Chapter 1

Multi-Component Distillation

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1.2 Introduction

In a brief statement, distillation is a method of separating homogeneous mixtures based on the difference in volatilities of the components in the mixtures. Distillation is a unit operation, or a physical separation process, and not a chemical reaction. Distillation often produces an overhead distillate (can be either vapour or liquid) and a bottom product (almost always liquid), whose compositions differ from that of the feed.

Unlike absorption and stripping, the second phase created in distillation is normally by thermal vaporisation or condensation rather than by introducing a second phase that may contain an additional component or components not present in the feed mixture.

Distillation normally requires repeated vaporisation and condensation and is the most common method for the separation of fluid mixtures with fluid products. The driving force for such a separation is the difference between the compositions of the vapour and liquid phases at equilibrium. Distillation is suitable for a wide range of throughputs and feed concentration and can produce high product purity, e.g. in the industry of recovering and purifying small biomolecules such as ethanol, acetone, and n-butanol, and solvent (e.g. organic alcohols, acids, and ketones) in biochemical industry. However, in bioseparation of large biological metabolites, polymers or the products that are thermolabile, distillation is not often seen.

Multistage distillation is the most widely used industrial method for separating chemical mixtures. However, it is a very energy-intensive technique, especially when the relative volatility of the key components being separated is low (< 1.5). About two-thirds of the distillation energy was consumed by petroleum refineries, where distillation is used to separate crude oil into petroleum chemicals.

Even distillation is widely used in the chemical industry, but it may not be suitable for:

- Low molecular weight materials.
- High molecular weight heat-sensitive materials.
- Components with a low concentration.
- Classes of components.
- Mixtures with low relative volatility or which exhibit azeotropic behaviour.
- Mixtures of condensable and non-condensable components.

As for binary mixtures, vapour-liquid equilibrium is governed by equation 1.3.2, when there are more than two components in the mixture as in Figure 1.1, the calculations become more complicated. In principle, the design equations for binary distillation still apply but $N - 1$ balance equations in an N component mixture make solutions harder to obtain. It is also worth noting that short-cut distillation models are useful for initialising more rigorous models and design methods.

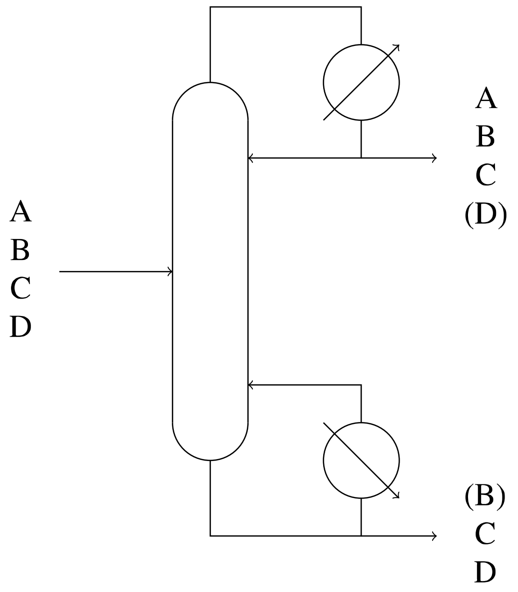


Figure 1.1: Multi-component distillation, where B and D are light and heavy key components and C is the intermediate boiling component.

1.3 Vapour-Liquid Equilibrium

The status of a matter existing in our physical world is determined by pressure and temperature. At the right temperature and pressure, a matter can be solid, liquid or vapour. In thermodynamics, the three phases can coexist - interpreted as phase equilibrium at a particular point of temperature and pressure. In distillation, we are normally concerned with the vapour-liquid equilibrium in multi-component mixtures.

We first introduce a concept - fugacity. In a pure liquid, the vapour generated by its escaping molecules necessarily has the same composition as that of the liquid. However, in a mixture, the composition of the vapour is not the same as that of the liquid, the vapour is richer in that component whose molecules have greater tendency to escape from the liquid phase. This tendency is measured by fugacity. Therefore, fugacity is a measure of the tendency of a component of a liquid mixture to escape, or vaporize, from the mixture. The fugacity of a component in a mixture is essentially the pressure that it exerts in the vapour phase when in equilibrium with the liquid mixture. It is the effective pressure of a real gas that differs from the behaviour of an ideal gas.

Thus, for each component in the mixture, when the vapour and liquid are in thermodynamic equilibrium, we have:

$$f_i^V = f_i^L \tag{1.3.1}$$

where f_i^V is the fugacity of component i in the vapour phase and f_i^L is the fugacity of the component i in the liquid phase.

Defining the vapour phase fugacity coefficient ϕ_i^V and the liquid phase fugacity coefficient

ϕ_i^L and activity coefficient γ_i, when the system pressure is P, we have:

$$f_i^V = \phi_i^V y_i P$$
$$f_i^L = \phi_i^L x_i P$$

or

$$f_i^L = \gamma_i x_i f_0 \tag{1.3.2}$$

where f_0 is the standard state fugacity of component i at the temperature of the system, x_i and y_i are the molar fractions of component i in liquid and vapour phases, respectively.

For moderate pressures f_0 is usually taken to be the saturated vapour pressure p_I^0

$$f_i^L = \gamma_i x_i P_i^0 \tag{1.3.3}$$

The above equations can be combined (using equation 1.3.1) to give an expression for the equilibrium 'constant' or K-value, K_i,

$$K_i = \frac{y_i}{x_i} = \frac{\phi_i^L}{\phi_i^V} = \frac{\gamma_i P_i^0}{\phi_i^V P} \tag{1.3.4}$$

At moderate pressure, $\phi_i^V \to 1$

$$K_i = \frac{\gamma_i P_i^0}{P} \tag{1.3.5}$$

When the liquid phase behaves as an ideal solution ($\gamma_i \to 1$), this expression simplifies to

$$K_i = \frac{P_i^0}{P} \tag{1.3.6}$$

which is the so called Raoult's Law.

Figure 1.2 shows an example of the vapour-liquid equilibrium in an ideal binary mixture: Benzene-Toluene mixture at 1 atm.

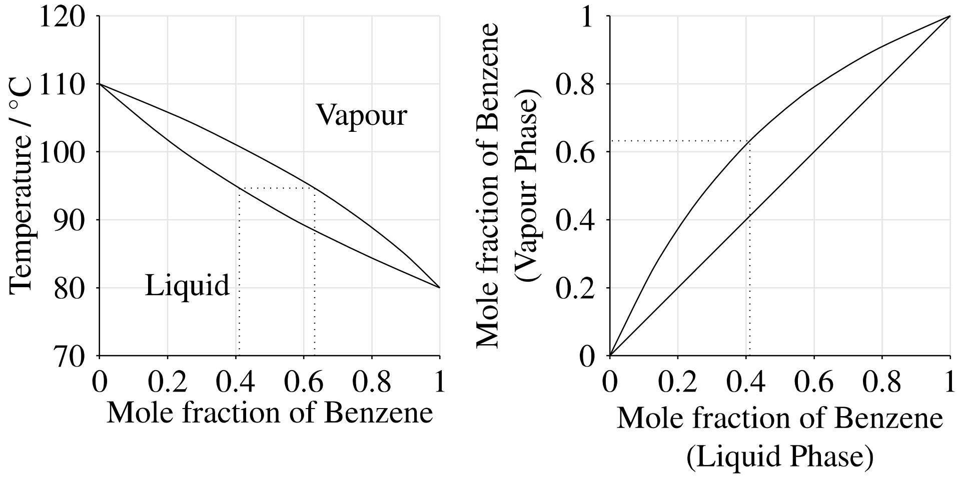


Figure 1.2: Vapour-liquid equilibrium of Benzene-Toluene mixture at 1 atm. The same equilibriumpair is shown in both styles of plot.

1.4 Short-cut methods for multi-component distillation

In short-cut design for multi-component distillation, the basic assumptions include,

1. constant relative volatility, which is the most important assumption for most problems although not often a good assumption for mixtures with non-ideal behaviour, and
2. constant molar overflow i.e. constant molar vapour and liquid flowrates.

There are a number of short-cut methods for simple columns including,

- Fenske equation: Minimum number of stages, N_{min}.
- Hengstebeck-Geddes method: Distribution of non-key components.
- Underwood equations: Minimum reflux ratio, R_{min}.
- Gilliland correlation: Actual number of stages, given the actual reflux ratio, R_{min} and N_{min}.

It should be pointed out that all these methods have approximations that the designer must understand. Otherwise, incorrect preliminary decisions on the choice of separator and operating conditions might be made.

1.4.1 Relative volatility

We first introduce relative volatity - a ratio of the K-values of two components:

$$\alpha_{ij} = \frac{K_i}{K_j} = \frac{\frac{y_i}{x_i}}{\frac{y_j}{x_j}} \tag{1.4.1}$$

The geometric mean of the top and bottom product α values is most commonly used in the short-cut calculations,

$$(\alpha_{ij})_{mean} = \sqrt{(\alpha_{ij})_{top}\,(\alpha_{ij})_{bottom}} \tag{1.4.2}$$

From equation 1.4.1, it is clear that the relative volatilities of distillate and bottom product depend on product compositions, but normally product compositions are not known and an iterative procedure may be required to estimate them using short-cut models.

1.4.2 The Fenske equation

The Fenske equation[5] is used to estimate the minimum number of the theoretical stages,

Assumptions:

- Total Reflux

Figure 1.3 shows two stages in a simple distillation column, where y_L and y_H refer to the compositions of light and heavy components in the vapour and x_L and x_H refer to the compositions of light and heavy components in the liquid.

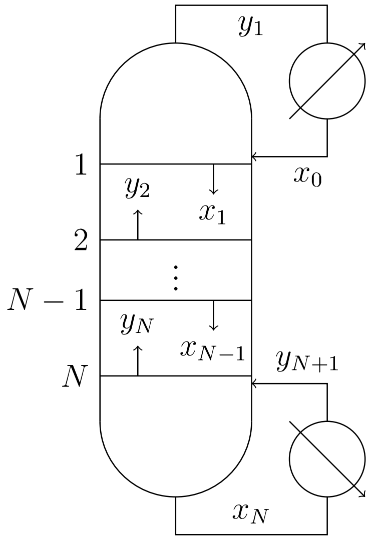


Figure 1.3: Simple distillation column for deriving the Fenske equation.

According to the definition of the relative volatity, equation 1.4.1, on each stage,

$$\alpha_{LH,n} = \frac{\left(\dfrac{y_{L,n}}{x_{L,n}}\right)}{\left(\dfrac{y_{H,n}}{x_{H,n}}\right)}$$

$$\alpha_{LH,n} = \frac{y_{L,n}x_{H,n}}{y_{H,n}x_{L,n}} \tag{1.4.3}$$

which can be transformed into:

$$\left(\frac{y_L}{y_H}\right)_n = \alpha_{LH,n}\left(\frac{x_L}{x_H}\right)_n \tag{1.4.4}$$

At the bottom of the column the mass balance is given by the stripping section operating line (remembering the assumption of constant L/V),

$$y_{n+1} = \frac{L}{V}x_n - \frac{B}{V}x_D \tag{1.4.5}$$

As the column is run under total reflux there is no bottom product, i.e. $B = 0$, therefore,

$$y_{n+1} = \frac{L}{V} x_n \tag{1.4.6}$$

This is true for all components in the system, which means that,

$$\begin{aligned} \frac{y_{L,n+1}}{y_{H,n+1}} &= \frac{\left(\frac{L}{V}\right) x_{L,n}}{\left(\frac{L}{V}\right) x_{H,n}} \\ \left(\frac{y_L}{y_H}\right)_{n+1} &= \left(\frac{x_L}{x_H}\right)_n \end{aligned} \tag{1.4.7}$$

Combining equation 1.4.7 with 1.4.4 (taken for $n + 1$) gives,

$$\alpha_{LH,n+1} \left(\frac{x_L}{x_H}\right)_{n+1} = \left(\frac{y_L}{y_H}\right)_{n+1} = \left(\frac{x_L}{x_H}\right)_n \tag{1.4.8}$$

For the each stage in the column we therefore have,

$$\begin{aligned} \left(\frac{x_L}{x_H}\right)_{N-1} &= \alpha_{LH,N} \left(\frac{x_L}{x_H}\right)_N \\ \vdots \qquad & \qquad \vdots \\ \left(\frac{x_L}{x_H}\right)_1 &= \alpha_{LH,2} \left(\frac{x_L}{x_H}\right)_2 \\ \left(\frac{x_L}{x_H}\right)_0 &= \alpha_{LH,1} \left(\frac{x_L}{x_H}\right)_1 \end{aligned} \tag{1.4.9}$$

Combining equation 1.4.9 produces,

$$\left(\frac{x_L}{x_H}\right)_0 = \prod_{n=1}^{N} \alpha_{LH,n} \left(\frac{x_L}{x_H}\right)_N \tag{1.4.10}$$

As the composition of the liquid entering the column at the top is equal to the distillate composition, and that leaving the column at the bottom is equal to the bottoms composition, then,

$$\left(\frac{x_L}{x_H}\right)_D = \prod_{n=1}^{N} \alpha_{LH,n} \left(\frac{x_L}{x_H}\right)_B \tag{1.4.11}$$

If we then define,

$$\bar{\alpha}_{LH} = \left(\prod_{n=1}^{N} \alpha_{LH,n}\right)^{1/N} \tag{1.4.12}$$

Which can be approximated by equation 1.4.2. Therefore,

$$\begin{aligned}\left(\frac{x_L}{x_H}\right)_D &= \prod_{n=1}^{N} \alpha_{LH,n} \left(\frac{x_L}{x_H}\right)_B \\ \left(\frac{x_L}{x_H}\right)_D &= \bar{\alpha}_{LH}^{N} \left(\frac{x_L}{x_H}\right)_B \\ \bar{\alpha}_{LH}^{N} &= \left(\frac{x_L}{x_H}\right)_D \left(\frac{x_H}{x_L}\right)_B \end{aligned} \qquad (1.4.13)$$

As this analysis is for total reflux this means that the number of stages is the minimum number, N_{min},

$$N_{min} = \frac{\log\left[\left(\frac{x_L}{x_H}\right)_D \left(\frac{x_H}{x_L}\right)_B\right]}{\log \bar{\alpha}_{LH}} \qquad (1.4.14)$$

or equivalently,

$$N_{min} = \frac{\log\left[\frac{D_L}{D_H}\frac{B_H}{B_L}\right]}{\log \bar{\alpha}_{LH}} \qquad (1.4.15)$$

If can also be expressed in terms of the recovery of the key components, r,

$$N_{min} = \frac{\log\left[\frac{r_{D,L}}{1-r_{D,L}}\frac{r_{B,H}}{1-r_{B,H}}\right]}{\log \bar{\alpha}_{LH}} \qquad (1.4.16)$$

Equation 1.4.14 is the original form of the Fenske equation for calculating the minimum number of theoretical stages. When $\bar{\alpha}$ is extended to be the geometric mean of the relative volatilities of the light and heavy key components in a multiple component system. Equation 1.4.14 are still approximately valid and largely useful for estimating the minimum number of the theoretical stages.

1.4.3 The Hengstebeck-Geddes Method

All components distribute between distillate and bottoms at total reflux; while at minimum reflux conditions, none or only a few of the nonkey components distribute. Distribution ratios for these two limiting conditions are given in Figure 1.4 [11].

For total reflux, the Fenske equation results in a plot as a straight line on log-log coordinates. Such that taking equation 1.4.15 for to general components, i and j,

$$\begin{aligned} N_{min} &= \frac{\log\left[\frac{D_i}{D_j}\frac{B_i}{B_j}\right]}{\log \alpha_{i,j}} \\ N_{min}\log\alpha_{i,j} &= \log\left(\frac{D_i}{B_i}\right) - \log\left(\frac{D_j}{B_j}\right) \\ \log\left(\frac{D_i}{B_i}\right) &= \log\left(\frac{D_j}{B_j}\right) + N_{min}\log\alpha_{i,j} \end{aligned} \qquad (1.4.17)$$

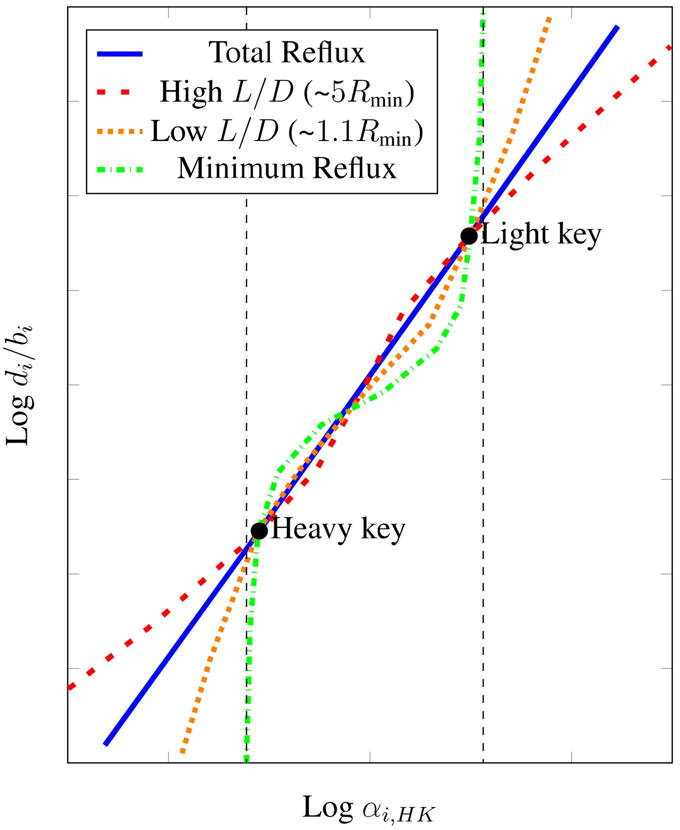


Figure 1.4: The Hengstebeck-Geddes relation (adapted from [11]).

As it can be said that $\alpha_{i,j} = \alpha_{i,r}\alpha_{r,j}$, where r refers to a reference component, equation 1.4.17 can be rearanged to be,

$$\log\left(\frac{D_i}{B_i}\right) = \log\left(\frac{D_j}{B_j}\right) + N_{min}\log\alpha_{i,r} + N_{min}\log\alpha_{r,j}$$
$$\log\left(\frac{D_i}{B_i}\right) = \log\left(\frac{D_j}{B_j}\right) + N_{min}\log\alpha_{i,r} - N_{min}\log\alpha_{j,r}$$
$$\log\left(\frac{D_i}{B_i}\right) = \log\left(\frac{D_j}{B_j}\alpha_{j,r}^{-N_{min}}\right) + N_{min}\log\alpha_{i,r} \tag{1.4.18}$$

which is known as the Hengstebeck-Geddes method [6]. If we take the heavy key, H, as the reference component, then the straighline equation can be written for each component, i, in the system as,

$$\log\frac{D_i}{B_i} = A + C\log\alpha_{i,H} \tag{1.4.19}$$

where C is equal to N_{min} and A can be conveniently obtained from the heavy key component as,

$$A = \log\left(\frac{D_H}{B_H}\right) \tag{1.4.20}$$

For minimum reflux, the Underwood equations (below) can be used to estimate the component distributions. Product-distribution curves for a given reflux might be expected to lie between the lines for total and minimum reflux. However, this is not the case, and product distributions are complex. Near R_{min}, product distribution (curve 3) lies between the two limits (curves 1 and 4). However, for a high reflux ratio, non-key distributions (curve 2) may lie outside the limits, thus causing inferior separations.

As the reflux ratio is decreased from total reflux while maintaining the key-component splits, stage requirements increase slowly at first, but then rapidly as minimum reflux is approached. Initially, large decreases in reflux cannot be compensated for by increasing stages. This causes inferior nonkey distributions. As R_{min} is approached, small decreases in reflux are compensated for by large increases in stages; and the separation of non-key components becomes superior to that at total reflux.

It appears reasonable to assume that, at a near-optimal reflux ratio of 1.3, nonkey-component distribution is close to that estimated by the Fenske equation for total-reflux conditions.

1.4.4 The Underwood Equation

The Underwood equation[12, 13] is widely used in estimating the minimum reflux ratio for multiple component systems even though it usually underestimates the minimum reflux ratio.

At the point of the minimum reflux ratio, there is a pinch point in the system, Figure 1.5

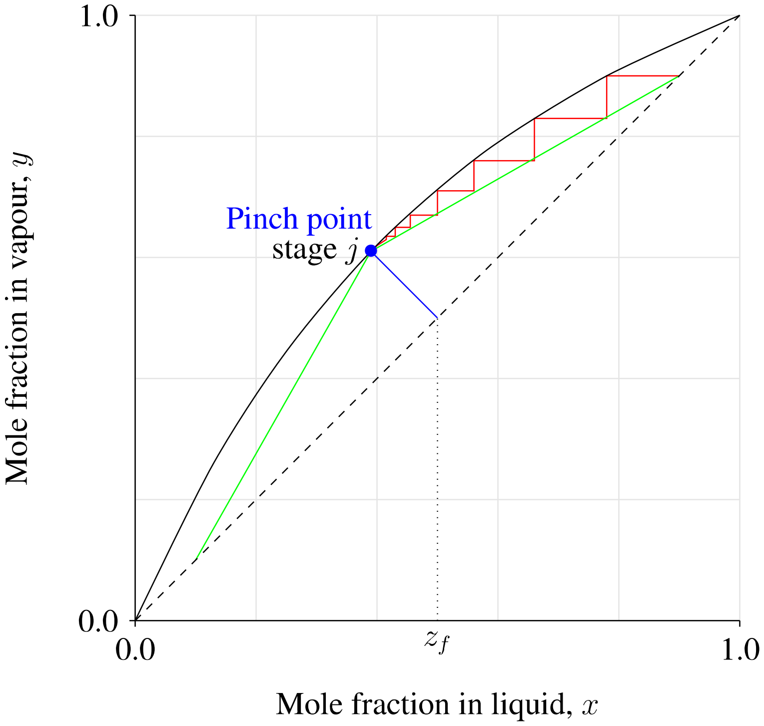


Figure 1.5: Pinch point condition for a binary system.

At this pinch point the concentration at stage j stays the same on the plates above and below,

$$\begin{aligned} x_{j-1} &= x_j = x_{j+1} \\ y_{j-1} &= y_j = y_{j+1} \end{aligned} \tag{1.4.21}$$

The pinch point is also on the equilibrium curve such that,

$$y_{j,i} = K_i x_{j,i} \tag{1.4.22}$$

Taking a mass balance over the rectifing section (i.e. pinch point up to the distillate) gives,

$$V_{min}y_{j+1,i} = L_{min}x_{j,i} + Dx_{D,i} \tag{1.4.23}$$

Substituting equation 1.4.22 into equation 1.4.23 gives,

$$V_{min}y_{j+1,i} = L_{min}\frac{y_{j,i}}{K_i} + Dx_{D,i} \tag{1.4.24}$$

and from equation 1.4.21,

$$V_{min}y_{j,i} = L_{min}\frac{y_{j,i}}{K_i} + Dx_{D,i} \tag{1.4.25}$$

This can be rearranged for $y_{j,i}$ as,

$$\begin{aligned} V_{min}y_{j,i} - L_{min}\frac{y_{j,i}}{K_i} &= Dx_{D,i} \\ y_{j,i}\left(V_{min} - \frac{L_{min}}{K_i}\right) &= Dx_{D,i} \\ y_{j,i} &= \frac{Dx_{D,i}}{\left(V_{min} - \frac{L_{min}}{K_i}\right)} \end{aligned} \tag{1.4.26}$$

As we don't know the position of the pinch point, we don't know the individual compositions of $y_{j,i}$, however we do know that the sum of the vapour fractions of all the components is equal to one, so that,

$$1 = \sum \frac{Dx_{D,i}}{\left(V_{min} - \frac{L_{min}}{K_i}\right)} \tag{1.4.27}$$

The equilibrium constant for any component can be given by the relative volatility and the equilibrium constant of the reference component $K_i = \alpha_{i,r}K_r$, so that,

$$1 = \sum \frac{Dx_{D,i}}{\left(V_{min} - \frac{L_{min}}{\alpha_{i,r}K_r}\right)} \tag{1.4.28}$$

This can now be rewritten as in a simplified form as an expression for V_{min},

$$\begin{aligned} 1 &= D\sum \frac{x_{D,i}}{\frac{V_{min}}{\alpha_{i,r}}\left(\alpha_{i,r} - \frac{L_{min}}{V_{min}K_r}\right)} \\ V_{min} &= D\sum \frac{\alpha_{i,r}x_{D,i}}{\left(\alpha_{i,r} - \frac{L_{min}}{V_{min}K_r}\right)} \end{aligned} \tag{1.4.29}$$

This same method analysis can also be performed on the stripping section of the column to produce,

$$-V'_{min} = B\sum \frac{\alpha_{i,r}x_{B,i}}{\left(\alpha_{i,r} - \frac{L'_{min}}{V'_{min}K'_r}\right)} \tag{1.4.30}$$

Under the assumptions of constant molar overflow and constant relative volatility it can be proved that,

$$\frac{L_{min}}{V_{min}K_r} = \frac{L'_{min}}{V'_{min}K'_r} = \phi \tag{1.4.31}$$

Substituting equation 1.4.31 into equations 1.4.29 and 1.4.30 produces to key linked equations for the minimum vapour flows,

$$V_{min} = D\sum \frac{\alpha_{i,r}x_{D,i}}{(\alpha_{i,r} - \phi)} \tag{1.4.32}$$

$$V'_{min} = -B\sum \frac{\alpha_{i,r}x_{B,i}}{(\alpha_{i,r} - \phi)} \tag{1.4.33}$$

$$\tag{1.4.34}$$

These key equations allow us to generate the two underwood equations. For the first equation, a mass balance around the feed stage gives,

$$V_{min} - V'_{min} = F\left(1 - q\right) \tag{1.4.35}$$

Substituting equations 1.4.32 and 1.4.33 in this gives,

$$\sum \frac{\alpha_{i,r}Dx_{D,i}}{(\alpha_{i,r} - \phi)} + \sum \frac{\alpha_{i,r}Bx_{B,i}}{(\alpha_{i,r} - \phi)} = F\left(1 - q\right)$$

$$\sum \frac{\alpha_{i,r}\left(Dx_{D,i} + Bx_{B,i}\right)}{(\alpha_{i,r} - \phi)} = F\left(1 - q\right) \tag{1.4.36}$$

From an overall column mass balance,

$$Dx_{D,i} + Bx_{B,i} = Fz_{F,i} \tag{1.4.37}$$

Substituting this into equation 1.4.36 gives, the first Underwood equation,

$$1 - q = \sum \frac{\alpha_{i,r}z_{F,i}}{(\alpha_{i,r} - \phi)} \tag{1.4.38}$$

As this only contains information about the feed it allows us to calculate ϕ. Note, there are multiple roots to this equation, Figure 1.6; each root of ϕ lies between an adjacent pair of relative volatilities of distributing species. Generally the root we are looking for is $\alpha_{HK} < \phi < \alpha_{LK}$ if the heavy key is the reference for the volatility.

The value of ϕ can then be used to find the minimum reflux ratio, R_{min} from,

$$R_{min} = \frac{V_{min}}{D} - 1 \tag{1.4.39}$$

Substituting V_{min} from equation 1.4.32 gives the second Underwood equation,

$$R_{min} + 1 = \sum \frac{\alpha_{i,r}x_{D,i}}{(\alpha_{i,r} - \phi)} \tag{1.4.40}$$

The Underwood minimum reflux equations are widely used, but often without examining the possibility of non-key distributions. In addition, the assumption is frequently made that R_{min} equals the reflux ratio without pinch points of the non-key components.

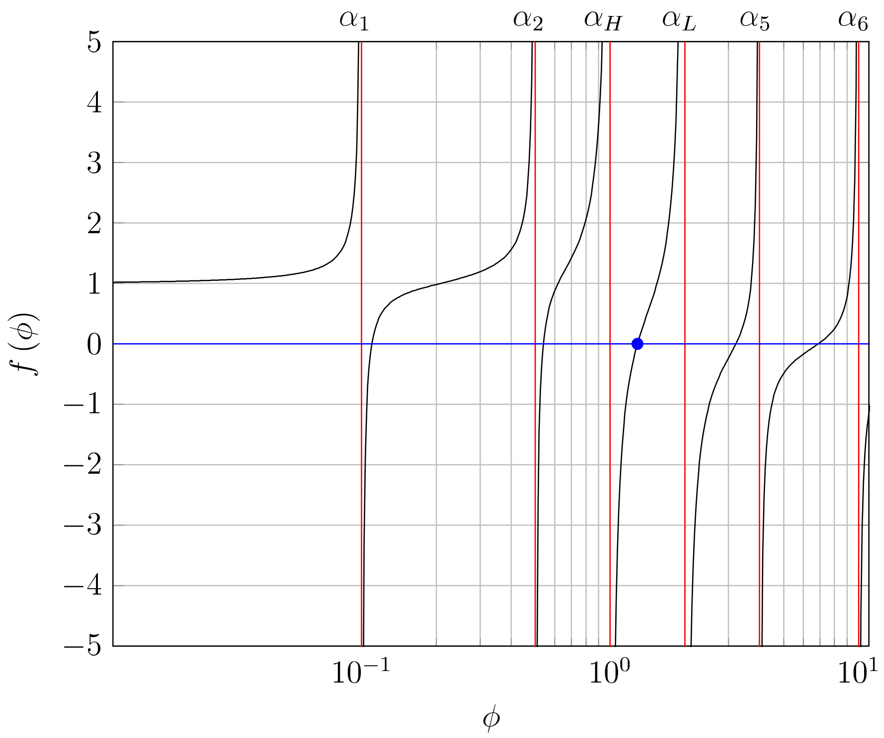


Figure 1.6: Graphical solution to the first Underwood equation for an example of 6 components.

When the Underwood assumptions appear valid and a negative minimum reflux ratio is computed, a rectifying section may not be needed for the separation. The Underwood equations show that the minimum reflux depends mainly on feed condition and α and, to a lesser extent, on degree of separation, as is the case with binary distillation. As with binary distillation, a minimum reflux ratio exists in a multi-component system for a perfect separation between the LK and HK. The Underwood method can also be extended for use with multiple feeds [2].

For calculations at actual reflux conditions with a process simulator by the computer programs knowledge of R_{min} is not essential, but N_{min} must be known if the split between two components is to be specified.

1.4.5 The Gilliland correlation

The Gilliland correlation[7] is probably the most widely used empirical relation in practical distillation design for the calculation of the number of the theoretical stages under a given reflux ratio.

As shown in Figure 1.7, the correlation was established between $Y = (N - N_{min})/(N+1)$ and $X = (R - R_{min})/(R+1)$. As the minimum number of theoretical stages, N_{min} and minimum reflux ratio, R_{min} can be obtained from Fenske equation and the Underwood equation, respectively, as long as the actual reflux ratio, R, is given, then the actual number of the theoretical stages, N, can be calculated according to the values given in the correlation. The data used covers the range of parameters,

- Number of components: 2 to 11
- α: 1.11 to 4.05

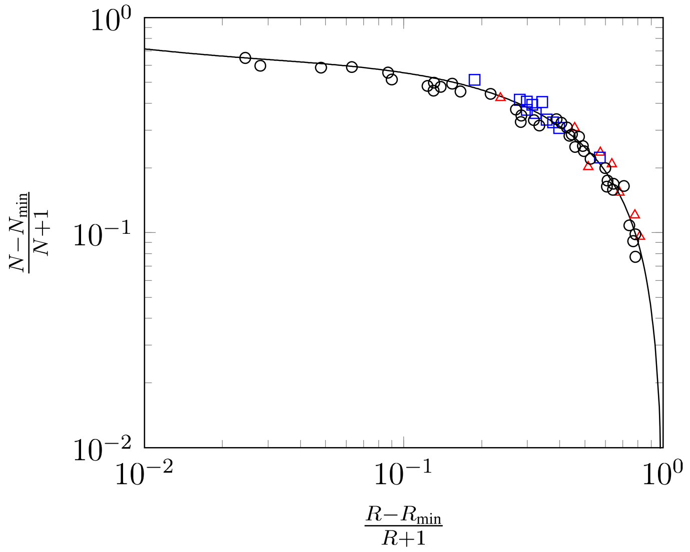


Figure 1.7: The Gilliland correlation. Data plotted: ○ from Gilland [7, 10], □ from van Winkle and Todd [14], and △ from Brown-Martin [3]. Line plotted from equation 1.4.41 by Molokanov [9].

- q: 0.28 to 1.42
- R_{min}: 0.53 to 9.09
- Pressure: vacuum to 600 psig
- N_{min}: 3.4 to 60.3

It is worth noting that the Gilliland correlation presents a graphical correlation, using the design data. This correlation is effectively accurate for $X < 0.17$. This graphical correlation has been fitted to an equation [9],

$$\frac{N-N_m}{N+1} = 1-\exp\left[\left(\frac{1+54.5\Psi}{11+117.2\Psi}\right)\left(\frac{\Psi-1}{\Psi^{0.5}}\right)\right] \qquad \text{where} \qquad \Psi = \frac{R-R_m}{R+1} \tag{1.4.41}$$

A more accurate correlation should utilize a parameter involving the feed condition, q. As feed conditions range from subcooled liquid to superheated vapour the number of stages required for the separation decreases. However, this effect is small unless $\alpha_{LK,HK}$ is high.

1.4.6 The Erbar-Maddox correlation

Similar to the Gilliland correlation, the Erbar-Maddox correlation [4] also relates the actual number of theoretical stages to the actual reflux ratio, the minimum reflux ratio and the minimum number of theoretical stages.

This is also an empirical correlation, it is sometimes considered more reliable than the Gilliland correlation, but its use is not as widespread as the latter, Figure 1.8.

1.4.7 The Kirkbride Correlation

Implicit in the application of the Gilliland correlation is the specification that stages be distributed optimally between the rectifying and stripping sections. The optimal feed

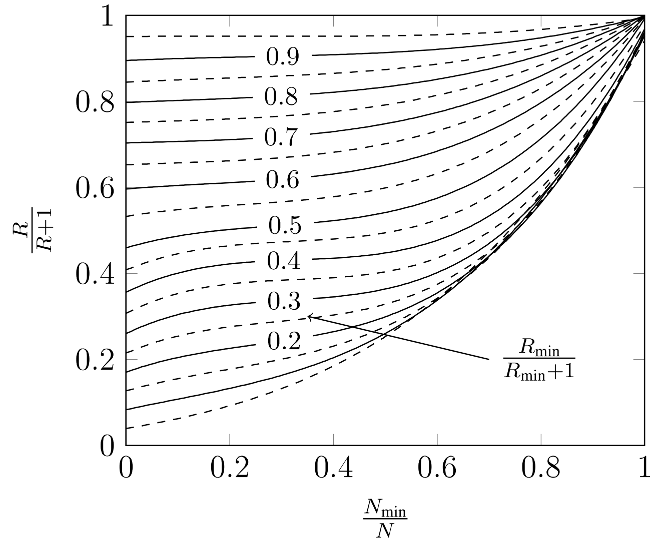


Figure 1.8: The Erbar-Maddox correlation [4] using prediction equations from [1].

stage can be located by assuming that the ratio of stages above the feed to stages below is the same as the ratio determined by applying the Fenske equation to the separate sections at total reflux conditions.

Unfortunately, this is not reliable except for fairly symmetrical feeds and separations.

A better approximation of the optimal feed-stage location can be made with the Kirkbride empirical equation [8],

$$\log\left(\frac{N_r}{N_s}\right) = 0.206\log\left[\left(\frac{B}{D}\right)\left(\frac{x_{HK,f}}{x_{LK,f}}\right)\left(\frac{x_{LK,b}}{x_{HK,d}}\right)^2\right] \tag{1.4.42}$$

where N_r is the number of stages in the rectifying section (above feed stage), N_s is the number of stages in the stripping section (below feed stage), $x_{HK,f}$ is the mole fraction of heavy key component in feed, $x_{LK,f}$ is the mole fraction of light key component in feed, B and D are the distillate and bottoms flow rates respectively, $x_{LK,b}$ is the mole fraction of light key component in bottom product, and $x_{HK,d}$ is the mole fraction of heavy key component in distillate.

1.4.8 Procedures of the short-cut design

For shortcut distillation design, several commercial software packages are available for simulations, optimizations, and optimal controls. Most of these packages are usually limited to conventional systems as they were developed in early or late 1980s.

Normally, in the use of the shortcut design, the following steps are required, for a feed of known composition, flow rate, thermal condition and pressure,

1. Specify top product phase, i.e. total or partial condenser.
2. Select light and heavy key components.
3. Specify mole fraction of light key in the bottoms and heavy key in the distillate.
4. Specify pressure of condenser and reboiler.
5. Estimate the relative volatilities for the top, bottom and feed point of the column.

6. Use the Fenske equation to calculate N_{min}.
7. Use the Hengstebeck-Geddes method to estimate the split of the non-key components.
8. Repeat steps 5 to 7 until N_{min} doesn't change.
9. Use the Underwood equations to calculate R_{min}.
10. Pick a R/R_{min} value.
11. Use the Gilliland correlation to calculate the actual number of stages, N.
12. Use the Kirkbride correlation to calculate the ratio of the rectifying to stripping number of stages.

1.5 Rigorous Model for Multi-component Distillation

A common design for distillation columns is that the condenser at the top of the column is assumed to be a complete condenser (i.e. all the vapour that enters the condenser leaves as a liquid at the same temperature with no-sub-cooling). This means that this does not represent an equilibrium stage. The first plate below the condenser is where the final stage of the enrichment occurs.

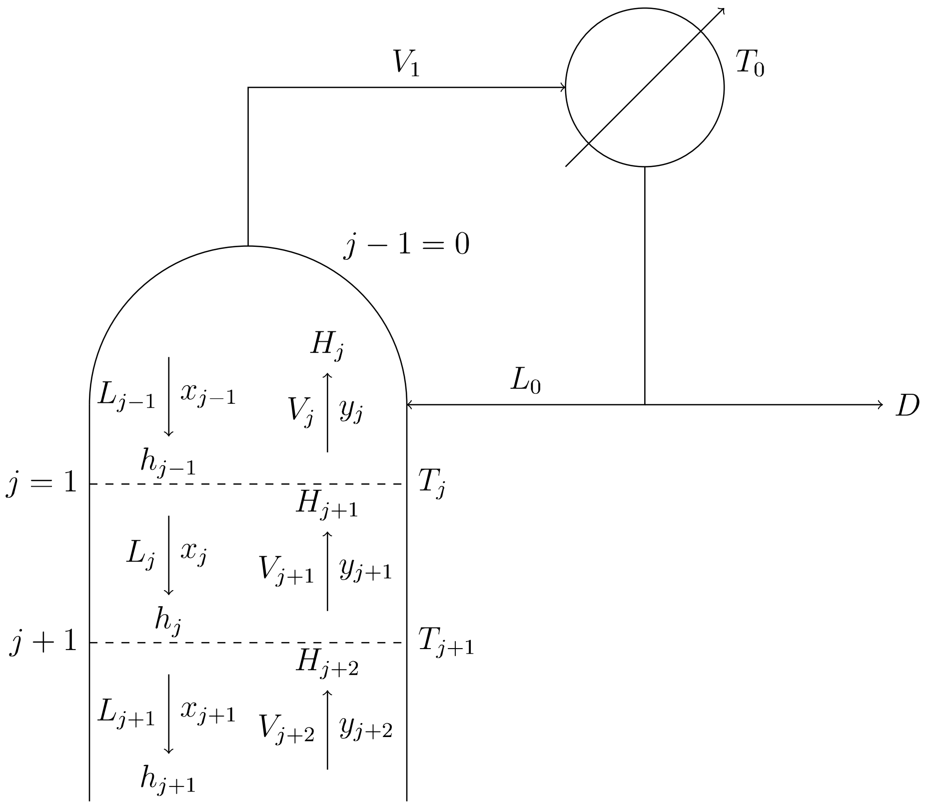


Figure 1.9: Mass and energy flow in the top of a distillation column.

Calculation of Plate Vapour-Liquid Equilibrium

The composition of the vapour on the plate, y_j, is known from calculation of the previous plate[1]. Therefore it is possible to calculate the composition of the liquid on the same tray, x_j, as it is in equilibrium.

The temperature of the plate is calculated using a mole weighted vapour pressure[2],

$$\sum \frac{y_i}{K_i} = 1 \tag{1.5.1}$$

The values of y_i/K_i produced are then the values of x_i for that plate, the x_js.

Plate Mass and Energy Balance

The second step for calculating the plate is to make an estimate for the composition of the vapour arriving from the next plate, y_{j+1}. Assuming that there is no chemical reaction then the molar flow from each plate should equal that to each plate,

$$L_j + V_j = L_{j-1} + V_{j+1} \tag{1.5.2}$$

and the flow of each component should also be constant,

$$x_j L_j + y_j V_j = x_{j-1} L_{j-1} + y_{j+1} V_{j+1} \tag{1.5.3}$$

Combining these two equation gives y_{j+1},

$$y_{j+1} = \frac{\left(L_{j-1} + V_{j+1} - V_j\right) x_j + y_j V_j - x_{j-1} L_{j-1}}{V_{j+1}} \tag{1.5.4}$$

At this point however the value of V_{j+1} is unknown, but as an initial guess assumed to be equal to V_j. The values of L_{j-1} and V_j are known from the previous plate balance[3].

The liquid flow down from the plate, L_j, and the vapour flow rate up from the next plate, V_{j+1} can also be calculated using a heat balance,

$$h_j L_j + H_j V_j = h_{j-1} L_{j-1} + H_{j+1} V_{j+1} \tag{1.5.5}$$

where H is the vapour enthalpy and h is the liquid enthalpy calculated from the calculated plate temperature and the estimate of y_{j+1}.

Rearranging this equation in terms of L_j and combining with the molar flow balance (equation 1.5.2) gives,

---

[1] In the case of the first plate the composition of y_1 is of course equal to that of the distillate, x_d, which is also equal to x_0

[2] The pressure at this point can be considered constant throughout the column and equal to that of the feed

[3] The value of V_1 and L_0 can be calculated from the distillate flow rate, D, and the reflux ratio , R, calculated from the short cut calculation,

$$\begin{aligned} V_1 &= (R+1)\,D \\ L_0 &= RD \end{aligned}$$

$$L_j = \frac{H_j V_j - h_{j-1} L_{j-1} + H_{j+1} \left(L_{j-1} - V_j\right)}{H_{j+1} - h_j} \tag{1.5.6}$$

Also from the molar flow balance (equation 1.5.2),

$$L_j = L_{j-1} + V_{j+1} - V_j \tag{1.5.7}$$

therefore iterating the value of V_{j+1} until the values of L_j calculated by the last two equation are equal solves the plate.

1.6 Considerations in Multi-component Distillation

There are some general considerations in designing a multi-component distillation. As many components are present in the feed, fixing the recovery or mole fraction of a single component does not specify the product composition or temperature; also, the selection of the key components is a process engineering decision, usually based on the objectives of the separation. Mass and energy balances are solved iteratively. It is useful to use commercial process simulation software to carry out the calculations. The short-cut design equations allow initial estimates of design variables and column performance to be obtained prior to rigorous simulation. Practical experience shows that convergence of rigorous simulation can be facilitated by good initial estimates.

1.6.1 Choice of distillation operating parameters

In determining the distillation operating parameters, usually the feed composition and flow rate are considered fixed. The product specifications are usually given in the design problem statement and may be expressed in terms of recovery of certain components, product purities or specific compositions.

The operating parameters to be selected by the design engineer would normally include,

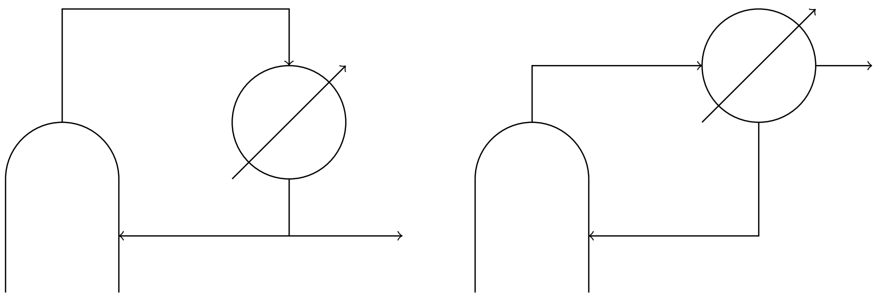
- Operating pressure.
- Reflux ratio.
- Feed condition.
- Feed stage location.
- Type of condenser.

In the preliminary distillation design, it is necessary for the design to be on the basis of the whole system rather than a specific part as distillation columns are energy-intensive any heat integration of distillation columns can significantly reduce the energy costs.

The heat integration opportunities for heat recovery in distillation columns can be created and the operating pressure, reflux ratio and feed condition can have effects on those opportunities thus their selection should be carefully considered.

Operating pressure

It is clear that the condenser temperature sets the distillation operating pressure as the vapour comes out of the top of the column. There are two types of condensers: total and partial condensers, Figure 1.10.



(a) Total condenser (b) Partial condenser

Figure 1.10: Condenser options.

A total condenser means all the vapour is to be condensed leaving only liquid as the top product, in such cases, pressure should be fixed. Additionally, if cooling water is to be used as the heat transfer agent, the bubble point of the overhead product should be typically 10 °C above the summer cooling water temperature. If air cooling is to be used, the bubble point of the overhead product should be typically 20 °C above the summer air temperature.

For a partial condenser: vapour is taken as the top product. The above criteria for the total condenser are applied to the dew point of the vapour top product.

There are exceptions for determining operating pressure in a distillation column, e.g.

- In the separation of gases and light hydrocarbons, a very high operating pressure may be required as a result of trying to operate the condenser against cooling water or air cooling, therefore, a combination of high operating pressure and low temperature condensation using refrigeration may be needed.
- For distilling high molecular weight materials, process constraints may restrict the maximum temperature of the distillation to avoid product decomposition, a vacuum operation must be used to reduce the boiling temperature.
- In a sequence of columns, the condenser of one operating at a high pressure may provide heat to the reboiler of another operating at a lower pressure thus heat recovery opportunities can be created.

The effect of pressure on distillation is shown in Figure 1.11 in an example of separating a benzene-toluene-ethylbenzene-styrene mixture.

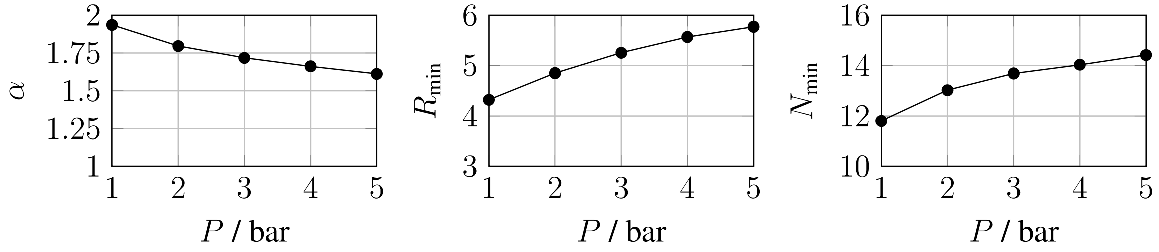
Effect of temperature on utility costs

The temperature of a distillation column has significant effects on utility costs as the typical utility costs depend on the temperature and source of heating or cooling. Figure 1.12 shows such effects.

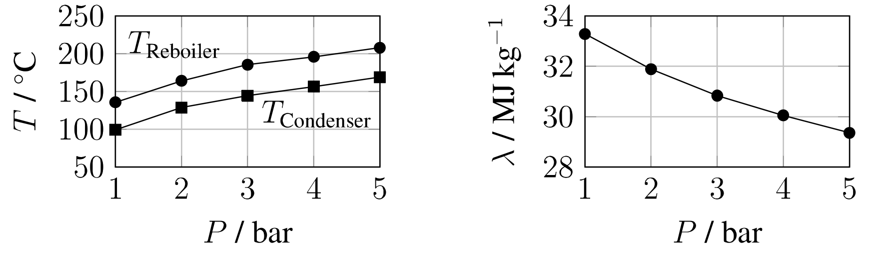
As seen from Figure 1.12, water used as the cooling agent would lower the costs significantly thus is the most economic source of creating condensation. This result is also based on a simulation of separating a benzene-toluene-ethylbenzene-styrene mixture.

1.6.2 Choice of reflux ratio

The reflux ratio has a direct effect on the number of theoretical stages. There are two extreme situations: minimum and total reflux ratio.



(a) Relative volatility decreases (b) Reflux requirement increase (c) No. stages required increases



(d) Reboiler and condenser temperature increases (e) Latent heat of vapourisation decreases

Figure 1.11: The effects of pressure on (a) relative volatility of components, (b) reflux, (c) number of theoretical stages, (d) reboiler and condenser temperatures, and (e) the latent heat of the vaporisation.

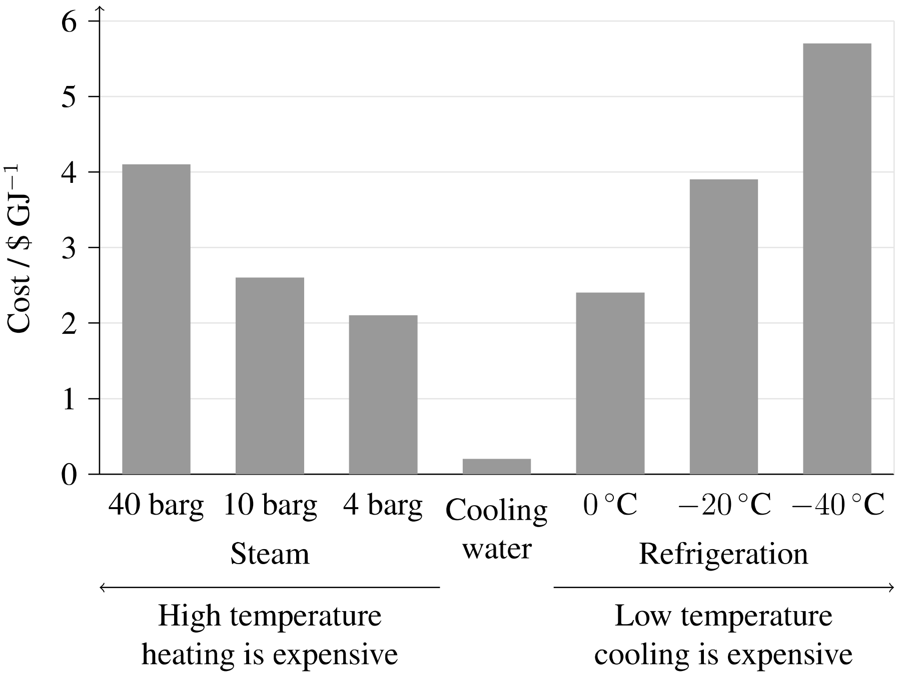


Figure 1.12: The effect of temperature on utility costs.

In the case of the minimum refux ratio, an infinite number of stages would be required which would incur a very high capital cost but minimise the energy requirements thus the operating cost is low. However, in case of total reflux ratio, a minimum number of stages is required, which means low capital cost but needs to maintain high energy supply resulting in high operating costs. Figure 1.13 shows the variation of the number of stages with the reflux ratio for the same benzene-toluene-ethylbenzene-styrene mixture.

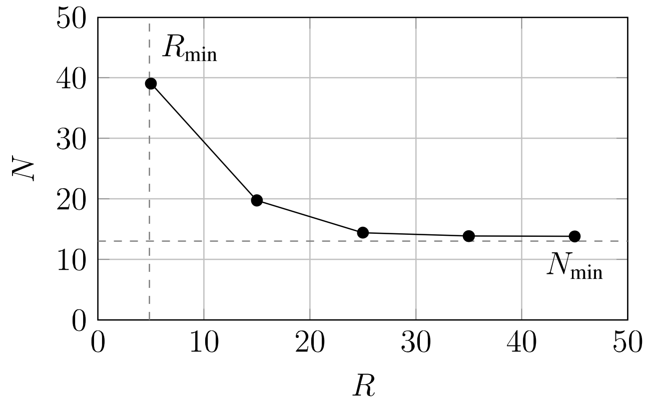


Figure 1.13: Effect of reflux ratio on the number of theoretical stages.

To avoid the two extremes, the actual reflux will lie between the minimum and the total ratio. Figure 1.14 illustrates that there are capital-energy trade-offs, to lower the total cost, and that there is often an optimum reflux ratio.

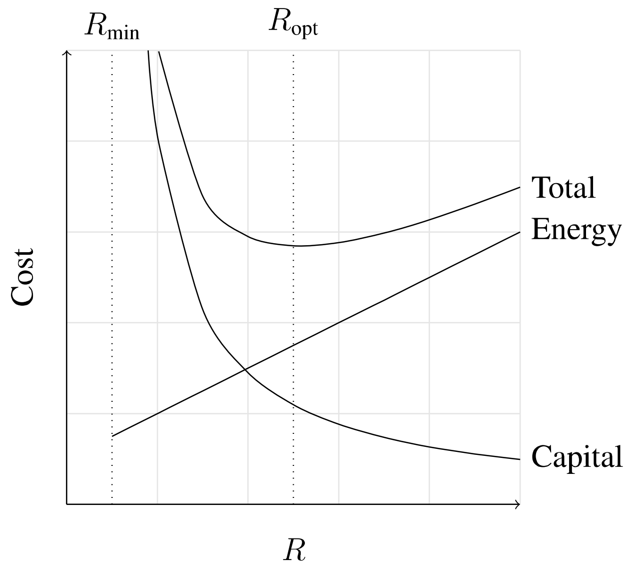


Figure 1.14: Trade-offs between the capital and energy costs.

The Rule of thumb for the choice of the optimum reflux ratio can be expressed as within the range of 1.1 to 1.3 times of the minimum reflux ratio. This rule of thumb assumes that heating and cooling duties will be provided by the utility system. Also, the reflux ratio should only be optimised in the context of the overall process heating and cooling requirements. Table 1.1 shows some industrial examples and the conditions used; this shows that the guide reflux ratio is not always the optimum for a variety of reasons.

Choice of feed condition and feed stage location

The feed condition and feed stage location are two other important factors that would affect the heating duty energy cost significantly.

The feed condition, q, value affects the vapour and liquid flow rates in the column. A simple rule is that the feed temperature usually lies between the extreme temperatures of the column (condenser and reboiler temperatures).

Table 1.1: Representative commercial distillation operations.

Binary Mixture	Average Relative Volatility	Number of Pressure / psia	Typical Operating Reflux Ratio	Reflux to Minimum
1,3-Butadiene/vinyl acetylene	1.16	130	75	1.70
Vinyl acetate/ethyl acetate	1.16	90	15	1.15
o-Xylene/m-xylene	1.17	130	15	1.12
Isopentane/n-pentane	1.30	120	30	1.20
Isobutane/n-butane	1.35	100	100	1.15
Ethylbenzene/styrene	1.38	34	1	1.71
Propylene/propane	1.40	138	280	1.06
Methanol/ethanol	1.44	75	15	1.20
Water/acetic acid	1.83	40	15	1.35
Ethylene/ethane	1.87	73	230	1.07
Acetic acid/acetic anhydride	2.02	50	15	1.13
Toluene/ethylbenzene	2.15	28	15	1.20
Propane/1,3-butadiene	2.18	40	120	1.13
Ethanol azeotrope/water	2.21	60	15	1.35
Isopropanol/water	2.23	12	15	1.28
Benzene/toluene	3.09	34	15	1.15
Methanol/water	3.27	60	45	1.31
Cumene/phenol	3.76	38	1	1.21
Benzene/ethylbenzene	6.79	20	15	1.14
HCN/water	11.20	15	50	1.36
Ethylene oxide/water	12.68	50	50	1.19
Formaldehyde/methanol	16.70	23	50	1.17
Water/ethylene glycol	81.20	16	4	1.20

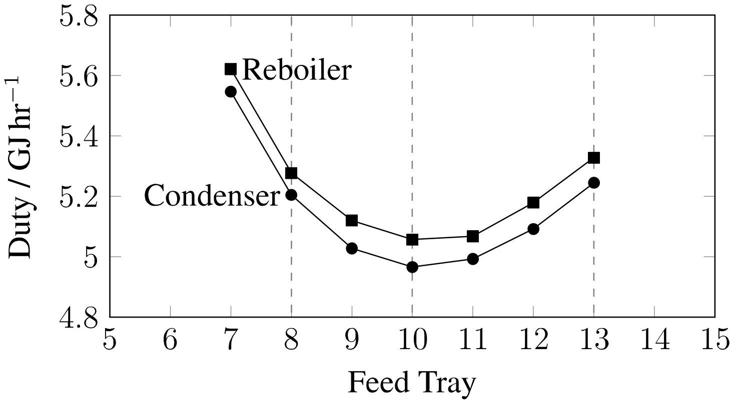
- $q = 1$, (saturated liquid feed) is preferred. This makes the column pressure easy to control by pumping the liquid to the correct pressure.
- If $q = 0$ (saturated vapour feed), a compressor, rather than a pump, is needed, as it is harder to adjust the pressure of a vapour feed than a liquid feed.
- Superheated or subcooled feeds cannot participate in the separation process until thermal equilibrium is achieved
- In columns with multiple feeds, it is not unusual to use feeds with $q < 0$ or $q > 1$, which would be determined by the practical needs.

Therefore, heating or cooling the feed can reduce overall energy costs. Preheating the feed typically decreases the reboiler duty and may increase the condenser duty. Cooling the feed, conversely, tends to decrease the condenser duty and may increase the reboiler duty. The best feed condition can only be determined by considering heat recovery opportunities with the overall process.

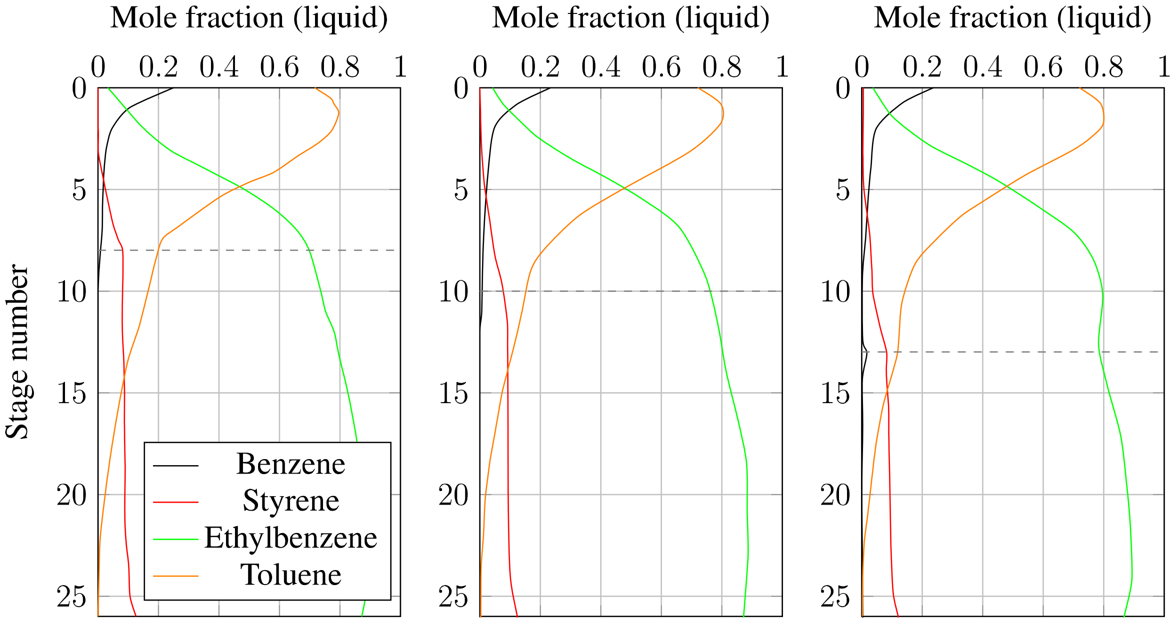
The choice of feed stage location should follow the best match with the feed stage in terms of composition and temperature or using the Kirkbride equation to provide an initial estimate. It should be noted that poor matches are thermodynamically unfavourable, leading to higher energy requirements. In multi-component distillation, it is highly unlikely that the composition of all components can be matched.

Again, in the simulation of separating a Benzene-toluene-ethylbenzene-styrene mixture, a comparison shown in Figure 1.15 between different feeding stages is made to highlight the fact that heating duty depends on the match between the feed compositions and that of the components on a particular stage.

Note in Figure 1.15, when the feed composition and feed stage composition are not well



(a) Change in duty of the condenser and reboiler with feed stage



(b) Composition with feed at stage 8

(c) Composition with feed at stage 10

(d) Composition with feed at stage 13

Figure 1.15: Heating duty depending on the match between the feed condition and thecomposition of the component on the stage. (a) Variation in heat duty, (b)-(d) composition of compentents on the stages with different feed stage positions.

matched, significant disruptions to the composition profiles have been observed.

Type of condenser

As shown in Figure 1.10, there are two types of condensers: total and partial condensers. The total condenser produces liquid product for intermediate or final product storage or the top product is to be fed to another distillation at a higher pressure. The partial condenser reduces the condenser duty and avoid expensive refrigeration as not all vapour is condensed.

1.7 References

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Chapter 2

Distillation Sequencing

Contents

2.1 Chapter 2 ILOs

ILO 2.1. Appraise simple distillation sequences using heuristic rules to find optimal sequences.

ILO 2.2. Compare distillation sequences with the calculation of key performance indicators.

ILO 2.3. Develop distillation sequences using complex distillation columns.

ILO 2.4. Analyse the reasons for the advantages of complex distillation columns.

2.2 Introduction

In almost any chemical industry, the mixtures intended to be separated are not simple binary mixtures, they normally contain more than two components. If the mixture is homogeneous as discussed in the previous chapters, distillation would be the most commonly used method for separating those multiple components. However, if the products required are not only two but more than that, then generally a separation sequence is needed to make the separation of the components in a viable and economic way. This is called distillation sequencing; the separations carried out based on distillation does not have only one sequence but other alternatives. The problem then becomes how to sieve those sequences and choose the best option.

2.3 Sequencing Simple Distillation Columns

A simple distillation column means that a single feed, two products, the key components are adjacent in volatility and the column has a reboiler and a condenser as shown in Figure 2.1.

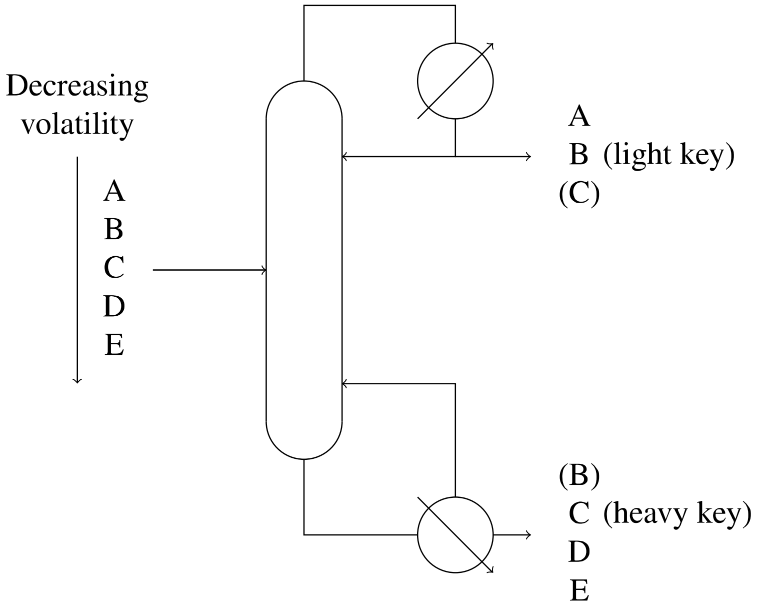


Figure 2.1: A simple column.

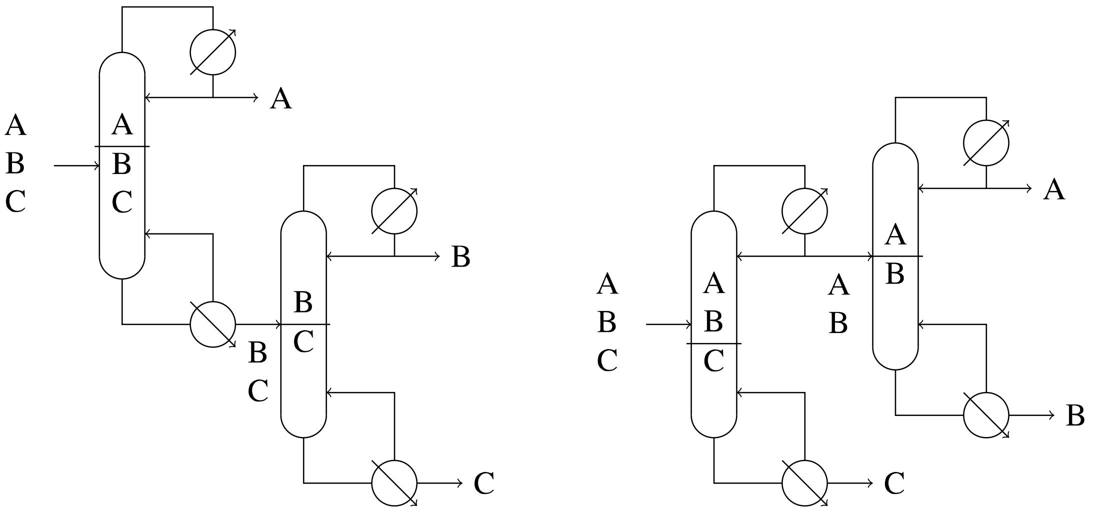
Suppose there is a homogeneous mixture with three components, A, B and C in which A is the lightest component and C is the heaviest component, to be separated, we can show there are in fact two alternative sequences - a direct sequence and an indirect sequence.

A direct sequence means in each column, the lightest component is taken as the overhead product.

An indirect sequence means the heaviest component is taken as the bottom product in each column.

The two sequences are illustrated in Figure 2.2.

As seen in Figure 2.2, in the two sequences, each column has a reboiler and a condenser. It is worth noting that the direct sequence often requires less energy than the indirect one;



(a) Direct Sequence A/BC - B/C (b) Indirect Sequence AB/C - A/B

Figure 2.2: The direct and indirect sequences.

this is because the light component is only vaporised once. However, the indirect sequence can be quite energy efficient compared to the direct one if the light component in the feed has a low flowrate but the heavy component has a high flowrate. This is because feeding a high flowrate of heavy component to both of the columns in the direct sequence is more important than vaporising the light component twice in the indirect sequence.

In a three component mixture, there are only two sequences. When the number of components in a mixture increases, the number of sequences increases dramatically. Figure 2.3 shows the alternative sequences for the separation of a five-component mixture.

Table 2.1 shows how dramatic the number of sequences changes with the increase of the number of the components in the mixture.

Table 2.1: Number of sequences changes with the number of components.

Number of components	Number of possible sequences
2	1
3	2
4	5
5	14
⋮	⋮
10	4862

As seen from Table 2.1, as the number of components increases to 10, the number of sequences increases to 4862. It is almost impossible to try so many sequences. Therefore, the problem becomes: how do we screen for the best few sequences and what criteria should we use to choose between alternative sequences? The way forward is to use simplified methods to identify several promising sequences and then evaluate these options further through rigorous simulations.

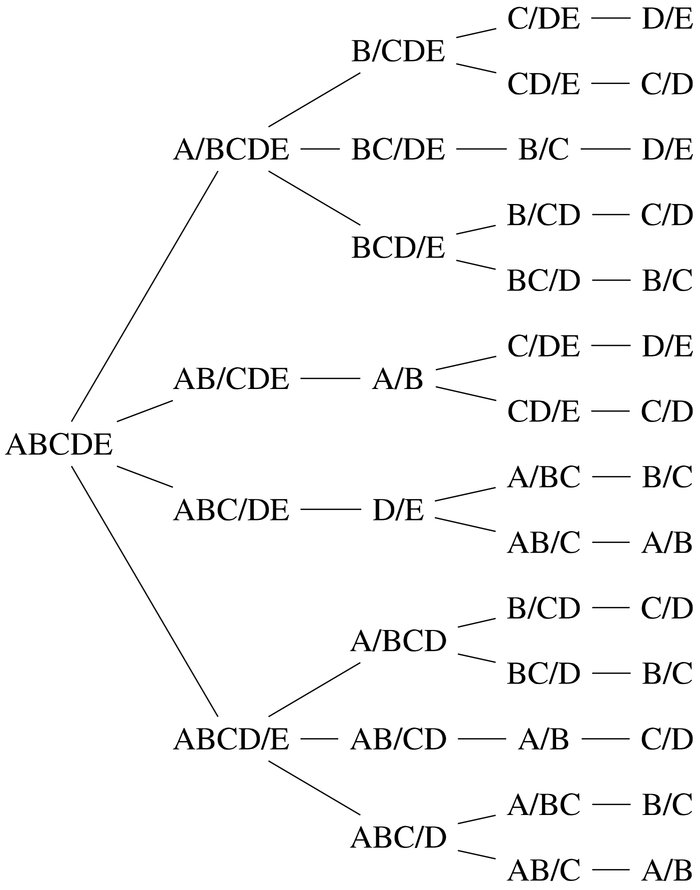


Figure 2.3: Alternative sequences for separating a five component mixture.

2.4 Distillation Columns Sequencing Heuristics

Certainly, not every sequence is to be feasible, there are practical constraints to sequencing columns including,

- Remove early: hazardous component, reactive and heat-sensitive components and corrosive components.
- Decomposition in the reboilers would contaminate products, which means no finished products should be taken from bottoms of columns.
- For those compounds which polymerise when distilled would require chemicals added to inhibit polymerisation but they tend to be involatile and end up in the column bottoms, so it would be necessary to take finished products overhead.
- For those components that are difficult to condense, a total condensation would normally be required under low-temperature refrigeration and/or very high operating pressures. The light components are normally removed from the top of the first column to minimise use of refrigeration and high pressures.

However, there are some heuristics developed for sequencing columns, they are useful as a guide line.

Heuristic 1 Separations where the relative volatility of the key components is close to unity or which exhibit azeotropic behaviour should be performed in the absence of non-key components (Difficult separations typically require large reflux ratios - by removing extraneous components, the internal flows in the column are reduced.) -

Do the most difficult separation last.

Heuristic 2 Sequences which remove the lightest components alone by one in column overheads should be favoured. In other words, favour the direct sequence (Less material is vaporised if only the lightest component is removed overhead, as in Figure 2.9(a).)

Heuristic 3 A component comprising a large fraction of the feed should be removed first (The inventory in subsequent columns is kept to a minimum.)

Heuristic 4 Favour near equimolar splits between top and bottom products in individual columns (The mass load in subsequent columns will be more evenly distributed in the sequence.)

However, difficulties can arise when those heuristics are in conflict with each other. Heuristics can be contradictory to each other but they do have advantages such as they incorporate engineering experience and insights and the designer stays in control (compared to automated screening techniques). However, they are still qualitative guidance, it is thus necessary to have some quantitative measures to classify the relative performance of the sequences.

2.5 Performance Indicators

Some quantitative performance indicators are proposed for screening distillation sequences. These are,

- Total vapour load.
- Total energy demand.
- Operating cost.
- Capital cost.
- Total annualised cost including capital, energy trade-offs, and the consideration of heat integration.

2.5.1 Vapour load

Vapour load of a column is a meaningful performance indicator, as it relates to heat duties in the reboiler and condenser (operating costs tend to dominate overall costs), size of reboiler and condenser also the column diameter.

Vapour load can be calculated using short-cut methods (e.g. Underwood Equation). The sum of the vapour loads for the sequence would provide a quantitative indicator of the performance of the sequence. However, it has limitations such as it is not directly related to energy demand or operating costs and the capital costs are only partially accounted for. The calculation of vapour load can be made using the reflux ratio as shown in Figure 2.4.

As the minimum reflux ratio R_{min} can be calculated from the Underwood equation and the minimum vapour load V_{min} based on R_{min} is,

$$V_{min} = (R_{min} + 1)\,D \tag{2.5.1}$$

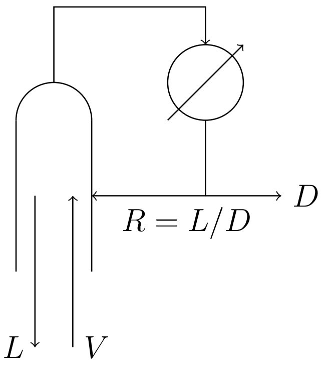


Figure 2.4: Schematic representation of the vapour load.

The typical values of R (1.1 - 1.3 times of R_{min}) have implicitly accounted for a capital-energy trade-offs.

The total vapour load in a distillation sequence is thus the addition of the vapour load for each column.

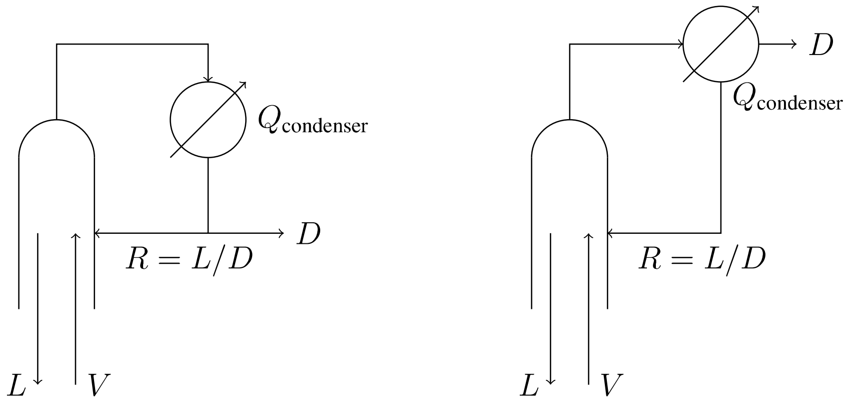
2.5.2 Energy demand

Energy demand is another useful indicator and allows total energy cost to be estimated. The energy demand can be calculated from the energy balances effectively from the vapour load. It should be pointed out that the heat of vaporisation depends on composition and temperature of stream (use product composition).

For a total condenser shown in Figure 2.5(a), the vapour to be condensed is V, thus the energy needed for the condensation is,

$$Q_{condenser} = V \cdot \Delta\, H^{vap} \tag{2.5.2}$$

where $\Delta\, H^{vap}$ is the enthalpy of vaporisation.



(a) Total condenser

(b) Partial condenser

Figure 2.5: Energy demand for condensers.

Similarly, for a partial condenser, Figure 2.5(b),

$$Q_{condenser} = L \cdot \Delta\, H^{vap} \tag{2.5.3}$$

where $L = RD$.

2.5.3 Costs

- Operating costs. This can be determined by knowing the reboiler, condenser and preheat duties, and given costs of utilities (e.g. steam, cooling water, refrigeration power). It should be noted that costs of utilities are highly site-specific and vary significantly from time to time.
- Capital costs. This can also be estimated, based on short-cut design results, e.g. using the Fenske equation to calculate the minimum number of theoretical stages (N_{min}) then using the Gilliland correlation to relate reflux ratio and number of stages to R_{min} and N_{min}. However, the capital cost models are typically highly inaccurate (±30-50%) and the actual cost of a installed equipment is typically 4 times purchase cost of the equipment.
- Total annualised costs account for both operating and capital costs.

2.6 Distillation Sequencing with Complex Columns

In addition to simple distillation columns, complex distillation columns can be considered. These can have more than 2 product streams, or have linking between columns by two-directional flows with one column having no condenser, or no reboiler - often referred to as thermally coupled distillation columns.

In sequencing distillation with complex columns, heuristics for simple columns are still available and useful as they provide qualitative guidance with insights; however, extra heuristics for the complex columns are also available.

2.6.1 Side-Steam Columns

A side-stream distillation column may replace two simple columns for some applications, sometimes at considerable savings in energy and investment. Intuitively, the side stream should contain primarily middle boiling components from a multicomponent mixture. Side streams may be useful when the middle boilers are the main product, as in a pasteurization column where the lighter trace components leave overhead.

Based on experience of using these column types, and simulations of ideal systems [2] heuristics have been suggested for when these columns provide a potential economic advantage.

Heuristic 1 For a pure sidestream product, for a three component mixture, A-B-C, if the feed has B $> 50\%$ and and C $< 5\%$, use a vapour sidestream, as B is more volatile than the other dominant component, C, at the draw stage. As shown in Figure 2.6(a).

Heuristic 2 For a pure sidestream product, for a three component mixture, A-B-C, if the feed has B $> 50\%$ and and A $< 5\%$, use a liquid sidestream, as B is less volatile than the other dominant component, A, at the draw stage. As shown in Figure 2.6(b).

2.6.2 Side-stripper arrangement

The use of complex nonstandard distillation columns can sometimes lead to substantial savings in capital costs as well as in energy consumption in comparison with conventional

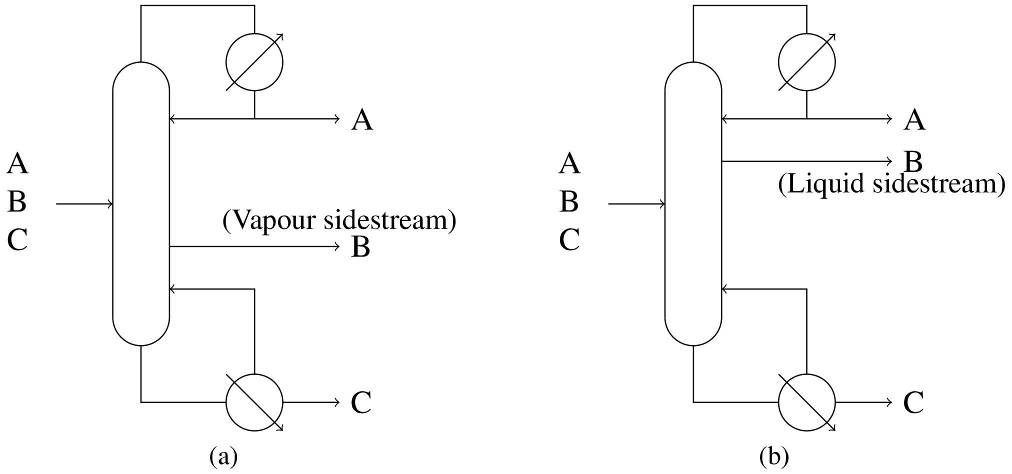
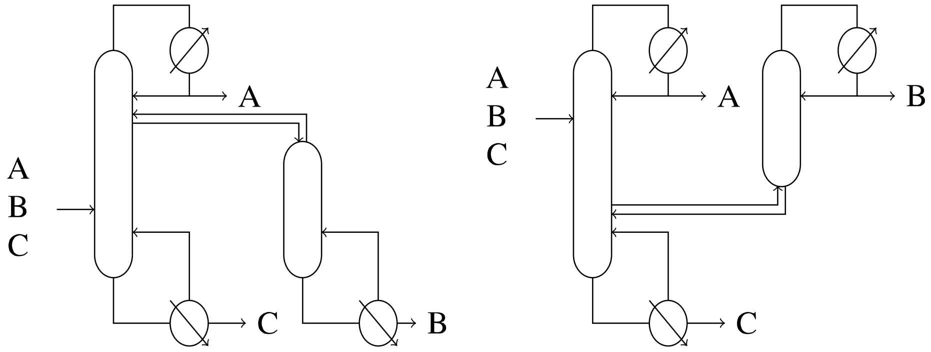


Figure 2.6: (a) Vapour side-stream for separating middle and heavy components, (b) Liquid side-stream for separating middle and light components.

one-feed two-product distillation columns. Complex columns are also suitable for retrofit design, since they can often be implemented with only small modifications to existing columns.

As can be seen from Figure 2.7(a), for the side-stripper arrangement, the column on the right hand side effectively takes liquid from and feeds vapour to the left column. For the right column a condenser is saved.



(a) Side-Stripper

(b) Side-Rectifier

Figure 2.7: Thermally coupled distillation configurations, (a) side-stripper and (b) side-rectifier.

The side-stripper sequence is a reconfiguration for the indirect sequence, Figure 2.2(b). As in the indirect sequences Figure 2.8(a), the left hand column produces only the bottom product and has part of the condensed liquid being fed into the right column and part of the liquid being refluxed. The idea was to remove the total condenser from the left column and to connect two streams to the right column, therefore to take liquid from the right column but feed vapour to it as needed (Figure 2.8(b)), which was then rearranged to become Figure 2.8(c).

Based on experience of using side-strippers, and calculations on energy use [1] heuristics have been suggested for when these columns provide a potential economic advantage. Figure 2.9(b) shows the percent savings when using a side stripper over the equivalent two simple column system.