9

DISTILLATION

9.1 INTRODUCTION

The method of separating the components from a solution depending on its distribution between a liquid phase and vapour phase is termed distillation. This is applied to mixtures which distribute in both the phases.

This can also be defined as an operation in which a liquid or vapour mixture of two or more components is separated into its component fractions of desired purity, by the application of heat. Thus, in this process, a vapour is obtained from a boiling mixture which will be richer in components that have lower boiling points.

9.2 VAPOUR LIQUID EQUILIBRIA (VLE)

The vapour liquid equilibrium data is the basis for design of distillation operation equipments.

9.2.1 Constant Pressure Equilibria

A typical VLE at constant pressure is shown in Fig. 9.1.

The upper curve is the dew point curve which provides the relationship between temperature and mole fraction of the more volatile component in vapour phase (y) and the lower curve is the bubble point curve which gives the relationship between the temperature and mole fraction of the more volatile component in liquid phase (x) at a particular pressure. The horizontal tie lines CD, EF and GH at different temperatures provide equilibrium compositions of liquid and vapour phase at each temperature. Any mixture lying on the lower (bubble point) curve will be a saturated liquid and the mixture lying on the upper (dew point) curve will be a saturated vapour. A mixture located in between the two

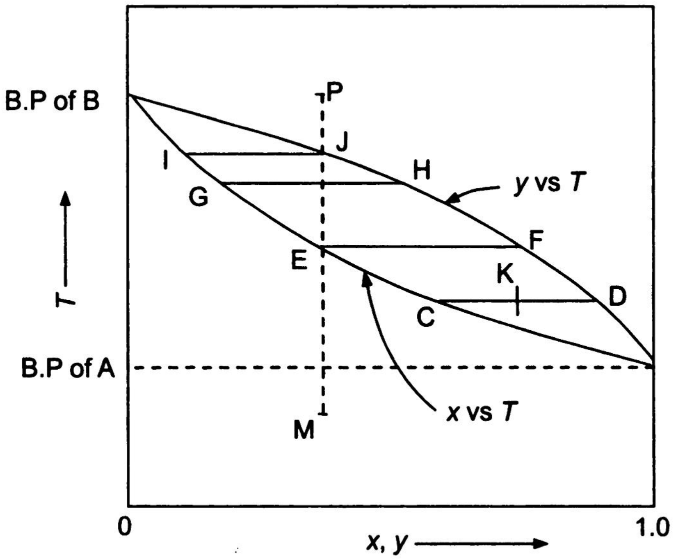

Fig. 9.1 VLE diagram at constant pressure.

curves, say K, will be a two-phase mixture of liquid and vapour with compositions C and D in liquid phase and vapour phase respectively. Their relative amounts are given by

$$\frac{\text{moles of } C}{\text{moles of } D} = \frac{\text{Length of line } KD}{\text{Length of line } KC}$$

Consider a mixture at point M. It is only a liquid. If it is kept inside a cylinder fitted with a frictionless piston and heated, its temperature will increase till it reaches ‘E’ when it will become a saturated liquid. The vapour in equilibrium with it will have a composition of F. As heating is further continued, more vapourization takes place, the liquid phase composition will move towards G and the associated vapour will have a composition of H. The effective composition of the entire mass comprising both liquid and vapour continues to remain at M. Finally, when the last droplet of liquid as indicated at point ‘I’ is vapourized, the vapour generated would have a composition of ‘J’. Further application of heat results in superheating of the vapour. During the entire operation, the pressure is kept constant.

9.2.2 Effect of Pressure

As pressure is increased, the boiling points of components increase and the looped curves become more and more narrow. As the critical pressure is exceeded for one of the components, there is no longer a distinction between vapour and liquid for that component, and for mixtures the looped curves are, therefore, shorter as depicted in Fig. 9.2, for case (C). Distillation is possible only in the region where a looped curve exists. It is also clear that relative volatility, α, also changes in such cases.

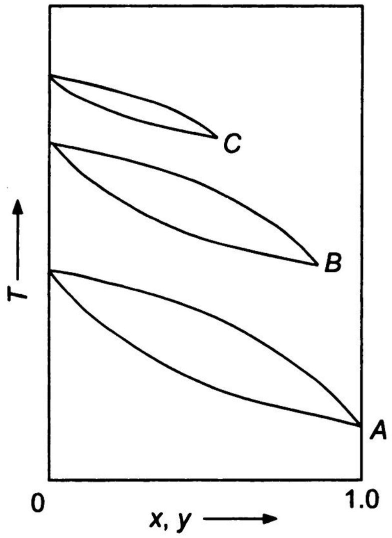

Fig. 9.2 Effect of pressure on VLE.

9.2.3 Constant Temperature Equilibria

A typical VLE at constant temperature is shown in Fig. 9.3.

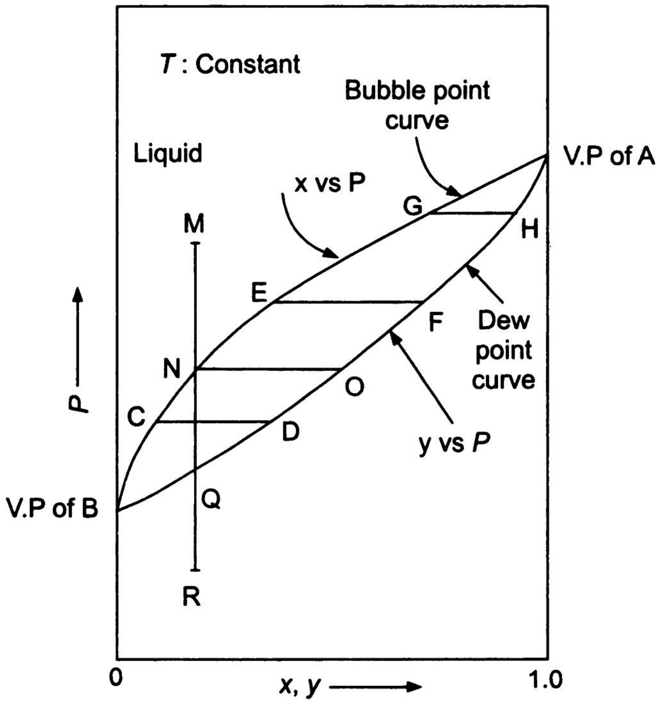

Fig. 9.3 VLE at constant temperature.

As in the case of constant temperature equilibria, lines CD, EF and GH are tie lines indicating the equilibrium compositions of liquid and vapour phase at various pressures. A liquid defined at point M is a liquid below its bubble point and as the pressure is reduced at constant temperature, at point 'N' on the upper (bubble point) curve, a saturated liquid is obtained. As the pressure is brought down further, at point Q on the lower (dew point) curve, a saturated vapour forms and a further reduction in pressure gives a fully superheated vapour as defined by R.

9.3 RELATIVE VOLATILITY (α)

This is defined as the ratio of vapour pressure of more volatile component to that of less volatile component. If P_A and P_B are the vapour pressures of A and B respectively, the relative volatility of A with respect to B, α_{AB} is defined as the ratio of vapour pressure of A to that of B.

i.e.

$$\alpha_{AB} = \frac{P_A}{P_B} \tag{9.1}$$

Raoult's law states that when a gas and a liquid are in equilibrium, the partial pressure of A, p_A is equal to the product of its vapour pressure, P_A at that temperature and its mole fraction x_A in the liquid.

i.e.

$$p_A = P_A \cdot x_A \tag{9.2}$$

Similarly,

$$p_B = P_B \cdot x_B \tag{9.3}$$

When the gas and liquid behave ideally, Raoult's law holds good.

We know that sum of the partial pressures of components in a gas mixture is equal to the total pressure, P_T. The composition of a component y, in gas phase is given by Dalton's law,

$$y_A = \frac{p_A}{P_T} \quad \text{and} \quad y_B = \frac{p_B}{P_T} \tag{9.4}$$

$$\therefore \quad \alpha_{AB} = \frac{P_A}{P_B} = \frac{\left(\frac{p_A}{x_A}\right)}{\left(\frac{p_B}{x_B}\right)} = \frac{\left(\frac{P_T y_A}{x_A}\right)}{\left(\frac{P_T y_B}{x_B}\right)} = \frac{\left(\frac{y_A}{y_B}\right)}{\left(\frac{x_A}{x_B}\right)}$$

$$\alpha_{AB} = \frac{\left(\frac{y_A}{1-y_A}\right)}{\left(\frac{x_A}{1-x_A}\right)} \tag{9.5}$$

Rearranging, we get

$$y_A = \frac{\alpha_{AB} x_A}{1 + x_A(\alpha_{AB} - 1)} \tag{9.6}$$

and more simply as

$$y = \frac{\alpha x}{1 + x(\alpha - 1)} \tag{9.7}$$

9.4 COMPUTATION OF VLE DATA (EQUILIBRIUM DATA)

The vapour pressure of the components involved is the basis for the computation of VLE data.

From Eqs. (9.2) and (9.3), $p_A = x_A P_A$

$$p_B = x_B P_B = (1 - x_A) P_B \tag{9.8}$$

For a binary system,

$$p_A + p_B = P_T = x_A P_A + (1 - x_A) P_B = P_B + x_A (P_A - P_B) \tag{9.9}$$

$$\therefore \qquad x_A = \frac{(P_T - P_B)}{(P_A - P_B)} \tag{9.10}$$

From the vapour pressure data at each temperature, x_A can be computed using Eq. (9.10). After computing x_A, the partial pressure p_A can be estimated by using Eq. (9.2). The mole fraction of A in gas phase, y_A is then determined by using Eq. (9.4). Thus, for the whole range of boiling points of components involved, the VLE data can be computed.

Whenever α lies in a narrow range, y can be computed by assuming various values of x using Eq. (9.7).

9.5 DEVIATION FROM IDEALITY

A mixture whose total pressure is either greater or lesser than that computed using Raoult's law is said to exhibit either a positive deviation or a negative deviation from ideality.

9.5.1 Positive Deviation from Ideality

When the total pressure of a mixture is greater than that for ideal mixtures computed using Raoult's law, the mixture is said to exhibit positive deviations from ideality and such mixtures are called minimum boiling azeotropes, i.e. at some composition the mixture shows minimum boiling point (at constant pressure) and maximum pressure (at constant temperature) as shown in Figs. 9.4 and 9.5. A typical x-y diagram is also shown in Fig. 9.6. Most of the azeotropic mixtures fall under this category.

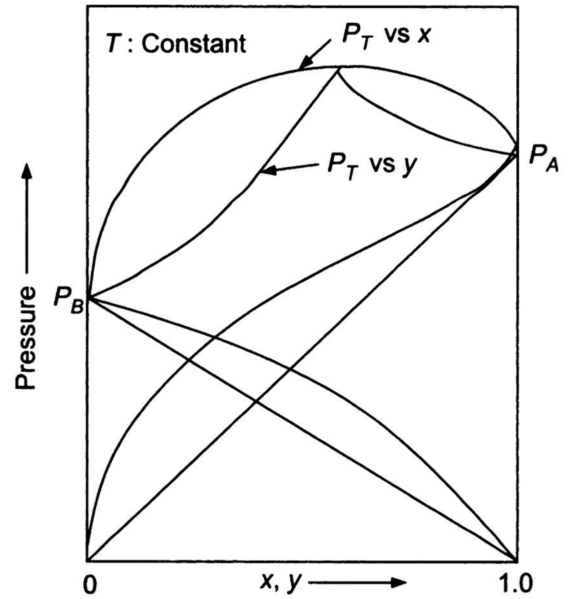

Fig. 9.4 Minimum boiling azeotrope at constant temperature.

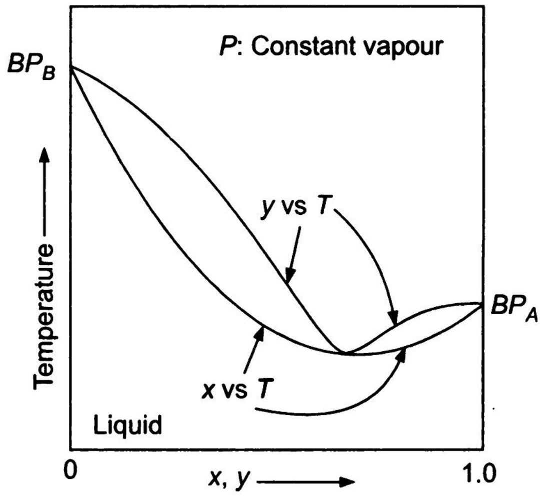

Fig. 9.5 Minimum boiling azeotrope at constant pressure.

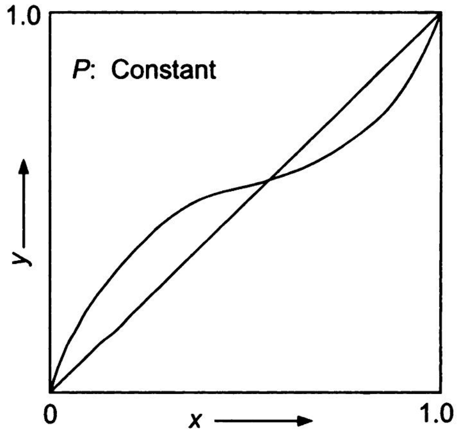

Fig. 9.6 VLE of minimum boiling azeotrope.

9.5.2 Negative Deviations from Ideality

When the total pressure of a system is less than the ideal value as computed using Raoult's law, the system is said to deviate negatively. Such systems are very rare and they are also called maximum boiling azeotropes, i.e. at some composition the mixture shows maximum boiling point. Typical *P–x–y*, *T–x–y* and *x–y* diagrams are shown in Figs. 9.7, 9.8 and 9.9.

9.6 TYPES OF DISTILLATION COLUMNS

Based on the nature of operation, distillation columns have been classified as batch and continuous columns as shown in Fig. 9.10.

9.6.1 Batch Columns

In batch columns, the feed to the column is introduced batchwise and the distillation is carried. When the desired quality is reached or when the desired quantity is distilled out, the operation is stopped and next batch of feed is introduced.

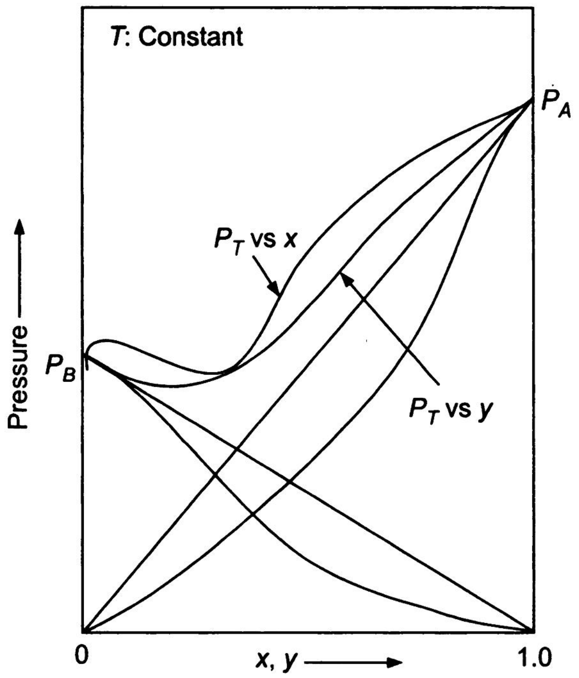

Fig. 9.7 Maximum boiling azeotrope at constant temperature.

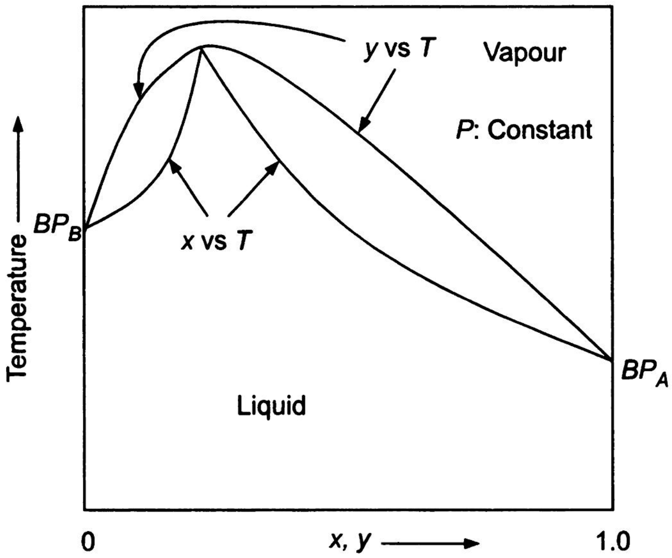

Fig. 9.8 Maximum boiling azeotrope at constant pressure.

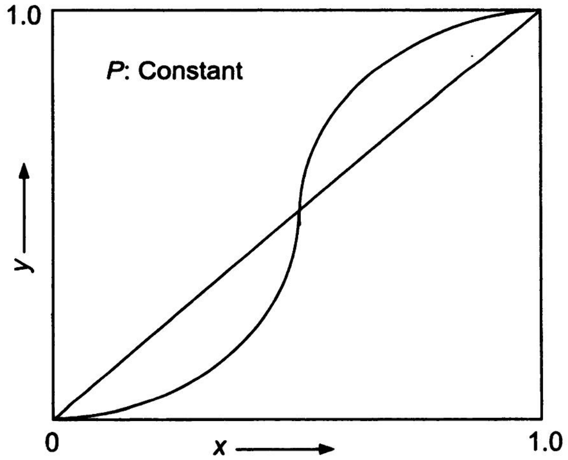

Fig. 9.9 VLE of maximum boiling azeotrope.

9.6.2 Continuous Columns

These columns have a continuous feed stream and are capable of handling high throughputs. These are further classified on the basis of,

- *The nature of the feed they are further processing*
  - Binary columns—Feed has only two components
  - Multicomponent column—Feed has more than two components
- *The number of product streams they have*
  - Two product streams
  - Multi product streams
- *The use of additional components in distillation*
  - Extractive distillation—use of solvent
  - Azeotropic distillation—use of entrainer
- *The type of columns*:
  - Tray columns—use of sieve plate columns/Bubble cap trays/Valve trays for better vapour–liquid contacting
  - Packed towers—use of packings in columns for better vapour—liquid contacting.

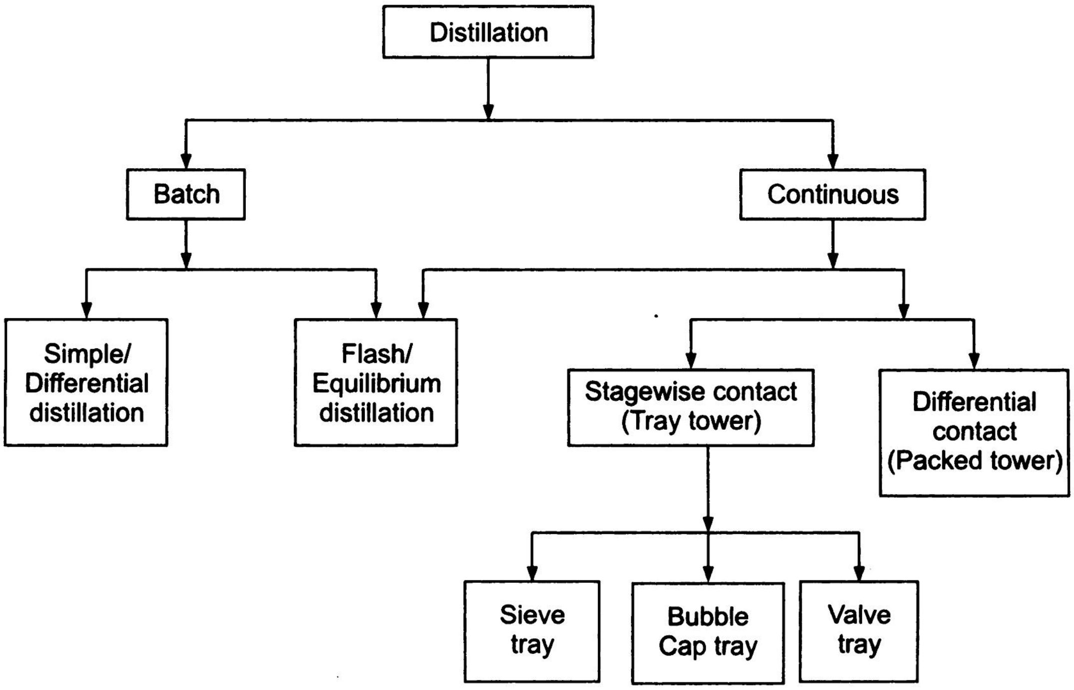

Fig. 9.10 Types of distillation and equipments.

9.7 STEAM DISTILLATION

Some systems have very high boiling points and some of these substances are unstable at high temperatures. Especially when such systems are completely insoluble with each other, steam distillation can be a useful method of separating such mixtures.

For example, consider a mixture of hydrocarbon and water which are immiscible. The vapour pressure of either component cannot be influenced by the presence of the other component and each exerts its own vapour pressure at the prevailing temperature. When the sum of the vapour pressures is equal to the total pressure, the mixture boils. With vapour pressure data of the individual components, one can also estimate the temperature at which such distillations take place.

$$P_T = P_A + P_B \tag{9.11}$$

It is clear from Fig. 9.11, that this type of distillation takes place at a temperature which will be even less than that of the boiling point water. This method suffers from poor efficiency in its operation, as large quantity of water has to be evaporated. However, one can introduce the effectiveness in such operations by

- Operating at different total pressures in which case the ratio of vapour pressure of the substances may be more favourable.
- Sparging the mixture with superheated steam or other insoluble gas.

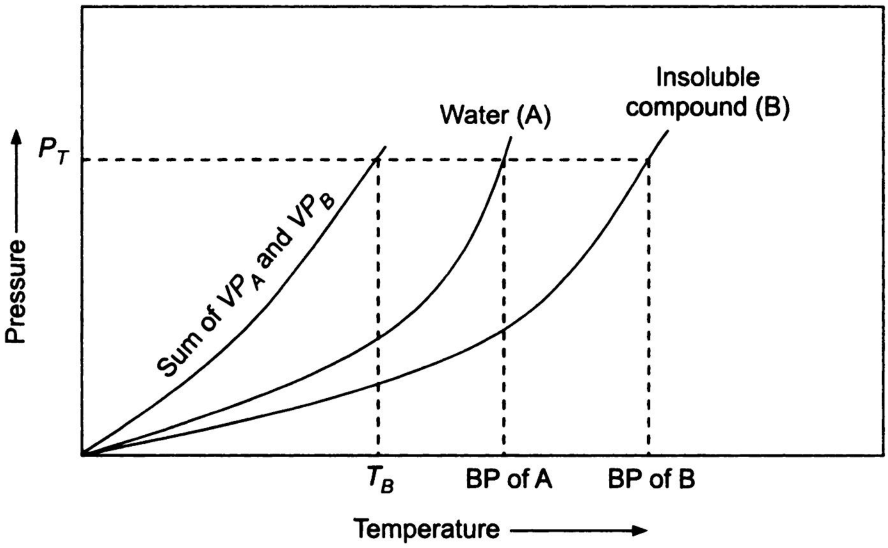

Fig. 9.11 Steam distillation.

9.8 DIFFERENTIAL OR SIMPLE DISTILLATION

Consider a feed F containing x_F mole fraction of more volatile component fed into a batch still as shown in Fig. 9.12. Let L be the total moles present in the still at any instant, t and x be the mole fraction of more volatile component. Let dL be the moles distilled out. The concentration of vapour in the leaving stream is y^*. The moles left behind in the still is $(L - dL)$. During this process the concentration of more volatile component left behind in the still is $(x - dx)$.

Total moles of more volatile component present initially is Lx

Total moles of more volatile component in distillate is y^*dL

Total moles of more volatile component in residue is $(L - dL)(x - dx)$

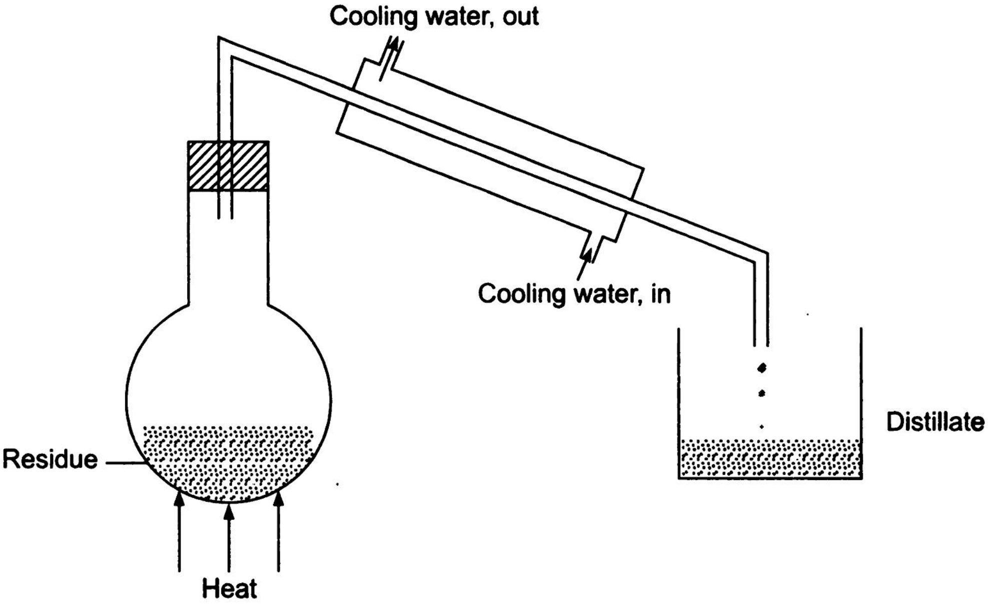

Fig. 9.12 Differential distillation.

Making a component balance, we get

$$Lx = y^*\,dL + (L - dL)\,(x - dx) \tag{9.12}$$

$$Lx = y^*\,dL + Lx - Ldx - x{\cdot}dL + \overset{0}{\cancel{dx\,dL}} \tag{9.13}$$

(∵ Product of two very small quantities)

Then, $$dL(y^* - x) = Ldx \tag{9.14}$$

∴ $$\frac{dL}{L} = \frac{dx}{(y^* - x)} \tag{9.15}$$

Integrating between limits $x = x_F \quad L = F$

$x = x_W \quad L = W$

∴ $$\int_W^F \frac{dL}{L} = \int_{X_W}^{X_F} \frac{dx}{(y^* - x)} \tag{9.16}$$

$$\ln \frac{F}{W} = \int_{X_W}^{X_F} \frac{dx}{(y^* - x)} \tag{9.17}$$

Equation (9.17) is called *Rayleigh's equation.*

The right-hand side cannot be integrated as y^* is a function of x.

Hence, the right-hand side of Eq. (9.17) can be evaluated either graphically or numerically with the help of x-y or VLE data.

For systems where the relative volatility lies in a narrow range, we can use Eq. (9.7) which states that

$$y = \frac{\alpha x}{[1 + (\alpha - 1)x]}$$

Hence, replacing y in terms of Eq. (9.7), we get

$$\ln \frac{F}{W} = \int_{x_W}^{x_F} \left[\frac{dx}{\dfrac{\alpha x}{\left[1+(\alpha-1)x\right]} - x} \right] \tag{9.18}$$

$$\text{RHS of Eq. (9.18)} = \int \left[\frac{dx}{\dfrac{\alpha x}{\left[1+(\alpha-1)x\right]} - x} \right]. \tag{9.19}$$

On simplification,

$$= \int \frac{[1+(\alpha-1)x]dx}{[\alpha x - x - \alpha x^2 + x^2]} = \int \frac{[1+(\alpha-1)x]dx}{x[\alpha - 1 - \alpha x + x]}$$

$$= \int \frac{[1+(\alpha-1)x]dx}{x[1(\alpha-1) - x(\alpha-1)]} = \int \frac{[1+(\alpha-1)x]dx}{[x(1-x)(\alpha-1)]}$$

$$= \int \frac{dx}{[x(1-x)(\alpha-1)]} + \int \frac{dx}{(1-x)} = \frac{1}{(\alpha-1)} \int \left[\left(\frac{A}{x}\right) + \left(\frac{B}{1-x}\right)\right] dx - \ln(1-x)$$

$$= \frac{1}{(\alpha-1)} \int \frac{A(1-x) + B(x)}{x(1-x)} dx - \ln(1-x)$$

$$= \frac{1}{(\alpha-1)} \left[\int \frac{dx}{x} + \int \frac{dx}{(1-x)} \right] - \ln(1-x)$$

Substituting the limits for x as x_F and x_W, we get

$$= \frac{1}{(\alpha-1)} \left[\ln x - \ln(1-x)\right] - \ln(1-x)$$

$$= \frac{1}{(\alpha-1)} \left[\ln\left(\frac{x_F}{x_W}\right) - \ln\frac{(1-x_F)}{(1-x_W)} \right] - \ln\frac{(1-x_F)}{(1-x_W)}$$

i.e.

$$\ln\left(\frac{F}{W}\right) = \frac{1}{(\alpha-1)} \left[\ln\left(\frac{x_F}{x_W}\right) - \ln\left(\frac{(1-x_F)}{(1-x_W)}\right) \right] - \ln\frac{(1-x_F)}{(1-x_W)}$$

$$\ln\left(\frac{F}{W}\right) + \ln\left(\frac{(1-x_F)}{(1-x_W)}\right) = \frac{1}{(\alpha-1)} \ln\left[\frac{\dfrac{x_F}{x_W}}{\dfrac{(1-x_F)}{(1-x_W)}} \right]$$

$$\ln \frac{F(1-x_F)}{W(1-x_W)} = \frac{1}{(\alpha-1)} \ln \left\{ \left[\frac{x_F}{(1-x_F)} \right] \left[\frac{(1-x_W)}{x_W} \right] \right\}$$

$$(\alpha-1) \ln \frac{F(1-x_F)}{W(1-x_W)} = \ln \left\{ \left[\frac{x_F}{(1-x_F)} \right] \left[\frac{(1-x_W)}{x_W} \right] \right\}$$

$$\alpha \ln \frac{F(1-x_F)}{W(1-x_W)} = \ln \left[\frac{\dfrac{x_F}{x_W}}{\dfrac{(1-x_F)}{(1-x_W)}} \right] + \ln \left[\frac{F(1-x_F)}{W(1-x_W)} \right]$$

$$\alpha \ln \left[\frac{F(1-x_F)}{W(1-x_W)} \right] = \ln \left(\frac{x_F}{x_W} \right) \left(\frac{F}{W} \right)$$

$$\alpha \ln \left[\frac{F(1-x_F)}{W(1-x_W)} \right] = \ln \left(\frac{F x_F}{W x_W} \right)$$

i.e.

$$\frac{F x_F}{W x_W} = \left[\frac{F(1-x_F)}{W(1-x_W)} \right]^{\alpha} \tag{9.20}$$

Equation (9.20) is very useful in the estimation of the amount of residue (alternatively, the estimation of the quantity to be distilled) in case of systems of constant relative volatility. This is also used in the estimation of relative volatility for such systems.

9.9 EQUILIBRIUM OR FLASH DISTILLATION

Consider a feed at a flow rate of F (moles per hour), containing the more volatile component with a composition of Z_F and an enthalpy of H_F (per mole of feed) entering a preheater. Let the heat added in the preheater be Q. The mixture then enters a flash chamber where a distillate leaves at a rate of D (moles per hour) with a composition of y_D and an enthalpy of H_D (per mole of distillate). The bottoms (residue) leave at a rate of W, with a composition of x_W and an enthalpy of H_W (per mole of residue). The entire process is shown in Fig. 9.13

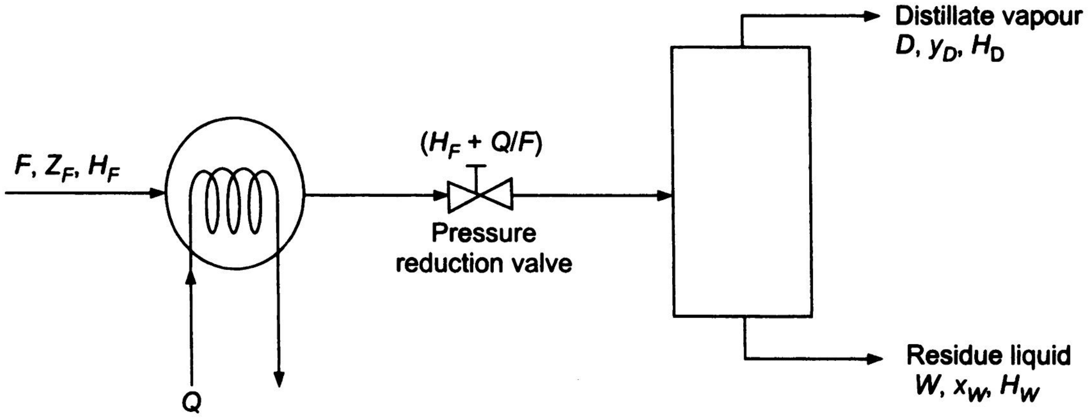

Fig. 9.13 Flash distillation.

A total material balance, gives,

$$F = W + D \tag{9.21}$$

A component balance gives,

$$FZ_F = Wx_W + Dy_D \tag{9.22}$$

An enthalpy balance gives,

$$FH_F + Q = WH_W + DH_D$$

i.e.

$$F\left(H_F + \frac{Q}{F}\right) = WH_W + DH_D \tag{9.23}$$

From Eqs. (9.21) and (9.22), we get

$$(W + D)Z_F = Wx_W + Dy_D \tag{9.24}$$

$$\therefore \quad W\,[Z_F - x_W] = -D[Z_F - y_D] \tag{9.25}$$

$$\therefore \quad -\frac{W}{D} = \frac{(Z_F - y_D)}{(Z_F - x_W)} \tag{9.26}$$

Similarly from Eqs. (9.21) and (9.23), we get

$$-\frac{W}{D} = \frac{\left[\left(H_F + \frac{Q}{F}\right) - H_D\right]}{\left[\left(H_F + \frac{Q}{F}\right) - H_W\right]} \tag{9.27}$$

Dividing Eq. (9.25) by F, we get

$$\frac{W}{F}(Z_F - x_W) = -\frac{D}{F}(Z_F - y_D) \tag{9.28}$$

Let f be the fraction of feed vaporised and subsequently condensed and removed. Hence, $(1 - f)$ is the fraction of feed left behind as residue.

$$\therefore \quad (1 - f)\,(Z_F - x_W) = f\,(y_D - Z_F) \tag{9.29}$$

$$Z_F - x_W - fZ_F + f\,x_W = fy_D - fZ_F$$

$$\therefore \quad Z_F - x_W = f\,(y_D - x_W)$$

$$Z_F + x_W\,(f - 1) = fy_D$$

$$\therefore \quad y_D = \frac{Z_F}{f} + x_W\,\frac{(f - 1)}{f} \tag{9.30}$$

So Eq. (9.30) can be called an operating line drawn with a slope of $[(f - 1)/f]$ and simplified as,

$$y = \left(\frac{Z_F}{f}\right) + x\left(\frac{f - 1}{f}\right) \tag{9.31}$$

The feed point is $x = y = Z_F$

Having seen the principles involved in flash distillation, let us now see how compositions are estimated in a flash distillation operation.

9.9.1 Steps

There are two methods available to estimate the composition of products. They are explained in detail below.

Case I

When the equilibrium data and the quantity of either the distillate or the residue and feed are available, the following procedure shall be adopted:

- Draw the equilibrium curve
- Draw the diagonal ($x = y$ line)
- Locate feed point corresponding to x_F on the diagonal ($x_F = y_F = Z_F$)
- Draw the operating line with a slope of $\left[-\frac{W}{D}\right]$
- The intersection of this line with equilibrium curve gives x_W and y_D as shown in Fig. 9.14.

Case II

When the enthalpy–concentration data (H_L vs x and H_G vs y) and heat added Q are available, the following procedure shall be adopted.

- Plot the enthalpy concentration data and also equilibrium curve below it.
- Locate the feed point corresponding to $F(Z_F, H_F + Q/F)$
- Draw a line by trial and error, passing through F such that it will be a tie line.
- The points of intersection of this line (drawn by trial and error) with enthalpy–concentration curves gives the enthalpy and concentration of both the distillate and the residue.

Figures (9.14) and (9.15) represent the procedures followed to determine the product concentrations for case I and case II respectively.

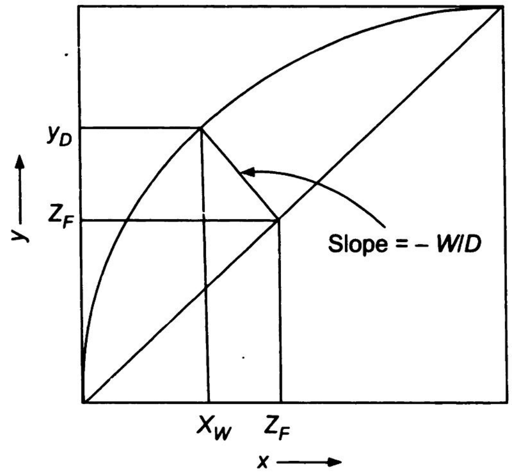

Fig. 9.14 Estimation of composition of products in flash distillation.

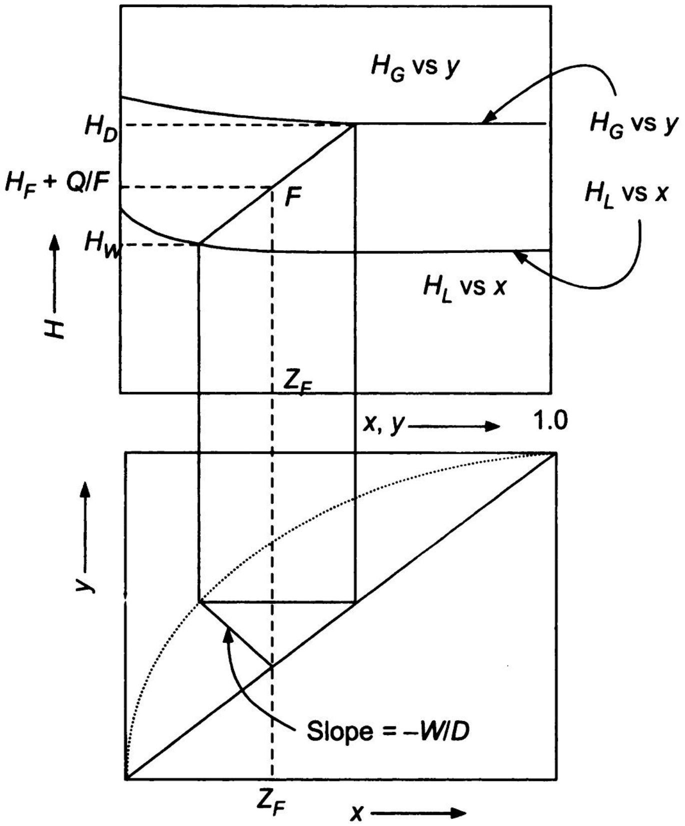

Fig. 9.15 Enthalpy–concentration diagram.

9.10 MULTICOMPONENT SIMPLE DISTILLATION

Let us consider a multicomponent mixture fed to a still. The distillate and the residue left behind will also be multicomponent mixtures. For our analysis, let us consider a three-component system wherein α remains fairly constant. Modified Rayleigh's equation can be applied for material balance,

$$\ln\left[\frac{Fx_{F,A}}{Wx_{W,A}}\right] = \ln\left[\frac{Fx_{F,B}}{Wx_{W,B}}\right]^{\alpha_{AB}} \tag{9.32}$$

$$\ln\left[\frac{Fx_{F,A}}{Wx_{W,A}}\right] = \ln\left[\frac{Fx_{F,C}}{Wx_{W,C}}\right]^{\alpha_{AC}} \tag{9.33}$$

Similarly,

$$\ln\left[\frac{Fx_{F,B}}{Wx_{W,B}}\right] = \ln\left[\frac{Fx_{F,C}}{Wx_{W,C}}\right]^{\alpha_{BC}} \tag{9.34}$$

Here, $BP_A < BP_B < BP_C$, where BP is the boiling point. We also know that

$$x_{WA} + x_{WB} + x_{WC} = 1.0 \tag{9.35}$$

In a typical feed mixture, the values of F, x_{FA}, x_{FB} and x_{FC} are known. The unknown quantities will be W, D, x_{WA}, x_{WB} and x_{WC}. To solve such problems one has to assume W. By substituting in the first three equations, a relationship between x_{WA}, x_{WB} and x_{WC} is obtained. Then it can be solved and checked for the validity of assumed value of W using Eq. (9.35). If Eq. (9.35) is satisfied, the assumed value of W is correct. If not, a new value for W is assumed and the above calculations are repeated till Eq. (9.35) is satisfied.

Subsequently using the total material balance equation, D can be calculated and then the mole fraction of each component in the distillate phase (vapour phase) can be evaluated by making a component balance.

However, in cases where α varies significantly, the Rayleigh's equation of the form $\ln \frac{F}{W} = \int_{x_w}^{x_F} \frac{dx}{(y-x)}$ has to be used taking two components at a time. Here also one has to assume W and suitably estimate x_W. The values of x_W will have to be determined for all the components and finally checked using Eq. (9.35). If Eq. (9.35) is not satisfied, one has to make a fresh assumption of W and has to proceed till Eq. (9.35) is satisfied.

9.11 MULTICOMPONENT FLASH DISTILLATION

At low pressures almost all systems behave ideally. As flash distillation occurs generally at low pressures, ideal behaviour can be expected and Raoult's law is applicable. Hence, the equilibrium relationship for any component may be expressed as

$$y_i = m_i x_i \tag{9.36}$$

where m_i = vapour pressure of component/total pressure. The suffix i denotes the component.

i.e.

$$y_{i,D} = m_i\,(x_{i,W}) \tag{9.37}$$

We know that $-\frac{W}{D} = \frac{(y_D - Z_F)}{(x_W - Z_F)}$ [from Eq. (9.26)]

$\therefore$

$$\frac{W}{D} = \frac{(y_{i,D} - Z_{i,F})}{(Z_{i,F} - x_{i,W})} \tag{9.38}$$

$$= \frac{\left[y_{i,D} - Z_{i,F}\right]}{\left[Z_{i,F} - \frac{y_{i,D}}{m_i}\right]} \tag{9.39}$$

or

$$\frac{W}{D}\left[Z_{i,F} - \frac{y_{i,D}}{m_i}\right] = [y_{i,D} - Z_{i,F}] \tag{9.40}$$

i.e

$$Z_{i,F}\left[\frac{W}{D} + 1\right] = y_{i,D}\left[1 + \left(\frac{1}{m_i}\right)\left(\frac{W}{D}\right)\right] \tag{9.41}$$

$$\therefore \qquad y_{i,D} = Z_{i,F} \frac{\left[\frac{W}{D}+1\right]}{\left[1+\left(\frac{1}{m_i}\right)\left(\frac{W}{D}\right)\right]} \tag{9.42}$$

and

$$x_{i,W} = Z_{i,F} \frac{\left[\frac{W}{D}+1\right]}{\left[m_i+\frac{W}{D}\right]} \tag{9.43}$$

$y_{i,D}$ is evaluated using Eq. (9.42) and $x_{i,W}$ is evaluated using Eq. (9.43) by assuming (W/D) value and finally checked for its validity by using

$$\Sigma x_{i,W} = 1.0; \qquad \Sigma y_{i,D} = 1.0$$

9.11.1 Steps Involved

- From vapour pressure, determine m for each component.
- Assume W/D value and determine $x_{i,W}$ and $y_{i,D}$
- Check whether $\Sigma x_{i,W}$ and $\Sigma y_{i,D}$ are 1.0.
- If they are 1.0, then the assumed W/D ratio is correct.
- If not, assume a new value for W/D and ensure that $\Sigma x_{i,W} = 1.0$; $\Sigma y_{i,D} = 1.0$ are satisfied.

9.12 CONTINUOUS RECTIFICATION

A schematic sketch of a typical distillation column with a feed stream and a distillate and residue stream is shown in Fig. 9.16 along with its main accessories.

9.12.1 Ponchon–Savarit Method

There are two methods by which the design of the continuous fractionator can be established. Let us first consider Ponchon–Savarit method where it requires both enthalpy and concentration data.

Envelope I: Condenser section

Envelope II: Full distillation unit

Envelope III: Enriching/Rectifying section

Envelope IV: Stripping/Exhausting section

The numbering of plates or trays is accounted from the top to bottom. Suffix denotes the properties of streams leaving a particular plate or tray. Let n and m, denote general plates in the enriching section and stripping section respectively.

Let G be the molar flow rate of vapour in enriching section, $\bar{G}$ the molar flow rate of vapour in stripping section, L the molar flow rate of liquid in enriching

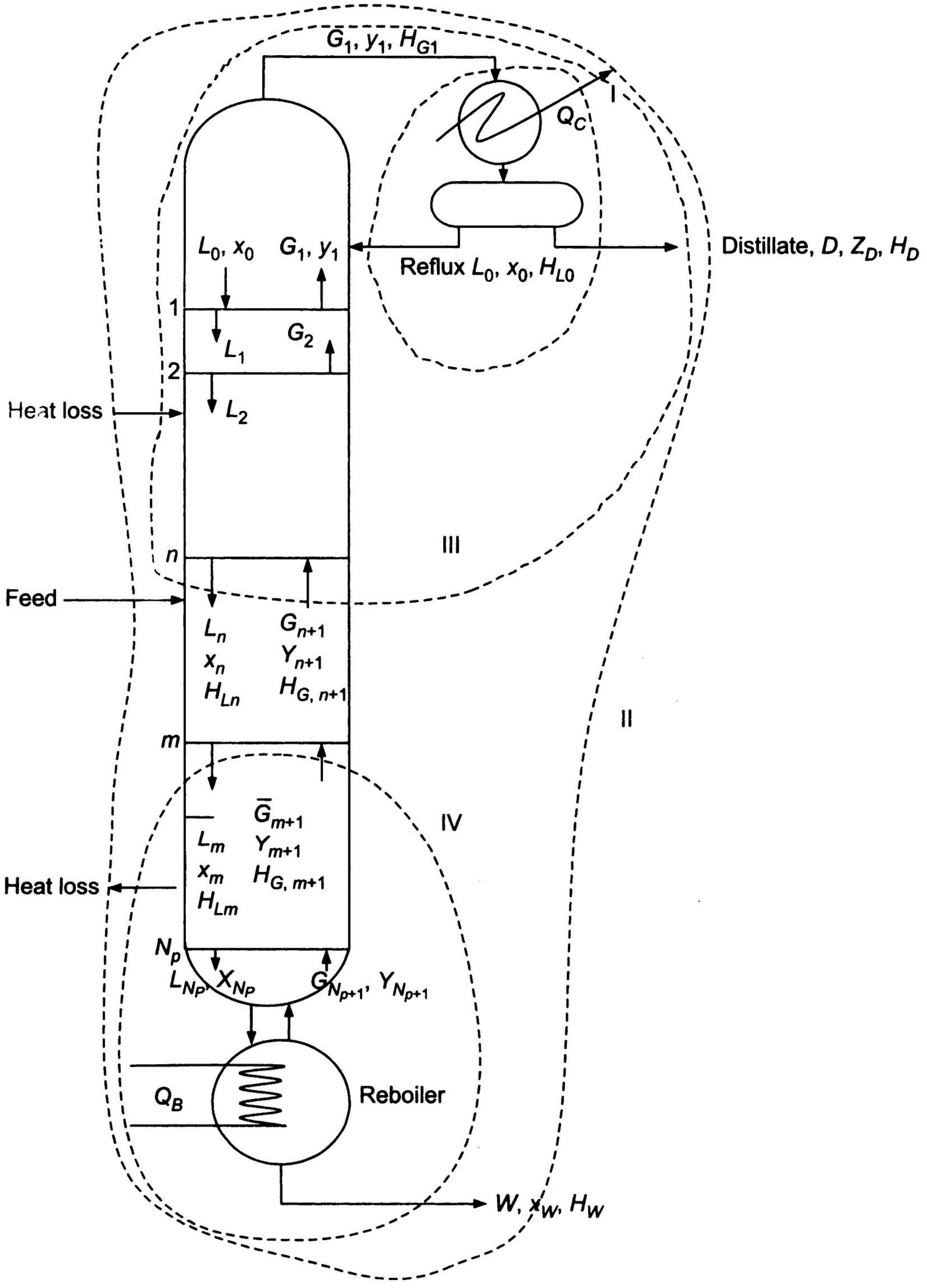

Fig. 9.16 Continuous fractionator.

section, $\bar{L}$ the molar flow rate of liquid in stripping section, H_G the Enthalpy of vapour, H_L the Enthalpy of liquid, y the mole fraction of more volatile component in vapour and x the mole fraction of more volatile component in liquid.

Let R be the *external reflux ratio* L_0/D, Q_C the load on condenser, Q_B the heat supplied in reboiler and Q_L the total heat loss.

Considering envelope I and making a mass balance,

$$G_1 = D + L_0 \tag{9.44}$$

$$G_1 = D + RD = D(R + 1) \tag{9.45}$$

A component balance gives

$$G_1 y_1 = DZ_D + L_0 x_0 \tag{9.46}$$

Making an energy balance, we get

$$G_1 H_{G_1} = L_0 H_{L_0} + DH_D + Q_C \tag{9.47}$$

$$\therefore \quad Q_C = G_1 H_{G_1} - L_0 H_{L_0} - DH_D \tag{9.48}$$

Substituting for G_1 from Eq. (9.45), we get

$$Q_C = [D(R + 1)\ H_{G_1}] - RDH_{L_0} - DH_D$$

$$= D[(R + 1)H_{G_1} - RH_{L_0} - H_D] \tag{9.49}$$

Considering envelope II and making an energy balance, we have
Heat in = Heat out

$$Q_B + FH_F = DH_D + WH_W + Q_C + Q_L \tag{9.50}$$

$\therefore$ Heat added in reboiler $Q_B = DH_D + WH_W + Q_C + Q_L - FH_F$ (9.51)

Now, let us consider envelope III, the enriching section and make mass and energy balance.
A total mass balance yields,

$$G_{n+1} = L_n + D \tag{9.52}$$

A component balance gives,

$$G_{n+1} y_{n+1} = L_n x_n + DZ_D \tag{9.53}$$

An energy balance gives,

$$G_{n+1} H_{G_{n+1}} = L_n \cdot H_{L_n} + DH_D + Q_C \tag{9.54}$$

Let Q' = (Net heat out/Net moles out) = $\left[\dfrac{(Q_C + DH_D)}{D}\right]$ (9.55)

Then, Eq. (9.54) becomes

$$G_{n+1}\ H_{G_{n+1}} = L_n \cdot H_{L_n} + DQ' \tag{9.56}$$

Eliminating D from Eq. (9.53) using Eq. (9.52), we get

$$(G_{n+1})(y_{n+1}) - L_n x_n = (G_{n+1} - L_n)Z_D \tag{9.57}$$

$$(G_{n+1})[Z_D - y_{n+1}] = L_n(Z_D - x_n)$$

$$\frac{L_n}{G_{n+1}} = \frac{(Z_D - y_{n+1})}{(Z_D - x_n)} \tag{9.58}$$

where (L_n/G_{n+1}) is defined as *internal reflux ratio.*

Similarly, Eqs. (9.52) and (9.56) yield

$$\frac{L_n}{G_{n+1}} = \frac{(Q' - H_{G_{n+1}})}{(Q' - H_{L_n})} \tag{9.59}$$

Equating Eq. (9.58) with Eq. (9.59), we get

$$\frac{L_n}{G_{n+1}} = \frac{(Z_D - y_{n+1})}{(Z_D - x_n)} = \frac{(Q' - H_{G_{n+1}})}{(Q' - H_{L_n})} \tag{9.60}$$

Equation (9.60) represents a straight line passing through

$(H_{G_{n+1}}, y_{n+1})$ at G_{n+1}, (H_{L_n}, x_n) at L_n and (Q', Z_D) at Δ_D

where Δ_D is called the difference point and it represents

Q': Net heat, out/Net moles, out and

Z_D: Net moles of more volatile component, out/Net moles, out

Let us consider Eq. (9.59).

$$\frac{L_n}{G_{n+1}} = \frac{(Q' - H_{G_{n+1}})}{(Q' - H_{L_n})}$$

Substituting for G_{n+1} in the above expression from Eq. (9.52), we have

$$\frac{L_n}{(L_{n+D})} = \frac{(Q' - H_{G_{n+1}})}{(Q' - H_{L_n})} \tag{9.61}$$

i.e.

$$\frac{(L_{n+D})}{L_n} = \frac{(Q' - H_{L_n})}{(Q' - H_{G_{n+1}})}$$

i.e.

$$1 + \left[\frac{D}{L_n}\right] = \frac{(Q' - H_{L_n})}{(Q' - H_{G_{n+1}})}$$

$\therefore$

$$\frac{D}{L_n} = \frac{(H_{G_{n+1}} - H_{L_n})}{(Q' - H_{G_{n+1}})} \tag{9.62}$$

$\therefore$

$$\frac{L_n}{D} = \frac{(Q' - H_{G_{n+1}})}{(H_{G_{n+1}} - H_{L_n})} \tag{9.63}$$

When $n = 0$, it indicates the condenser and for $n = 0$, we get

$$\frac{L_0}{D} = \frac{(Q' - H_{G_1})}{H_{G_1} - H_{L_0}} \tag{9.64}$$

i.e. R, the external reflux ratio $= \dfrac{L_0}{D} = \dfrac{\text{Length of line } \Delta_D G_1}{\text{Length of line } G_1 L_0}$ (9.65)

as indicated in Fig. 9.17(a).

Hence, if the reflux ratio R is known, then it will be easy for us to locate Δ_D point (Z_D, Q').

Also, $$\frac{L_n}{D} = \frac{(Z_D - y_{n+1})}{(y_{n+1} - x_n)} \qquad \text{[from Eq. (9.58)]}$$

Let us consider envelope IV in the stripping section.
A mass balance yields

$$\therefore \qquad \bar{L}_m = \bar{G}_{m+1} + W \tag{9.66}$$

A component balance yields

$$\bar{L}_m x_m = \bar{G}_{m+1} y_{m+1} + W x_W \tag{9.67}$$

$$\therefore \qquad L_m x_m - \bar{G}_{m+1} y_{m+1} = W x_W \tag{9.68}$$

An energy balance yields

$$L_m \cdot H_{L_m} + Q_B = \bar{G}_{m+1} H_{G_{m+1}} + W H_W \tag{9.69}$$

Let $$Q'' = \frac{(W H_W - Q_B)}{W} \tag{9.70}$$

$$\therefore \qquad \bar{L}_m \cdot H_{L_m} - \bar{G}_{m+1} H_{G_{m+1}} = W Q'' \tag{9.71}$$

Eliminating W from Eqs. (9.66) and (9.68), we get

$$\frac{\bar{L}_m}{G_{m+1}} = \frac{(y_{m+1} - x_W)}{(x_m - x_W)} \tag{9.72}$$

Similarly eliminating W from Eqs. (9.66) and (9.71), we have

$$\frac{\bar{L}_m}{\bar{G}_{m+1}} = \frac{(H_{G_{m+1}} - Q'')}{(H_{L_m} - Q'')} \tag{9.73}$$

Hence from Eqs. (9.72) and (9.73), we get

$$\frac{\bar{L}_m}{\bar{G}_{m+1}} = \frac{(y_{m+1} - x_W)}{(x_m - x_W)} = \frac{(H_{G_{m+1}} - Q'')}{(H_{L_m} - Q'')} \tag{9.74}$$

From Eq. (9.66), we have

$$\frac{\bar{L}_m}{\bar{G}_{m+1}} = 1 + \left(\frac{W}{\bar{G}_{m+1}}\right)$$

Hence, rearrangement of Eq. (9.74) using Eq. (9.66) gives

$$\frac{W}{\bar{G}_{m+1}} = \frac{(y_{m+1} - x_W)}{(x_m - x_W)} \tag{9.75}$$

$$\therefore \qquad \frac{\bar{L}_m}{W} = \frac{(H_{G_{m+1}} - Q'')}{(H_{G_{m+1}} - H_{L_m})} = \frac{y_{m+1} - x_W}{(y_{m+1} - x_m)} \tag{9.76}$$

Equation (9.76) represents a line passing through (x_W, Q''), (x_m, H_{L_m}) and (y_{m+1}, $H_{G_{m+1}}$) where Q'' represents net heat out /Net moles out and x_W denotes moles of A out/net moles out. Now let us consider the fractionator as single unit and make mass and energy balances;

Total mass balance gives

$$F = D + W \tag{9.77}$$

A component balance gives

$$FZ_F = Dy_D + Wx_W \tag{9.78}$$

An enthalpy balance gives

$$F \cdot H_F = (DQ' + WQ'') \tag{9.79}$$

(Neglecting Q_L, the heat loss)

Eliminating F from Eqs. (9.77), (9.78) and (9.79) we get

$$\frac{D}{W} = \frac{(Z_F - x_W)}{(Z_D - Z_F)} = \frac{(H_F - Q'')}{(Q' - H_F)} \tag{9.80}$$

Equation (9.80) represents a line passing through (Z_D, Q'), (Z_F, H_F) and (x_W, Q'') In other words,

$$F = \Delta_D + \Delta_W \tag{9.81}$$

The schematic representation of enthalpy concentration and distribution diagrams for determination of number of stages for a total condensation of distillate vapour is shown in Fig. 9.17(a).

Steps involved

1. Draw H vs x, y diagram and the equilibrium curve.
2. Locate z_D, y_D and x_W in both the diagrams and draw vertical lines from z_D in positive y-axis direction and from x_W in the negative y-axis direction.
3. Locate $F(x_F, H_F)$ in the H–x, y diagram.
4. Obtain Q' using the given reflux ratio, $R = \dfrac{(Q' - H_{G_1})}{(H_{G_1} - H_{L_0})}$

   where H_{G_1}, H_{L_0} indicate the enthalpy of vapour and liquid correspond to the distillate composition for a total condensation.
5. In cases where the reflux ratio is not given, an optimum reflux ranging from 1.5 to 2 times the minimum reflux can be chosen.
6. To determine the *minimum reflux*, several lines can be drawn through the feed point F in the entire range of x and projected downwards from both H vs x and H vs y curves to the x-y diagram as shown in Fig. 9.17(b) and one such horizontal line in x-y diagram will be a tie line. This line is extended to cut the vertical line drawn at z_D and this intersection point corresponds to the value of Q' at minimum reflux and the value of R estimated is R_{min}.

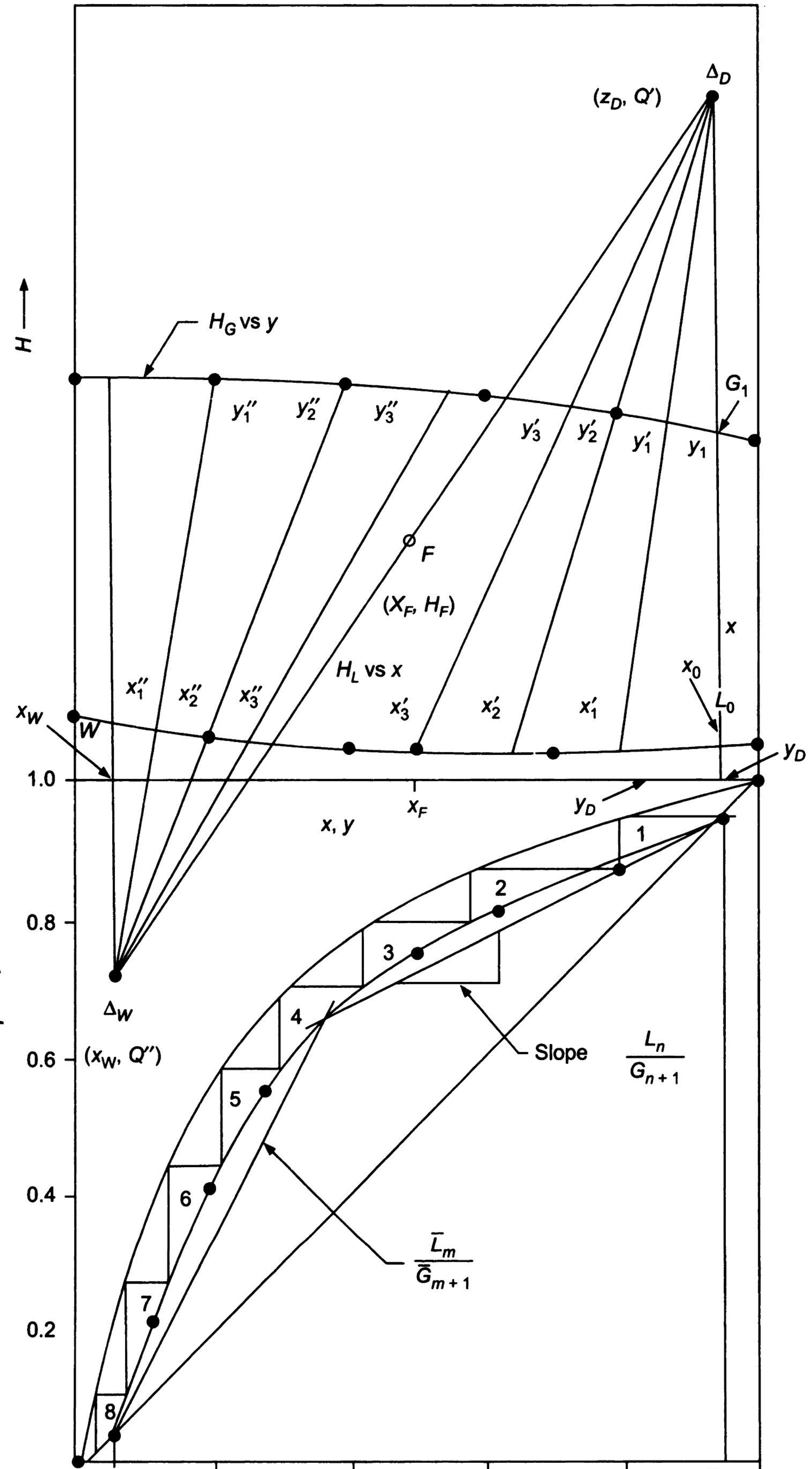

Fig. 9.17(a) Determination of number of stages by Ponchon–Savarit method.

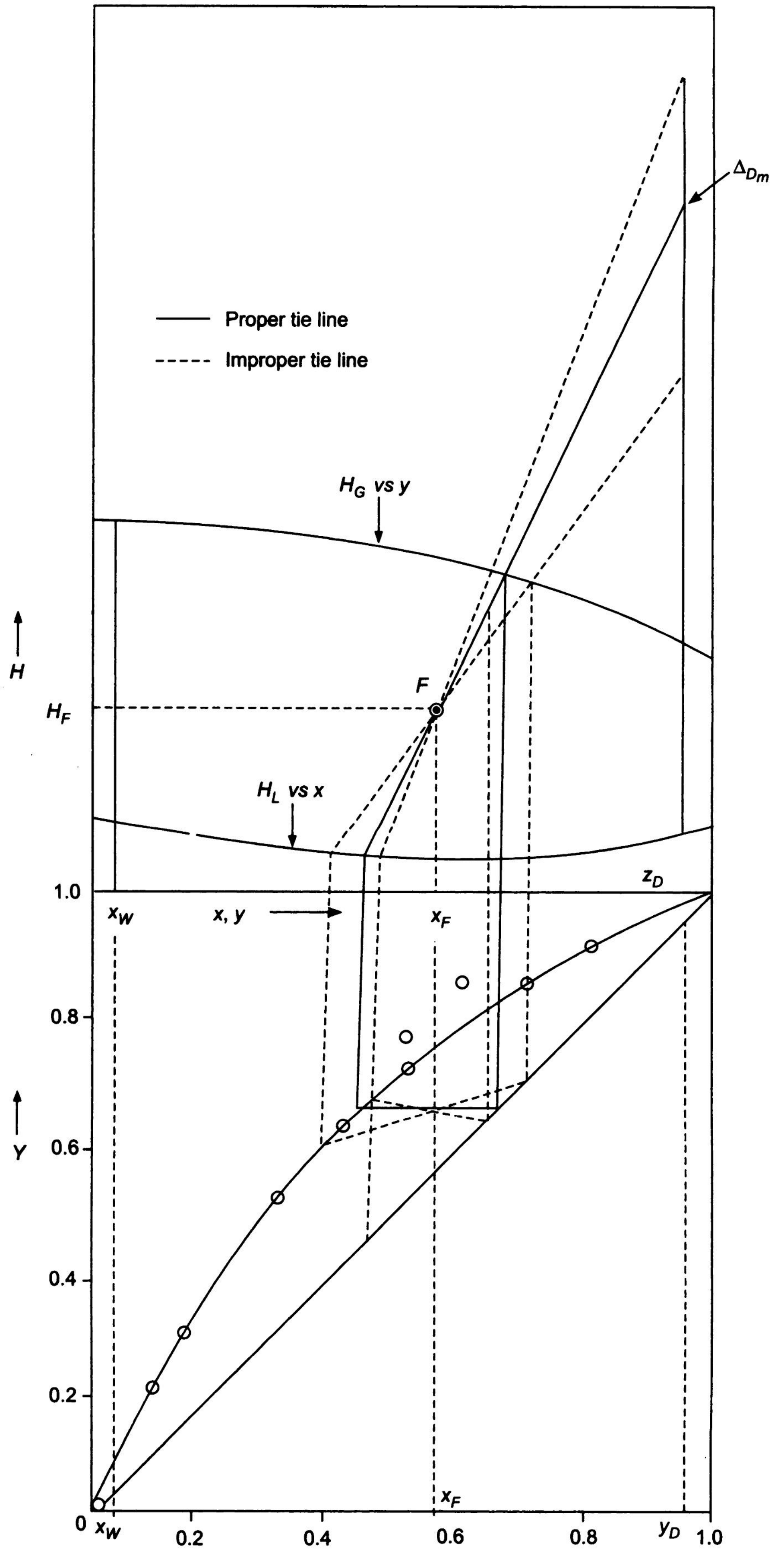

Fig. 9.17(b) Determination of minimum reflux in Ponchon–Savarit method.

7. Locate Δ_D (y_D, Q') using the H_{G_1}, H_{L_0} and reflux ratio.
8. Join Δ_D with F and project it to cut the vertical line at x_W and that point is Δ_W.
9. Draw arbitrarily several lines both from Δ_D and Δ_W to cut both the curves. The values taken from H vs y give y' and the line from H vs x gives x'. For each line drawn we will have a set of x' and y' values with which we can construct the operating line in the distribution diagram for both enriching and stripping sections.
10. Draw the equilibrium curve and plot x', y' data obtained from step (9).
11. By stepwise construction starting from point D, between the equilibrium curve and operating line up to W, the number of stages for the desired separation is determined.

9.12.2 McCabe–Thiele Method

When systems exhibit ideal behaviour, the time-consuming Ponchon–Savarit method of determining the number of ideal stages, can be replaced with the following technique. Let us begin our analysis by considering enriching section for total condensation of distillate as shown in Fig. 9.18.

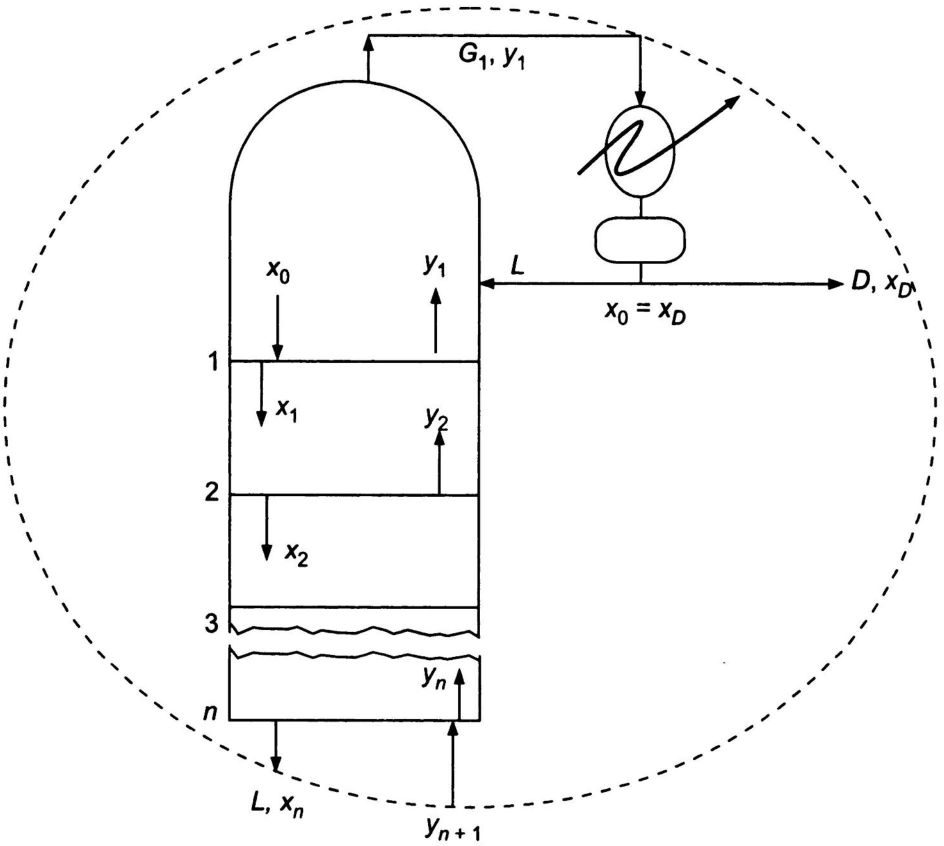

Fig. 9.18 Enriching section of a fractionator.

Assuming the application of equimolar counter diffusion, i.e. the molar flow rates are assumed to be constant for both the vapour and liquid steams irrespective of the stages, we get

$$G_1 \approx G_2 \approx G_{n+1} \approx G$$

A total material balance gives

$$G = L + D \tag{9.82}$$

Let the external reflux ratio R be given by

$$R = \frac{L_0}{D} \approx \frac{L}{D}$$

then,

$$G = DR + D = D(R + 1) \tag{9.83}$$

A component balance for A in enriching section gives

$$Gy_{n+1} = Lx_n + Dy_D \tag{9.84}$$

i.e.

$$y_{n+1} = \left[\frac{L}{G}\right]x_n + \left[\frac{D}{G}\right]x_D \tag{9.85}$$

$$\frac{L}{G} = \frac{L}{L+D} = \frac{\left(\frac{L}{D}\right)}{\left(\frac{L}{D}\right)+\left(\frac{D}{D}\right)} = \frac{R}{R+1} \tag{9.86}$$

$$\frac{D}{G} = \frac{D}{L+D} = \frac{1}{R+1} \tag{9.87}$$

∴

$$y_{n+1} = \left[\frac{R}{R+1}\right]x_n + \left[\frac{1}{R+1}\right]x_D \tag{9.88}$$

Equation (9.88) represents the operating line for enriching section, which has a slope of $\left[\frac{R}{R+1}\right]$ and an intercept of $\left[\frac{x_D}{R+1}\right]$. If $x_n = x_D$, then substituting in Eq. (9.88), we get

$$y_{n+1} = \left(\frac{1}{R+1}\right)(Rx_D + x_D) = y_D \tag{9.89}$$

i.e. when $x_n = x_D$, $y_{n+1} = x_D$.

Hence, this line passes through $x = y = x_D$, i.e. it lies on the diagonal. This point on the diagonal and the y-intercept $\left[\frac{x_D}{R+1}\right]$ permit us the construction of operating line for enriching section.

Let us consider the stripping section as shown in Fig. 9.19.

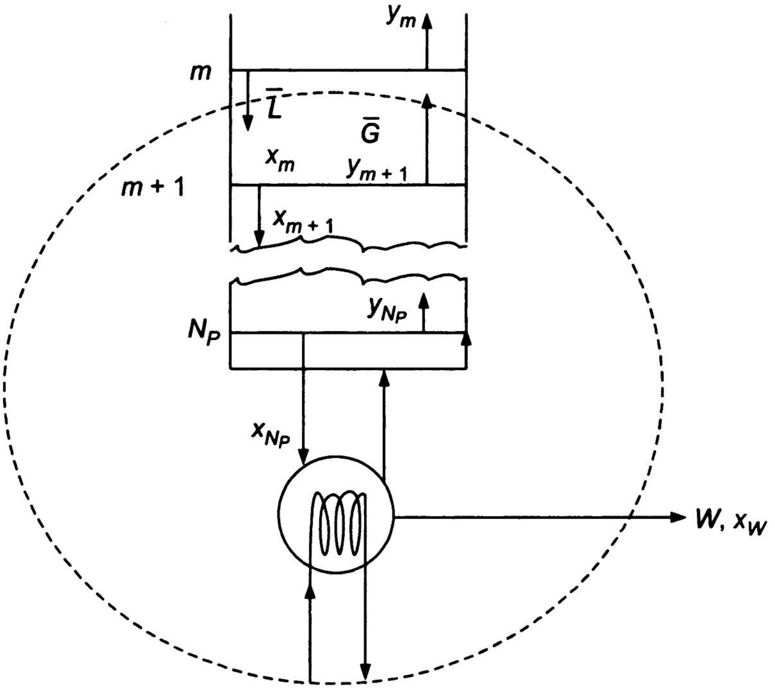

Fig. 9.19 Stripping section.

Material balance gives

$$\bar{L} = \bar{G} + W \tag{9.90}$$

A component balance for A gives,

$$\bar{L}x_m = \bar{G}y_{m+1} + Wx_W \tag{9.91}$$

$\therefore$
$$y_{m+1} = \left(\frac{\bar{L}}{\bar{G}}\right)x_m - \left(\frac{W}{\bar{G}}\right)x_W \tag{9.92}$$

i.e.
$$y_{m+1} = \left(\frac{\bar{L}}{\bar{L} - W}\right)x_m - \left(\frac{W}{\bar{L} - W}\right)x_W \tag{9.93}$$

Equation (9.93) describes the operating line for stripping section. The operating line has a slope of $\left(\frac{\bar{L}}{\bar{L} - W}\right)$ and an intercept of $-\left(\frac{W}{\bar{L} - W}\right)$.

Let us assume that $x_m = x_W$ (Reboiler)

$\therefore$
$$y_{m+1} = \left(\frac{\bar{L}}{\bar{L} - W}\right)x_W - \left(\frac{W}{\bar{L} - W}\right)x_W = x_W \tag{9.94}$$

i.e.
$$x_m = y_{m+1} = x_W$$

Hence, the operating line passes through the point $x = y = x_W$ (i.e. it lies on the diagonal). Having seen the analysis of enriching section and stripping section separately, let us analyze the feed plate, f, shown in Fig. 9.20.

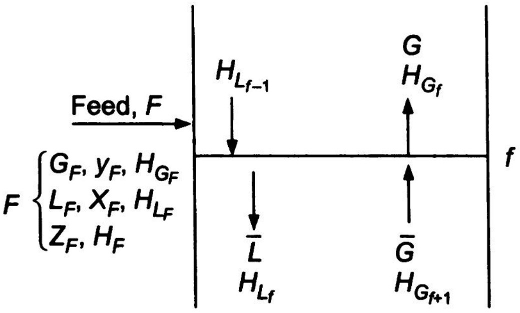

Fig. 9.20 Feed plate section.

A mass balance on feed plate gives

$$F + L + \bar{G} = G + \bar{L} \tag{9.95}$$

$$(L - \bar{L}) = (G - \bar{G}) - F \tag{9.96}$$

i.e.
$$(\bar{L} - L) = (\bar{G} - G) + F \tag{9.97}$$

Enthalpy balance on feed plate gives

$$F \cdot H_F + L \cdot H_{L_{f-1}} + \bar{G} \cdot H_{G_{f+1}} = G \cdot H_{G_f} + \bar{L} \cdot H_{L_f} \tag{9.98}$$

As an approximation, $H_{G_f} \approx H_{G_{f+1}} = H_G$ and $H_{L_{f-1}} = H_{L_f} = H_L$ (9.99)

∴
$$(\bar{L} - L)H_L = (\bar{G} - G)H_G + FH_F \tag{9.100}$$

i.e.
$$\frac{(\bar{L} - L)}{F} H_L = \frac{(\bar{G} - G)}{F} H_G + H_F \tag{9.101}$$

Substituting for $(\bar{L} - L)$ from Eq. (9.97) we get

$$\left[\frac{(\bar{G} - G) + F}{F}\right] HL = \frac{(\bar{G} - G)}{F} H_G + H_F \tag{9.102}$$

i.e.
$$\frac{(\bar{G} - G)}{F} H_L + H_L = \frac{(\bar{G} - G)}{F} H_G + H_F \tag{9.103}$$

$$\frac{(\bar{G} - G)}{F}(H_L - H_G) = (H_F - H_L) \tag{9.104}$$

$$\left[\frac{(\bar{G} - G)}{F}\right] = \frac{(H_F - H_L)}{(H_L - H_G)} \tag{9.105}$$

Substituting for $(\bar{G} - G)$ from Eq. (9.97), we get

$$\frac{(\bar{L} - L) - F}{F} = \frac{(H_F - H_L)}{(H_L - H_G)} \tag{9.106}$$

i.e.
$$\frac{(\bar{L}-L)}{F}=\frac{(H_F-H_L)}{(H_L-H_G)}+1 \tag{9.107}$$

$$=\frac{(H_G-H_F)}{(H_G-H_L)} \tag{9.108}$$

Let us now define $\frac{(H_G-H_F)}{(H_G-H_L)}$ as q, where q is the quantity of heat required to convert one mole of feed at its thermal condition to a saturated vapour, to the molal latent heat of vaporization.

$\therefore$
$$q=\frac{(\bar{L}-L)}{F}=\frac{(H_G-H_F)}{(H_G-H_L)} \tag{9.109}$$

Similarly,
$$\frac{(\bar{G}-G)}{F}=\frac{(\bar{L}-L)}{F}-1=(q-1) \tag{9.110}$$

i.e.
$$(\bar{G}-G)=F(q-1) \tag{9.111}$$

A solute balance above feed plate gives

$$Gy=Lx+Dx_D \tag{9.112}$$

A solute balance below feed plate gives

$$\bar{G}y=\bar{L}x-Wx_W \tag{9.113}$$

Subtracting Eq. (9.112) from Eq. (9.113), we have

$$(\bar{G}-G)y=(\bar{L}-L)x-(Dx_D+Wx_W) \tag{9.114}$$

Total component balance for the distillation column gives

$$FZ_F=Dx_D+Wx_W \tag{9.115}$$

Substituting Eqs. (9.109), (9.111) and (9.115) in Eq. (9.114), we get

$$\cancel{F}(q-1)y=\cancel{F}q\cdot x-\cancel{F}Z_F$$

$\therefore$
$$y=\frac{q}{(q-1)}x-\frac{Z_F}{(q-1)} \tag{9.116}$$

Equation (9.116) is the equation for feed line. It has a slope of $q/q-1$ and passes through $y=x=Z_F$.

The various values of slope obtained under different thermal conditions of feed are given below and shown in Fig. 9.21.

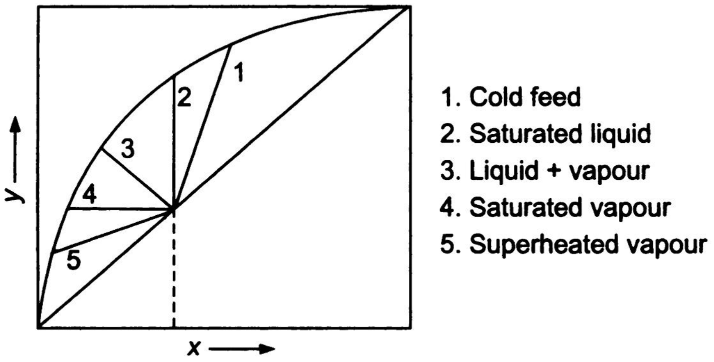

Fig. 9.21 Feed line for different thermal conditions of feed.

Feed condition	G_F	L_F	H_{G_F}	H_{L_F}	Enthalpy of feed, H_F	$q = \left[\frac{(H_G - H_F)}{(H_G - H_L)}\right]$	$\frac{q}{q-1}$
Liquid below boiling point	0	F	–	H_F	$H_F < H_L$	> 1.0	>1.0
Saturated liquid	0	F	–	H_F	$H_F = H_L$	1.0	∞
Liquid + vapour	G_F	L_F	H_G^*	H_L^*	$H_G^* > H_F > H_L^*$	1.0 to 0	$\frac{L_F}{L_F - F}$
Saturated vapour	F	0	H_F	–	$H_F = H_G$	0	0
Superheated vapour	F	0	H_F	–	$H_F > H_G$	< 0	1.0 to 0

* indicates H_G and H_L are enthalpies per mole of individual phases.

Determination of q is as follows:

(i) *Cold feed*

From Eq. (9.109), we have

$$q = \frac{(H_G - H_F)}{(H_G - H_L)}$$

Let T_b be the boiling point of mixture and T_F be the feed temperature. Let H_G and H_L be the enthalpies of saturated vapour and liquid respectively. If λ is the latent heat of vaporization, $C_{P,L}$ is the specific heat of feed liquid and T_0 is the reference temperature, then

$$H_G = C_{P,L}(T_b - T_0) + \lambda$$

$$H_F = C_{P,L}(T_F - T_0) \text{ and } (H_G - H_L) = \lambda \tag{9.117}$$

$$q = \frac{[C_{P,L}(T_b - T_0) + \lambda] - [C_{P,L}(T_F - T_0)]}{\lambda} = \frac{[C_{P,L}(T_b - \cancel{T_0} - T_F - \cancel{T_0}) + \lambda]}{\lambda} \tag{9.118}$$

i.e.

$$q = 1 + \frac{C_{P,L}(T_b - T_F)}{\lambda} \tag{9.119}$$

(ii) *Saturated liquid*

$$q = \frac{(H_G - H_F)}{(H_G - H_L)} \tag{9.120}$$

For saturated liquid $H_F = H_L$, $\therefore$ $q = 1.0$

(iii) *Mixture of liquid and vapour*

Let x be the mole fraction of liquid in feed in the case of liquid + vapour mixture. Then,

$$H_F = xH_L + (1 - x)H_G \tag{9.121}$$

Therefore,
$$q = \frac{\left[\cancel{H_G} - xH_L - \cancel{H_G} + xH_G\right]}{\left(H_G - H_L\right)}$$

$$= \frac{x\cancelto{1.0}{(H_G - H_L)}}{\cancel{(H_G - H_L)}} = x \tag{9.122}$$

(iv) *Saturated vapour*

$$q = \frac{(H_G - H_F)}{(H_G - H_L)} \tag{9.123}$$

For saturated vapour $H_F = H_G$, $\therefore\ q = 0$

(v) *Superheated vapour*

Let $C_{P,V}$ be the specific heat of feed vapour

$$H_G = H_G$$

$$H_F = H_G + C_{P,V}\,(T_F - T_b) \tag{9.124}$$

$$q = \frac{\cancel{H_G} - [\cancel{H_G} + C_{P,V}(T_F - T_b)]}{\lambda} = -\frac{C_{P,V}(T_F - T_b)}{\lambda} \tag{9.125}$$

Steps involved in the determination of number of trays

The equilibrium curve along with the operating lines for both enriching and stripping sections to determine the number of stages is shown in Fig. 9.22.

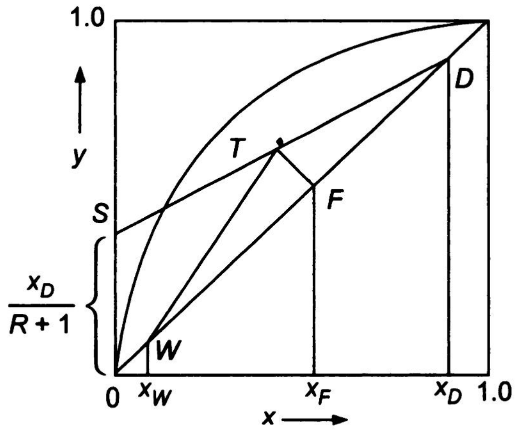

Fig. 9.22 Determination of number of stages by McCabe–Thiele method.

1. Draw the equilibrium curve and diagonal.
2. Locate *F*, *D* and *W* corresponding to feed, distillate and residue compositions based on more volatile component.
3. Estimate $x_D/(R + 1)$ and locate it on *y*-axis as *S*.
4. Join *SD*, this is the operating line for enriching section.
5. From *F* draw *q*-line depending on feed condition. Let it cut the operating line for enriching section at *T*.

6. Join *TW*-operating line for stripping section.
7. Construct stepwise from *D* to *W* and the steps so constructed will give the number of stages.

9.13 LOCATION OF FEED TRAY

For an optimal design or when a column is designed first (wherein one goes for optimal design) the feed tray is located at the intersection of operating lines of enriching and exhausting sections of the tower.

However, we may at times use a column which has been designed with some other objectives. Whenever the quality and condition of feed is fixed along with reflux ratio, x_D and x_W, the operating lines are fixed. It may so happen that in an existing column, the location of feed nozzle is fixed and it may not really lie at the optimal point as shown in Figs. 9.23(a), (b) and (c).

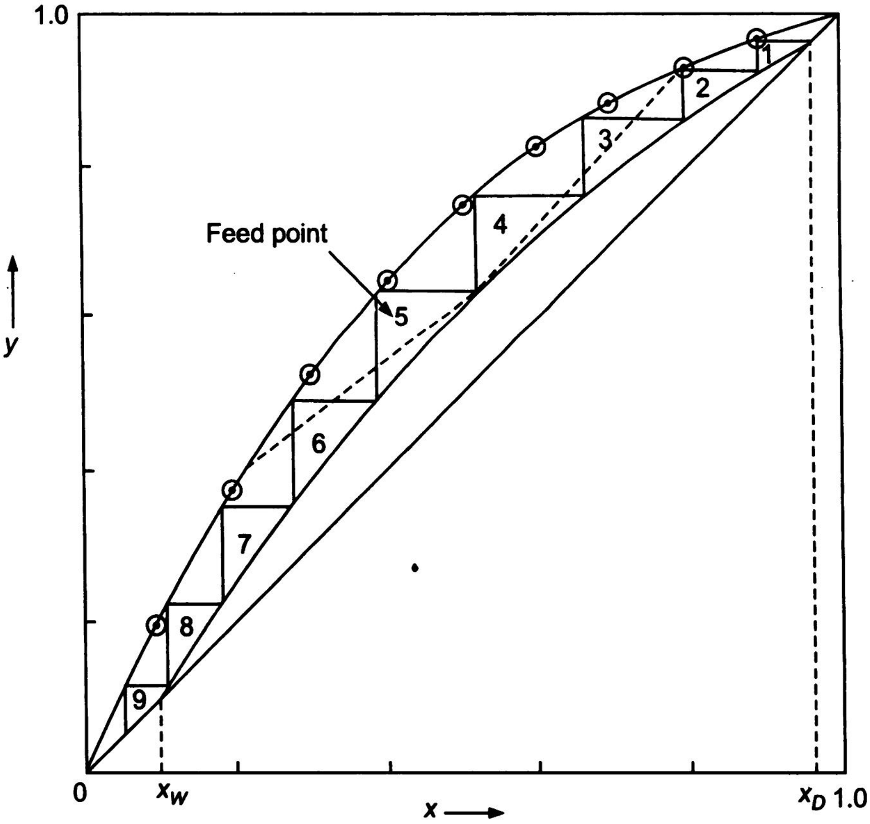

Fig. 9.23(a) Optimal feed location.

The point of intersection of the two operating lines is generally believed to be the point that demarcates enriching and exhausting sections. This normally occurs in the newly designed columns from a specific x_w, x_D, x_F and condition of feed. However, in an existing column, designed for a different utility, the feed point location is fixed and may not be at the optimum location. Further, the feed entry point will not demarcate the enriching and exhausting sections.

Generally when the reflux ratio and the x_D values are fixed, the operating line for enriching section is fixed. Further, when the x_w and the condition of the feed are fixed the operating line for exhausting section is fixed.

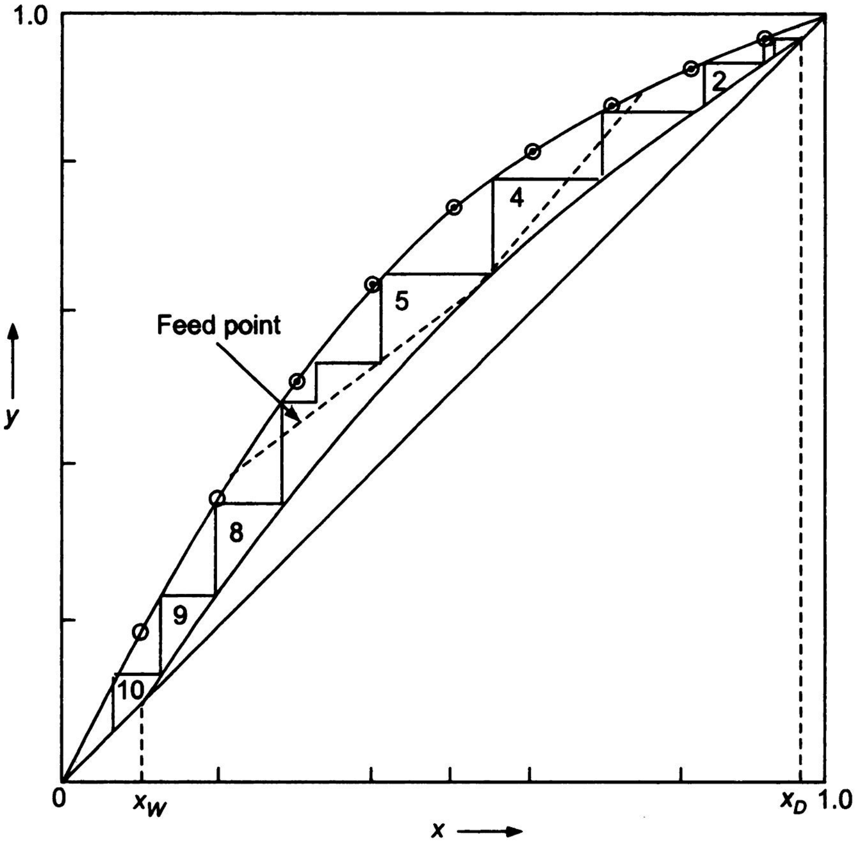

Fig. 9.23(b) Delayed feed entry.

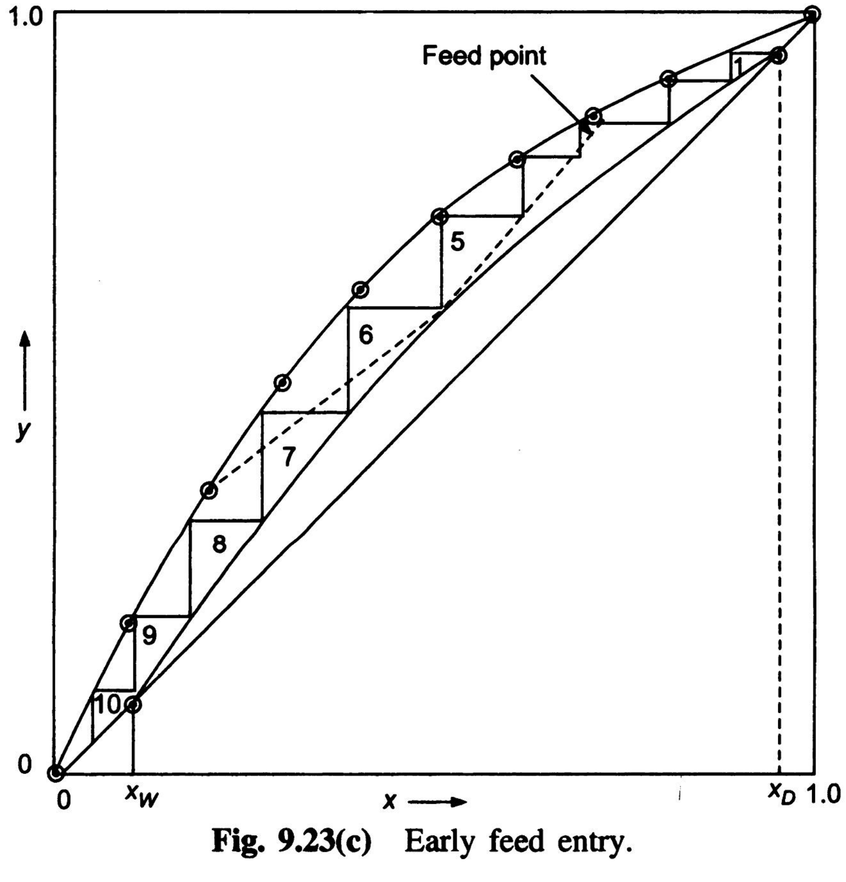

Fig. 9.23(c) Early feed entry.

Once a feed enters a specific plate, below the point of intersection of operating lines and q-line (in an existing column), from the top plate to feed entry point, the operating line for enriching section is to be used and subsequently the operating line for exhausting section. Such an arrangement indicates a *delayed feed entry*. If the feed enters at a specific plate, above the point of intersection of operating lines and q-line (in an existing column), from the top plate to feed entry point, the operating line for enriching section is to be used and subsequently the operating line for exhausting section. This arrangement indicates an *early feed entry*. In both the cases the number of stages estimated will always be more compared to the number of stages estimated with feed entering exactly at the point of intersection of operating lines and q-line.

Consider the above three figures,

Figure 23(a): Optimal design with 9 plates and the 5th plate is feed plate.

Figure 23(b): An existing column with 10 plates and feed enters at 7th plate.

Figure 23(c): An existing column with 10 plates and feed is introduced at 3rd plate.

9.14 REFLUX RATIO

It is one of the important operating parameters in distillation, by which the quality of the products can be changed. Let us deal with the relationship between reflux ratio and the number of trays in the tower.

9.14.1 Determination of Minimum Reflux Ratio

To determine the minimum reflux ratio, draw the q-line from F to cut the equilibrium curve at T'. Join DT' and extend it to intersect on y-axis and indicate it as S'. OS' gives $\left[\frac{x_D}{R_{\min}+1}\right]$ from which $R_{\min}$ is estimated as shown in Fig. 9.24.

Normally at $R_{\min}$ condition, the number of stages will be infinity as the equilibrium curve and operating line get pinched.

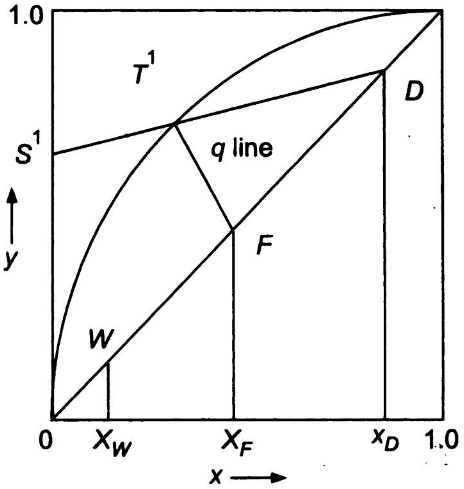

Fig. 9.24 Determination of minimum reflux ratio.

Steps involved in the determination of minimum reflux

1. Draw the equilibrium curve and diagonal.
2. Locate F, D and W corresponding to feed, distillate and residue compositions based on more volatile component.
3. Draw the q-line from F and allow it to intersect the equilibrium curve at T'.
4. Join $T'D$ and allow it to intersect the y-axis at S'.
5. OS' corresponds to $\left[\dfrac{y_D}{R_{\min}+1}\right]$ from which $R_{\min}$, the minimum reflux ratio is estimated.

9.14.2 Total Reflux

At total reflux, all the distillate is returned to the column and no product is taken out as distillate.

i.e.

$$D = 0$$

$\therefore$

$$R = \frac{L}{D} = \infty$$

Hence, the operating line [Eq. (9.88)] for enriching section is

$$y_{n+1} = \left[\frac{R}{R+1}\right]x_n + \left[\frac{1}{R+1}\right]x_D$$

becomes,

$$y_{n+1} = x_n \tag{9.126}$$

i.e. it merges with the diagonal ($x = y$ line) for both enriching and stripping sections. Under such circumstances, the minimum number of theoretical stages can be estimated by the same graphical procedure described in the Section of 9.12.2. For systems where the relative volatility is constant and under total reflux conditions the theoretical number of stages needed could be estimated analytically.

We know that for a binary system, $\alpha_{AB} = \dfrac{\left[\dfrac{y_A}{x_A}\right]}{\left[\dfrac{y_B}{x_B}\right]} = \dfrac{\left[\dfrac{y_A}{y_B}\right]}{\left[\dfrac{x_A}{x_B}\right]}$

$\therefore$

$$\frac{y_A}{y_B} = \alpha_{AB}\frac{x_A}{x_B}$$

$$\frac{y_A}{1-y_A} = \alpha_{AB}\frac{x_A}{1-x_A}$$

Let us apply this relationship to $(n + 1)$th plate

$$\therefore \qquad \frac{y_{n+1}}{(1-y_{n+1})} = \alpha \frac{x_{n+1}}{(1-x_{n+1})} \tag{9.127}$$

At total reflux $D = 0$ and $\frac{L}{G} = \frac{R}{R+1} = 1.0$

Hence, from Eq. (9.126), we get

$$(y_{n+1}) = x_n \tag{9.128}$$

When $n = 0$, i.e. at the top of the column, $x_n = x_0$

$y_1 = x_0 = x_D$, when total condenser is used.

Substituting for y_{n+1} in terms of x_n in Eq. (9.128), we get

$$\therefore \qquad \frac{x_n}{(1-x_n)} = \alpha_{AB} \frac{x_{n+1}}{(1-x_{n+1})} \tag{9.129}$$

When $n = 0$,

$$\frac{x_0}{(1-x_0)} = \alpha_{AB} \frac{x_1}{(1-x_1)} \tag{9.130}$$

When $n = 1$,

$$\frac{x_1}{(1-x_1)} = \alpha_{AB} \frac{x_2}{(1-x_2)} \tag{9.131}$$

When $n = n - 1$,

$$\frac{x_{n-1}}{(1-x_{n-1})} = \alpha_{AB} \frac{x_n}{(1-x_n)} \tag{9.132}$$

Substituting for $x_1, x_2, \ldots, x_{n-1}$ from Eqs. (9.131) and (9.132), we get

$$\frac{x_0}{(1-x_0)} = (\alpha_{AB})^n \frac{x_n}{(1-x_n)} \tag{9.133}$$

Substituting $n = N_P + 1$ (last stage, i.e. reboiler), we get

$$\frac{x_0}{(1-x_0)} = (\alpha)^{N_P+1} \frac{x_{N_P+1}}{(1-x_{N_P+1})} \tag{9.134}$$

As $(N_P + 1)$th stage accounts for reboiler, $x_{N_P+1} = x_w$

i.e.

$$\frac{y_D}{(1-y_D)} = (\alpha)^{N_P+1} \frac{x_W}{(1-x_W)} \tag{9.135}$$

Equation (9.135) is called *Fenske equation.* To apply this equation, α, the relative volatility must be fairly constant and the column has to be operated under total reflux conditions. This may not be possible in industries, but has theoretical importance.

9.14.3 Optimum Reflux Ratio

At minimum reflux ratio the column requires infinite number of stages or trays. However, as reflux ratio increases from minimum, for a given feed and specified quality of distillate and residue, the number of stages or trays decrease. At minimum reflux ratio when the stages or trays are infinite, the fixed cost and the maintenance cost are also infinite. However, the operating cost for operating condenser, reboiler etc. is the least. When the reflux increases, the trays or stages reduce but the column diameter has to be increased to handle larger capacities of liquid being recycled. The size of other accessories like condenser and reboiler increase which will result in a higher requirement of cooling water or heating. Ultimately this will result in a higher operating cost. Thus, the total cost which includes both operating cost and fixed cost, vary with reflux ratio and reach a minimum value for a certain reflux ratio which is called the ***optimum or economic reflux ratio***. This value is normally in the range of 1.2 R_{min} to 1.5 R_{min}. This is shown in Fig. 9.25.

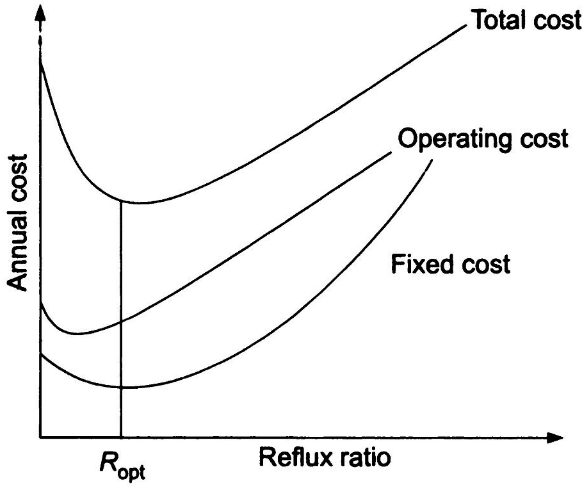

Fig. 9.25 Effect of Reflux ratio on cost.

9.15 REBOILERS

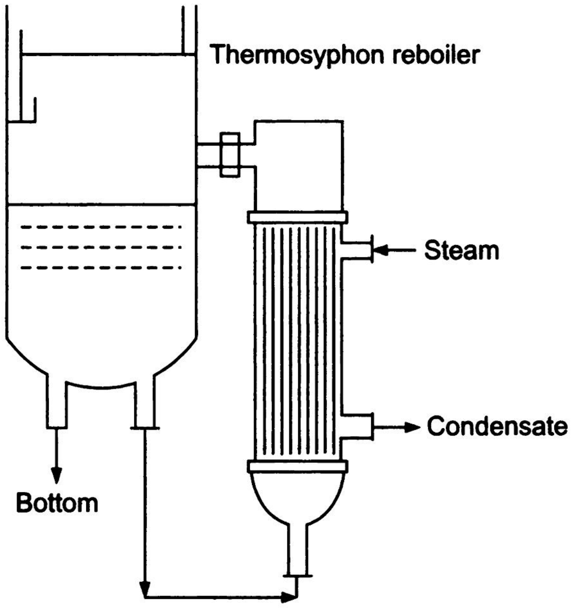

Fig. 9.26(a) Thermosyphon reboiler.

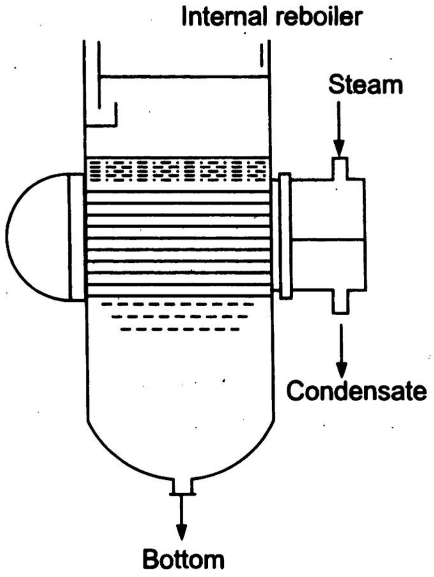

Fig. 9.26(b) Internal reboiler.

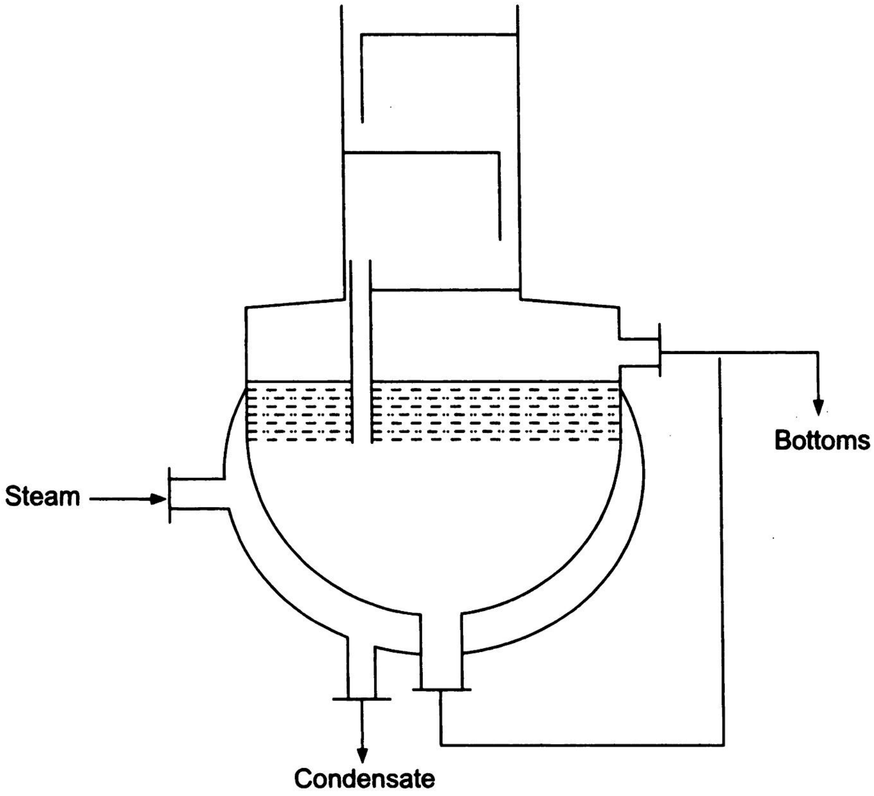

Fig. 9.26(c) Jacketed kettle reboiler.

They are heat exchangers of different configurations used to supply the heat to the liquid at the bottom of the column to vaporize them. In effect all the heat needed is basically supplied at the reboiler only.

A simple Jacketed kettle is one such reboiler which has a low heat transfer area and hence vapour generation capacity will also be poor.

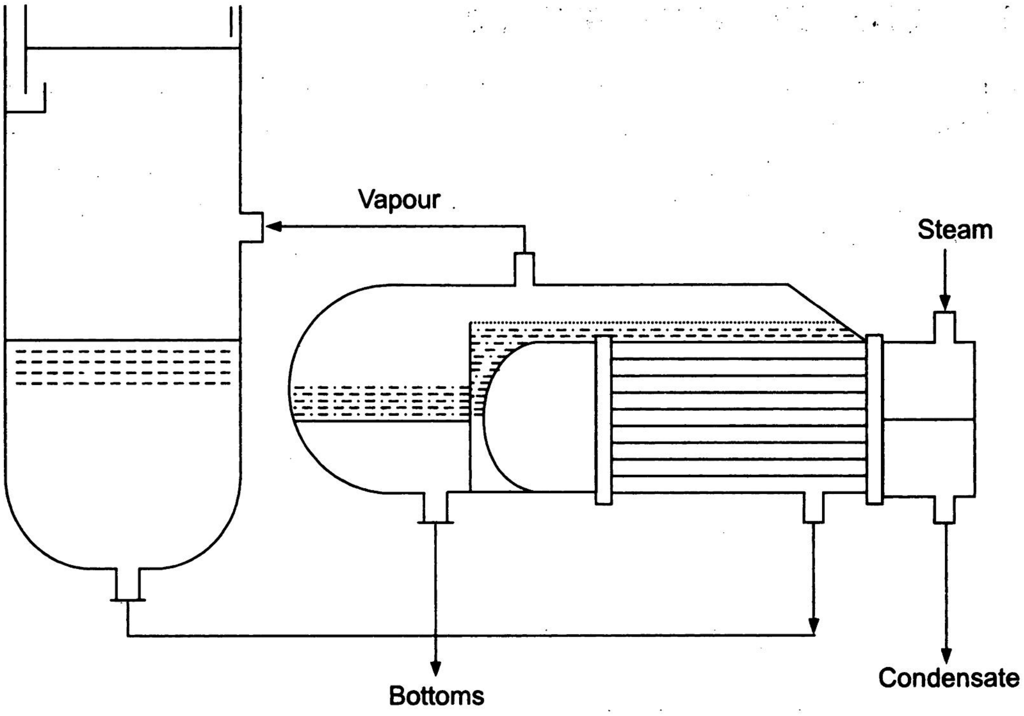

Fig. 9.26(d) External reboiler

Tubular heat exchangers (both of vertical and horizontal configurations) provide larger area of heat transfer. They can be placed inside the column or outside the column. When they are located inside, during cleaning of the exchanger, the distillation operation has to be stopped. However, when external reboilers are used, a standby exchanger is always kept which can be used during cleaning of the exchanger attached to the column. Thus, the distillation operation will proceed without any interruption. The liquid can flow either through the tube side or shell side. Reboilers can be heated by steam, oil or other hot fluids. Different types of reboilers are shown in Figs. 9.26(a)–(d).

9.16 CONDENSERS

The condensers are generally heat exchangers of horizontal orientation with coolant flowing through the tube side. However, in rare instances vertical condensers are used with the coolant flowing on either side of the tubes. They are placed above the tower in the case of laboratory scale units for gravity flow of the condensed reflux to the topmost tray. Sometimes they are placed at ground level for easy maintenance, in which case the liquid is pumped from accumulator to the top tray. The coolant is normally water. The condensers may either be a total condenser or a partial condenser. Whenever a partial condenser is used, the condensate is returned as reflux and the vapour from condenser is the main distillate product. The partial condenser itself acts as one stage for separation. In an existing distillation column, if one desires to have a highly enriched distillate (richer than the designed value) then one can resort to partial condensation and obtain an enriched product. However, when a column is being designed fresh it is always preferable to go for additional trays compared to partial condensation technique for enrichment.