We know that, $\frac{1}{K'_y} = \frac{1}{k'_y} + \frac{m}{k'_x}$

$$\frac{1}{K'_x} = \frac{1}{k'_x} + \frac{1}{mk'_y}$$

Similarly, H_{t_0G} and H_{t_0L} can also be written as

$$H_{t_0G} = H_{tG} + \left(\frac{mG}{L}\right)H_{tL} \tag{9.150}$$

$$H_{t_0L} = H_{tL} + \left(\frac{L}{mG}\right)H_{tG} \tag{9.151}$$

where m is the slope of the equilibrium curve.

9.18.1 Steps Involved in the Determination of the Height of Tower

1. Draw the equilibrium curve and diagonal.
2. Locate S, corresponding to $\frac{x_D}{R+1}$ in y-axis.
3. Locate F, D and W on diagonal corresponding to x_F, x_D and x_W
4. Join DS. This is the operating line for enriching section.
5. From F draw q-line. Let the point of intersection on operating line DS for enriching section be T.
6. Join TW. This is the operating line for stripping section.
7. From D to T and T to W draw lines of slope $\left[-\frac{k'_x}{k'_y}\right]$ or $\left[-\frac{K'_x}{K'_y}\right]$ (as the case may be) to obtain (x_i and y_i) or (x^* and y^*) and (x and y) values.
8. x and y values are read from operating lines and (x_i, y_i) or (x^*, y^*) values are read from equilibrium curve.
9. Evaluate $\int \frac{dx}{(x - x_i)}$ or $\int \frac{dy}{(y_i - y)}$ or $\int \frac{dx}{(x - x^*)}$ or $\int \frac{dy}{(y^* - y)}$ graphically to determine N_{tL} or N_{tG} or N_{toL} or N_{toG}.
10. H_{tL}, H_{tG}, H_{toG} or H_{toL} are determined with the help of liquid and vapour flow rates and mass transfer coefficients.
11. Height is then estimated based on the values from steps (9) and (10).
12. The tower diameter is normally set by the conditions at the top of stripping section because of large liquid flow rate at that point.

9.19 AZEOTROPIC DISTILLATION

This is a technique which is used for the separation of binary mixtures which are either difficult or impossible to separate by ordinary fractionation. This happens when either the mixture to be separated has a very low relative volatility, in which case one may require high reflux ratio and more number of trays, or when the mixture forms an azeotrope. Under such circumstances, a third component called an 'entrainer' is added to the binary mixture to form a new low boiling hetero-azeotrope with one of the components in the original mixture whose volatility is such that it can be separated from the other original constituent.

A typical example for this operation is presented in the flow diagram of Fig. 9.31 where the separation of acetic acid (BP: 118.1°C) and water (BP: 100°C) mixture is demonstrated. This mixture has a low relative volatility and hence separation by conventional methods is not economical. Here Butyl acetate, which is slightly soluble in water, is added to the mixture from the top of the column as an entrainer. It forms a 'hetero-azeotrope' with all the water in the feed and readily distills out from the high boiling acetic acid and the acetic acid leaves as a residue product. The hetero-azeotrope on condensation forms two insoluble layers which can easily be separated. The water layer obtained is saturated with ester and vice versa. The ester layer saturated with water is returned back to the column as a source of entrainer for further separation. The aqueous layer is also sent to another column to separate water and ester. The separated ester is also sent back as entrainer.

Sometimes the new azeotrope formed contains all the three constituents. In the dehydration of ethanol water mixture, benzene is added as an entrainer which gives a ternary azeotrope containing benzene (53.9 mole %), water (3.3 mole %) and ethanol (22.8 mole %) boiling at 64.9°C as distillate and ethanol (BP: 78.4°C) as residue. Benzene is separated and sent back to the top of the column as entrainer. Since water – ethanol are equally present in distillate, the mixture should be given a preliminary rectification to produce an alcohol rich binary azeotrope. Azeotropic distillation is shown in Fig. 9.31.

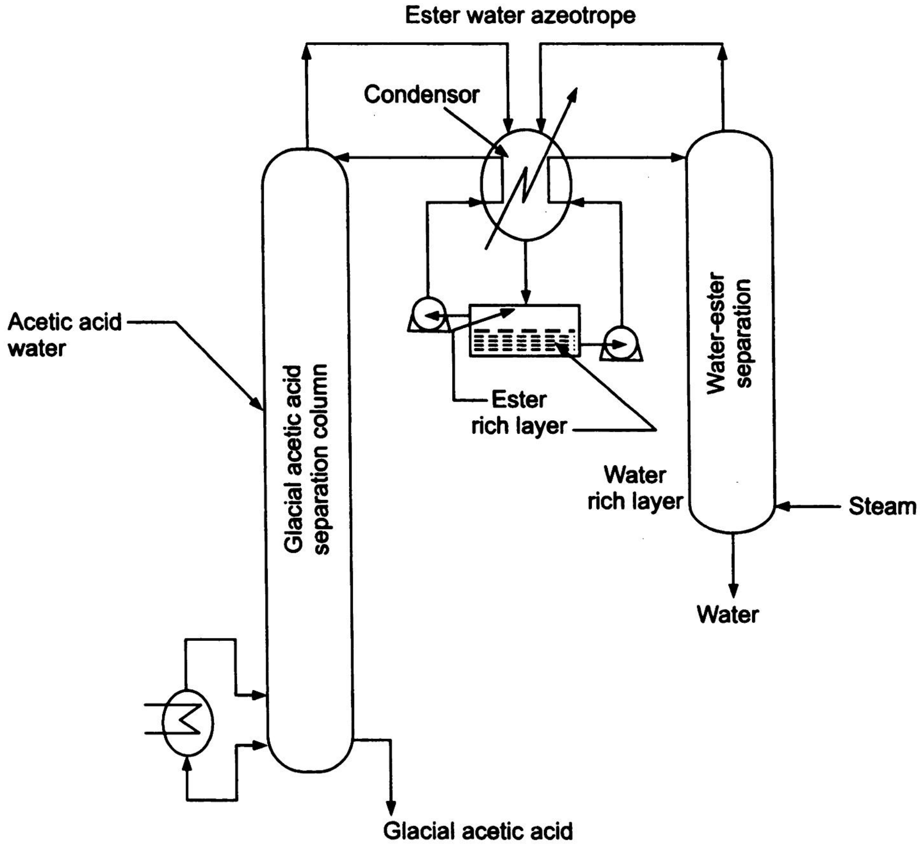

Fig. 9.31 Azeotropic distillation.

9.19.1 Desired Properties of an Entrainer for Azeotropic Distillation

(i) Should be cheap and easily available.
(ii) Chemically stable and inactive towards the solution to be separated.
(iii) Non-corrosive.
(iv) Non-toxic.
(v) Low latent heat of vaporization.
(vi) Low freezing point to facilitate storage and easy handling.
(vii) Low viscosity to provide high tray efficiency and minimum pumping cost.

9.20 EXTRACTIVE DISTILLATION

This method is also used under similar circumstances as in the case of azeotropic distillation. Here a third component called solvent is added, instead of entrainer, which alters the relative volatility of the original constituents, thus permitting the separation. The added solvent should have low volatility and not vaporized in the fractionator.

One such example is the separation of toluene (BP: 110.8°C) from isooctane (BP: 99.3°C). Their separation is relatively difficult. In the presence of Phenol (BP: 181.4°C), the relative volatility of isooctane increases, so that with an increase in phenol content, the separation becomes more and more easy. A typical flow diagram of the process is shown in Fig. 9.32.

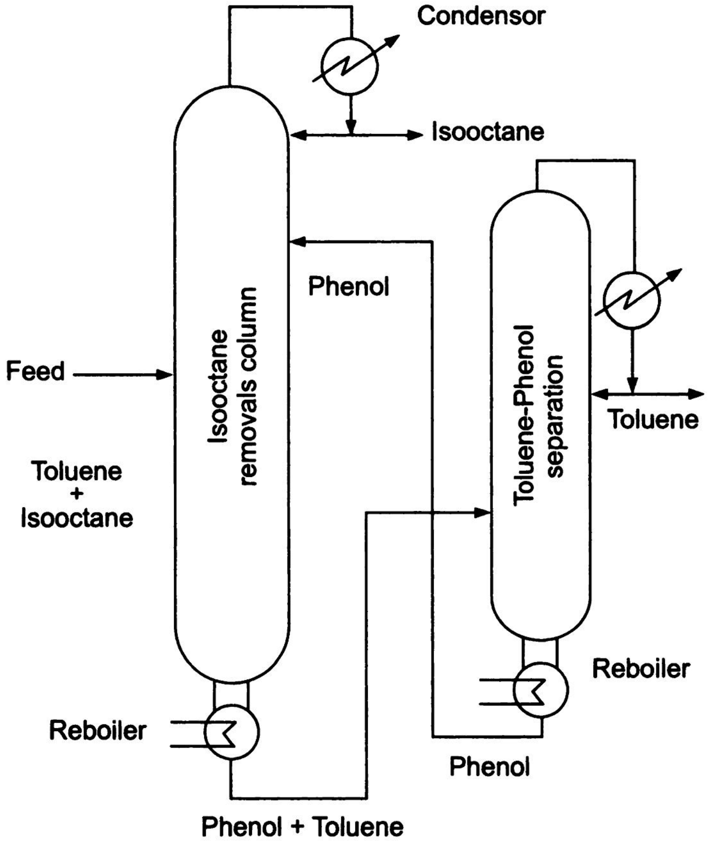

Fig. 9.32 Extractive distillation.

Here, the toluene–isooctane binary mixture is introduced in the middle of the column and phenol near the top of the column. Isooctane is readily distilled as an overhead product, while toluene and phenol are collected as residue. The residue from the tower is rectified in the auxiliary tower to separate toluene and phenol as distillate and residue respectively. Phenol is returned to the main column as solvent. Similarly a mixture of acetone (BP: 56.4°C) and methanol (BP: 64.7°C) can be separated by using Butanol (BP: 117.8°C) solvent.

9.20.1 Desired Properties of Solvent for Extractive Distillation

(i) High selectivity and capability to alter VLE for easy separation.
(ii) Ability to dissolve the components in the mixture.
(iii) Low volatility in order to prevent vaporization of solvent.
(iv) Easy separability, for easier removal of solvent.
(v) Non-corrosive.
(vi) Non-toxic.
(vii) Cheap and easily available.
(viii) Low freezing point.
(ix) Low viscosity.
(x) Chemical stability and inertness towards the components to be separated.

9.21 COMPARISON OF AZEOTROPIC AND EXTRACTIVE DISTILLATION

In both the processes an additional external agent is added, which is undesirable. Solvent to feed ratio in extractive distillation greater than 3 or 4 is found to be effective. Proper choice of material of construction and recovery technique are to be examined. However, of the two, extractive distillation is said to be more favoured than azeotropic distillation since (i) there is a greater choice of solvent, (ii) the smaller quantity of solvent to be volatilized. In spite of the above advantages, the azeotropic distillation is said to be more effective in the dehydration of ethanol from an 85.6 mole % Ethanol–water solution. In this case water is azeotroped with *n*–pentane and then separated rather than using extractive distillation with ethylene glycol as solvent.

9.22 LOW PRESSURE DISTILLATION

Whenever the heat sensitive materials are to be separated, as in the case of many organic mixtures, low pressure distillation will be effective. In this case the time of exposure of the substances to high temperature is kept minimum. Packed towers can be used for distillation under pressures of 50 to 250 mm Hg. Bubble cap and sieve trays can be used for pressure drops around 2.6 mm Hg and shower trays for pressure drops around 0.75 mm Hg. This is used in the separation of vitamins from animal and fish oils as well as the separation of plasticizers.