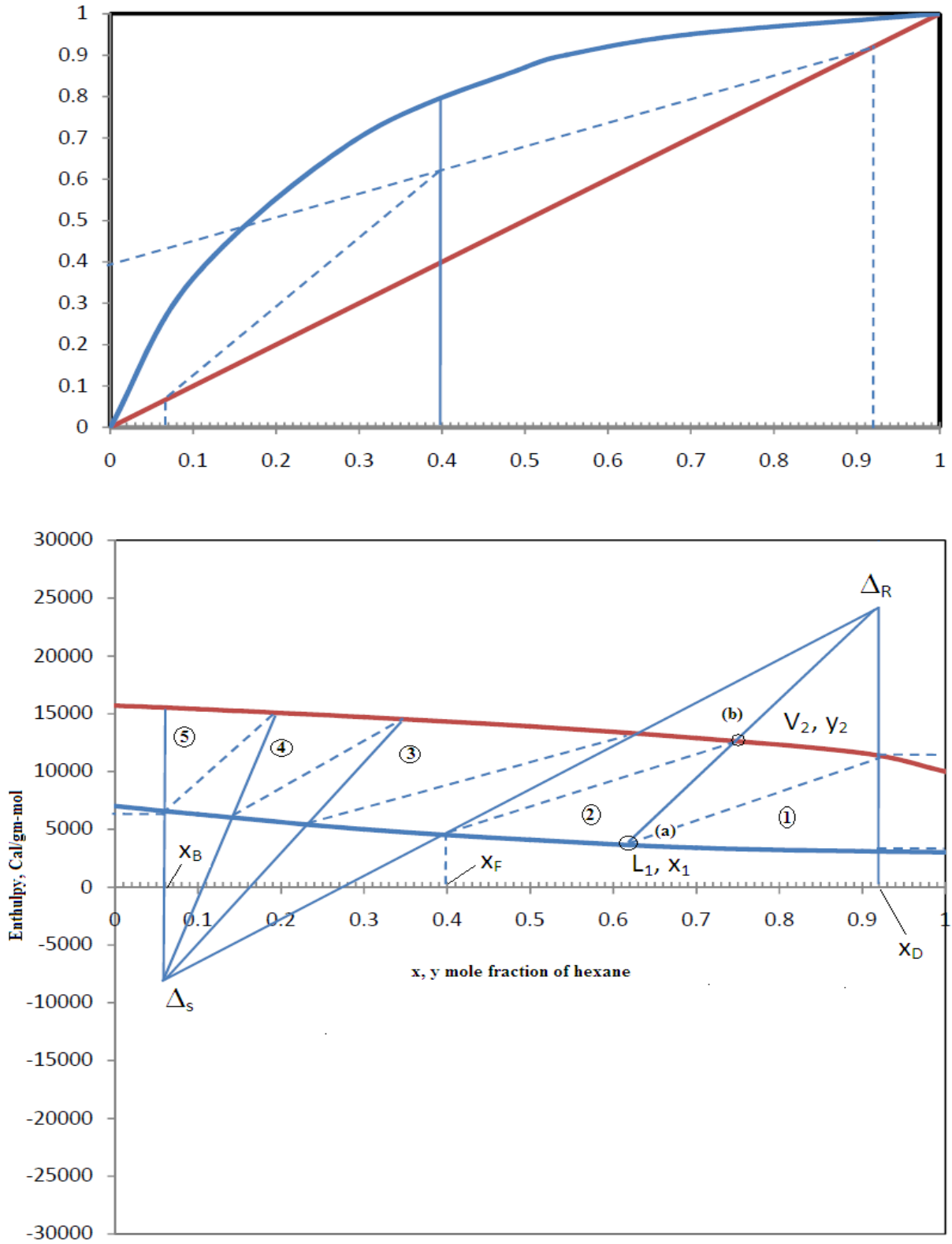


Figure E3: Actual stages for Example problem 4.3.

Thus $D = 40.9$ gmmol/h

$B = 100 - 40.9 = 59.1$ gmmol/h

$$(v) \left(\frac{L_D}{D} \right)_{act} = 1.5 \left(\frac{L_D}{D} \right)_{min} = 1.5(0.88) = 1.32$$

Using Equation (5.52) gives a new value for Δ_R :

$$1.32 = \frac{(H_D + Q_C / D) - 11500}{11500 - 3000} \quad \text{Or, } H_D + Q_C / D = 22750$$

A new Δ_R is located in Figure E3 and the actual number of theoretical stages is found as 5.

(vi) From the Figure E3

$$H_D + Q_C / D = 22750$$

So $Q_C / D = 22750 - 11500 = 11250$

(vii) Reading enthalpies for the points (a) and (b) and using Equation (5.53) gives

$$\frac{L_n}{V_{n+1}} = \frac{(H_D + Q_C / D) - H_{V,n+1}}{(H_D + Q_C / D) - H_{L,n}} = \frac{22750 - 12050}{22750 - 3750} = 0.563$$

MODULE 5: DISTILLATION

LECTURE NO. 8

5.3. Introduction to Multicomponent Distillation

In industry, most of the distillation processes involve with more than two components. The multicomponent separations are carried out by using the same type of distillation columns, reboilers, condensers, heat exchangers and so on. However some fundamental differences are there which is to be thoroughly understood by the designer. These differences are from phase rule to specify the thermodynamic conditions of a stream at equilibrium. In multicomponent systems, the same degree of freedom is not achieved because of the presence of other components. Neither the distillate nor the bottoms composition is completely specified. The components that have their distillate and bottoms fractional recoveries specified are called key components. The most volatile of the keys is called the light key (LK) and the least volatile is called the heavy key (HK). The other components are called non-keys (NK). Light non-key (LNK) is referred when non-key is more volatile than the light key whereas heavy non-key (HNK) is less volatile than the heavy key. Proper selection of key components is important if a multicomponent separation is adequately specified. Several short-cut methods are used for carrying out calculations in multicomponent systems. These involve generally an estimation of the minimum number of trays, the estimation of minimum reflux rate and number of stages at finite reflux for simple fractionators. Although rigorous computer methods are available to solve multicomponent separation problems, approximate methods are used in practice. A widely used approximate method is commonly referred to as the Fenske-Underwood-Gilliland method.

5.3.1. Estimation of Minimum number of trays: Fenske Equation

Fenske (1932) was the first to derive an Equation to calculate minimum number of trays for multicomponent distillation at total reflux. The derivation was based on the assumptions that the stages are equilibrium stages. Consider a multicomponent distillation column operating at total reflux as shown in Figure 5.24.

Equilibrium relation for the light key component on the top tray is

$$y_1 = K_1 x_1 \quad (5.57)$$

For total condenser, $y_1 = x_D$ then

$$x_D = K_1 x_1 \quad (5.58)$$

An overall material balance below the top tray and around the top of the column can be written as:

$$V_2 = L_1 + D \quad (5.59)$$

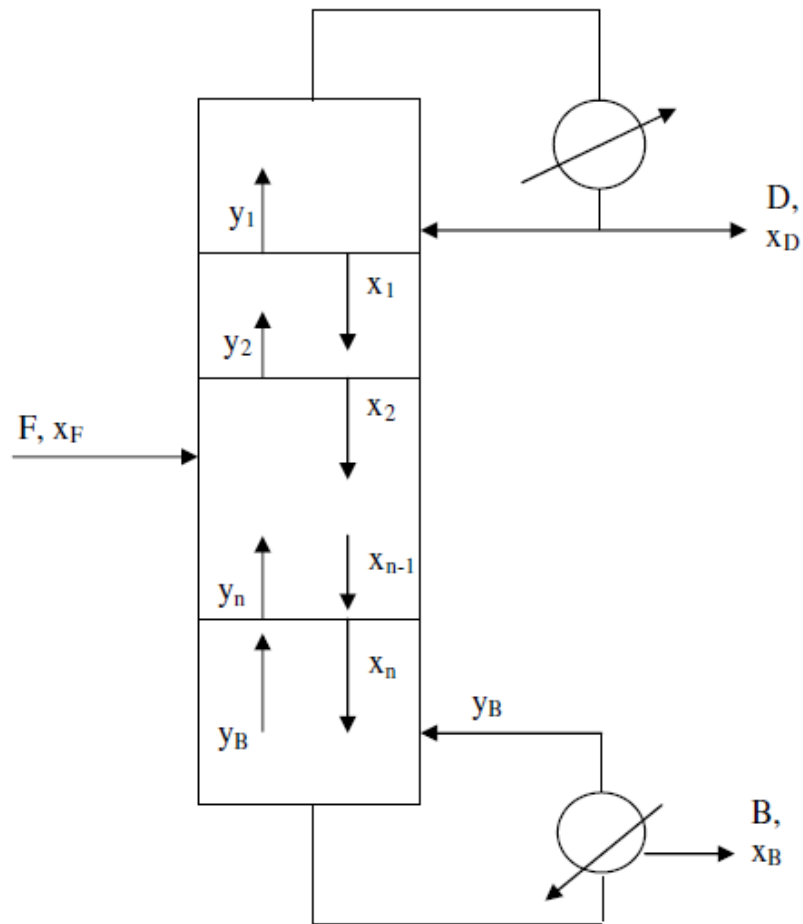


Figure 5.24: Multicomponent column at minimum trays

Under total reflux condition, $D = 0$, thus, $V_2 = L_1$. The component material balance for the light key component around the first plate and the top of the column is

$$V_2 y_2 = L_1 x_1 + D x_D \quad (5.60)$$

Then under the conditions of the minimum trays, the Equation (5.60) yields $y_2 = x_1$. The equilibrium relation for plate 2 is

$$y_2 = K_2 x_2 \quad (5.61)$$

The Equation (5.61) becomes at $y_2 = x_1$

$$x_1 = K_2 x_2 \quad (5.62)$$

Substituting Equation (5.62) into Equation (5.58) yields

$$x_D = K_1 K_2 x_2 \quad (5.63)$$

Continuing this calculation for entire column, it can be written as:

$$x_D = K_1 K_2 \dots\dots\dots K_n K_B x_B \quad (5.64)$$

In the same fashion, for the heavy key component, it can be written as:

$$x'_D = K'_1 K'_2 \dots\dots\dots K'_n K'_B x'_B \quad (5.65)$$

Equation (5.64) upon Equation (5.65) gives

$$\frac{x_D}{x'_D} = \frac{K_1 K_2 \dots\dots\dots K_n K_B x_B}{K'_1 K'_2 \dots\dots\dots K'_n K'_B x'_B} \quad (5.66)$$

The ratio of the K values is equal to the relative volatility, thus the Equation (5.66) can be written as

$$\frac{x_D}{x'_D} = \alpha_1 \alpha_2 \dots\dots\dots \alpha_n \alpha_B \frac{x_B}{x'_B} \quad (5.67)$$

If average value of the relative volatility applies for all trays and under condition of minimum trays, the Equation (5.67) can be written as

$$\frac{x_D}{x'_D} = \alpha_{avg}^{N_{min}} \frac{x_B}{x'_B} \quad (5.68)$$

Solving Equation (5.68) for minimum number of trays, N_{min}

$$N_{\min} = \frac{\ln\left(\frac{x_D x'_B}{x'_D x_B}\right)}{\ln \alpha_{ave}} \quad (5.70)$$

The Equation (5.70) is a form of the Fenske Equation. In this Equation, N_{\min} is the number of equilibrium trays required at total reflux including the partial reboiler. An alternative form of the Fenske Equation can be easily derived for multi-component

calculations which can be written as
$$N_{\min} = \frac{\ln\left(\frac{(Dx_D)(Bx'_B)}{(Dx'_D)(Bx_B)}\right)}{\ln \alpha_{ave}} \quad (5.71)$$

The amount of light key recovered in the distillate is (Dx_D) . This is equal to the fractional recovery of light key in the distillate say FR_D times the amount of light key in the feed which can be expressed as:

$$Dx_D = (FR_D)Fx_{LK} \quad (5.72)$$

From the definition of the fractional recovery one can write

$$Bx_B = (1 - FR_D)Fx_{LK} \quad (5.73)$$

Substituting Equations (5.72) and (5.73) and the corresponding Equations for heavy key into Equation (5.71) yields

$$N_{\min} = \frac{\ln\left(\frac{(FR_D)(FR'_B)}{(1 - FR_D)(1 - FR'_B)}\right)}{\ln \alpha_{ave}} \quad (5.74)$$

Once the minimum number of theoretical trays, N_{\min} is known, the fractional recovery of the non-keys can be found by writing Equation (5.74) for a non-key component and either the light or heavy key. Then solve the Equation for $FR_{NK,B}$ or $FR_{NK,D}$. If the key component is chosen as light key, then $FR_{NK,D}$ can be expressed as

$$FR_{NK,D} = \frac{\alpha_{NK-LK}^{N_{\min}}}{\frac{FR'_B}{1 - FR'_B} + \alpha_{NK-LK}^{N_{\min}}} \quad (5.75)$$

4.3.2. Minimum Reflux: Underwood Equations

For multi-component systems, if one or more of the components appear in only one of the products, there occur separate pinch points in both the stripping and rectifying sections. In this case, Underwood developed an alternative analysis to find the minimum reflux ratio (Wankat, 1988). The presence of non-distributing heavy non-keys results a pinch point of constant composition at minimum reflux in the rectifying section whereas the presence of non-distributing light non-keys, a pinch point will occur in the stripping section. Let us consider the pinch point is in the rectifying section. The mass balance for component i around the top portion of the rectifying section as illustrated in Figure 5.24 is

$$V_{\min} y_{i,n+1} = L_{\min} x_{i,n} + Dx_{i,D} \quad (5.76)$$

The compositions are constant at the pinch point then

$$x_{i,n-1} = x_{i,n} = x_{i,n+1} \quad (5.77)$$

and

$$y_{i,n-1} = y_{i,n} = y_{i,n+1} \quad (5.78)$$

The equilibrium relation can be written as

$$y_{i,n+1} = m_i x_{i,n+1} \quad (5.79)$$

From the Equations (5.76) to (5.79) a balance in the region of constant composition can be written as

$$V_{\min} y_{i,n+1} = \frac{L_{\min}}{m_i} y_{i,n+1} + Dx_{i,D} \quad (5.80)$$

Defining the relative volatility $\alpha_i = m_i/m_{HK}$ and substituting in Equation (5.80) one can express after rearranging as

$$V_{\min} y_{i,n+1} = \frac{\alpha_i D x_{i,D}}{\alpha_i - \frac{L_{\min}}{V_{\min} m_{HK}}} \quad (5.81)$$

The total vapor flow in the rectifying section at minimum reflux can be obtained by summing Equation (5.81) over all components as:

$$V_{\min} = \sum_i V_{\min} y_{i,n+1} = \sum_i \frac{\alpha_i D x_{i,D}}{\alpha_i - \frac{L_{\min}}{V_{\min} m_{HK}}} \quad (5.82)$$

Similarly after analysis for the stripping section, one can get

$$-V_{st,\min} = \sum_i \frac{\alpha_{i,st} B x_{i,B}}{\alpha_{i,st} - \frac{L_{st,\min}}{V_{st,\min} m_{HK,st}}} \quad (5.83)$$

Defining

$$\lambda_1 = \frac{L_{\min}}{V_{\min} m_{HK}} \quad \text{and} \quad \lambda_2 = \frac{L_{st,\min}}{V_{st,\min} m_{HK,st}} \quad (5.84)$$

Equations (5.82) and (5.83) then become

$$V_{\min} = \sum_i \frac{\alpha_i D x_{i,D}}{\alpha_i - \lambda_1} \quad (5.85)$$

$$\text{and} \quad -V_{st,\min} = \sum_i \frac{\alpha_i B x_{i,B}}{\alpha_i - \lambda_2} \quad (5.86)$$

For constant molar overflow and constant relative volatilities, $\lambda_1 = \lambda_2 = \lambda$ that satisfies both Equations (5.86). The change in vapor flow at the feed stage (ΔV_F) is then written as by adding the Equations (5.86)

$$\Delta V_F = V_{\min} - V_{st,\min} = \sum_i \left[\frac{\alpha_i D x_{i,D}}{\alpha_i - \lambda} + \frac{\alpha_i B x_{i,B}}{\alpha_i - \lambda} \right] \quad (5.87)$$

Combining (5.87) with the overall column mass balance for component i can be expressed as

$$\Delta V_F = \sum_i \left[\frac{\alpha_i F x_{f,i}}{\alpha_i - \lambda} \right] \quad (5.88)$$

Again if the fraction q is known, the change in vapor flow at the feed stage can be expressed as

$$\Delta V_F = F(1 - q) \quad (5.89)$$

Comparing Equation (5.88) and (5.89)

$$(1 - q) = \sum_i \left[\frac{\alpha_i x_{f,i}}{\alpha_i - \lambda} \right] \quad (5.90)$$

Equation (5.90) is known as the first Underwood Equation which is used to calculate appropriate values of λ . whereas Equation (5.86) is known as the second Underwood Equation which is used to calculate V_m . From the mass balance L_m can be calculated as

$$L_{\min} = V_{\min} - D \quad (5.91)$$

4.3.3. Estimation of Numbers of Stages at Finite Reflux: Gilliland Correlation

Gilliland (1940) developed an empirical correlation to relate the number of stages N at a finite reflux ratio L/D to the minimum number of stages and to the minimum reflux ratio. Gilliland represented correlation graphically with $(N - N_{\min})/(N + 1)$ as y-axis and $(R - R_{\min})/(R + 1)$ as x-axis. Later Molokanov et al. (1972) represented the Gilliland correlation as:

$$\frac{(N - N_{\min})}{N + 1} = 1 - \exp \left[\left(\frac{1 + 54.4(R - R_{\min})/(R + 1)}{11 + 117.2(R - R_{\min})/(R + 1)} \right) \left(\frac{(R - R_{\min})/(R + 1) - 1}{[(R - R_{\min})/(R + 1)]^{0.5}} \right) \right] \quad (5.92)$$

According to Seader and Henley (1998), an approximate optimum feed-stage location, can be obtained by using the empirical Equation of Kirkbride (1944) as

$$\frac{N_R}{N_S} = \left[\left(\frac{x_{HK,F}}{x_{LK,F}} \right) \left(\frac{x_{LK,B}}{x_{HK,D}} \right)^2 \left(\frac{B}{D} \right) \right]^{0.206} \quad (5.93)$$

where N_R and N_S are the number of stages in the rectifying and stripping sections, respectively.

Example problem 5.4:

A feed 100 kmol/h of saturated liquid containing 10 mole % LNK, 55 mole % LK, and 35 mole % HK and is to be separated in a distillation column. The reflux ratio is 1.2 the minimum. It is desired to have 99.5 % recovery of the light key in the distillate. The mole fraction of the light key in the distillate should be 0.75. Equilibrium data: $\alpha_{\text{LNK}} = 4.0$, $\alpha_{\text{LK}} = 1.0$, $\alpha_{\text{HK}} = 0.75$.

Find

- (i) Minimum number of stages required by Fenske method
- (ii) Minimum reflux ratio by Underwood method
- (iii) Number of ideal stages at $R = 1.2 R_{\min}$ by Gilliland method
- (iv) Also find the number of ideal stages at rectifying section and the stripping section at the operating reflux ratio and location of feed stage.

Solution 5.4:

(i) Feed $F = 100$ kmol/s, $x_{\text{LNK},F} = 0.1$, $x_{\text{LK},F} = 0.55$, $x_{\text{HK},F} = 0.35$, $x_{\text{LK},D} = 0.75$, $FR_{\text{LK},D} = 0.995$, $\alpha_{\text{HK}} = 0.75$, $\alpha_{\text{LNK}} = 4.0$, $\alpha_{\text{LK}} = 1.0$.

From the material balance

$$D = \frac{F \cdot x_{\text{LK},F} \cdot FR_{\text{LK},D}}{x_{\text{LK},D}} = 72.967 \text{ kmole/h}$$

Therefore $W = F - D = 27.033$ kmole/h

The amount of kmoles of different component in distillate:

$$n_{\text{LND}} = F \cdot x_{\text{LNK},F} \cdot FR_{\text{LK},D} = 54.725 \text{ kmole/h}$$

$$n_{\text{LNK},D} = F \cdot x_{\text{LNK},F} = 10 \text{ kmole/h}$$

$$n_{\text{HK},D} = D - n_{\text{LK},D} - n_{\text{LNK},D} = 8.242 \text{ kmole/h}$$

The amount of kilo moles of different component in bottoms:

$$n_{\text{LK},B} = F \cdot x_{\text{LK},F} (1 - FR_{\text{LK},D}) = 0.275 \text{ kmole/h}$$

$$n_{\text{LNK},B} = 0$$

$$n_{\text{HK},B} = B - n_{\text{LK},B} - n_{\text{LNK},B} = 26.758 \text{ kmole/h}$$

$$\alpha = 1/\alpha_{\text{HK}}$$

$$x_{\text{HK},D} = n_{\text{HK},D}/D = 0.113$$

$$x_{\text{LK},B} = n_{\text{LK},B}/B = 0.0102$$

Then as per Equation (5.71)

$$N_{\min} = 22.50$$

(ii) To find the minimum reflux at the condition of saturated liquid, $q = 1$, $\lambda_1 = \lambda_2 = \lambda$, using Equation (5.90)

$$0 = \frac{(4.0)(0.1)}{4.0 - \lambda} + \frac{(1.0)(0.55)}{1.0 - \lambda} + \frac{(0.75)(0.35)}{0.75 - \lambda}$$

gives $\lambda = 0.83$

Then from Equation (5.85)

$$V_{\min} = 253.5 \text{ kmole/h}$$

$$\text{And } L_{\min} = V_{\min} - D = 180.53 \text{ kmole/h}$$

$$R_{\min} = L_{\min}/D = 2.47$$

(iii) Now using the Gilliland correlation (Equation (5.92) to determine number of ideal stages at $R = 1.2 R_{\min} = 2.97$ one can get $N = 48.89$

(iv) Using Kirkbride Equation

$$\frac{N_R}{N_S} = \left[\left(\frac{x_{HK,F}}{x_{LK,F}} \right) \left(\frac{x_{LK,B}}{x_{HK,D}} \right)^2 \left(\frac{B}{D} \right) \right]^{0.206} = 0.275$$

$$\text{Again } N_R + N_S = N = 48.89$$

So by solving the above two Equations one get $N_R = 10.56$ and $N_S = 38.34$ and feed at stage 11

Nomenclature

B	Moles of bottoms	F	Moles of feed
BPC	Bubble point curve	f	Molal fraction of feed
D	Moles of distillate	H	Enthalpy
DPC	Dew point curve	K	Equilibrium constant
E _o	Overall tray efficiency	L	Moles of liquid
E _{mv}	Tray efficiency based on vapor phase	L'	Moles of liquid in stripping section
E _{mL}	Tray efficiency based on liquid phase	N, n	Number of tray
		P	Pressure, Pinch point
		Q	Rate of heat transfer

R	Reflux ratio
V	Moles of vapor
V'	Moles of vapor in stripping section
x	mole fractions in liquid
y	mole fractions in vapor
T	Temperature
α	Relative volatility
μ	Viscosity

Subscripts

B	Bottom
C	Condenser
D	Distillate
F	Feed
HK	Heavy key
i	1, 2, 3,, n
L	Liquid
LK	Light key
min	Minimum
NK	Non key
R	Rectifying section
S	Stripping section
V	Vapor

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