Chapter 3: Introduction to Column Distillation

Distillation is the ***MOST common separation technique*** in chemical process industry, accounting for 90-95% of the separation operations

This is why more than a half of this course is devoted to *"distillation"* or *"column distillation"*, which includes

- *Binary-mixture* distillation
- *Multi-component* distillation
- *Extractive & azeotropic* distillation
- *Batch* distillation

3.1 Distillation Cascade

Students have already been introduced to the *"flash distillation"* and how to perform the calculations for flash distillation

Although *flash distillation* is a *very simple* (probably "simplest") separation technique, it produces a limited amount of separation (*i.e.* it **cannot** yield a product with a ***desired*** **purity**)

From the Examples in Chapter 2, it is clear that the *flash distillation* yields *liquid* and *vapour* phases that comprise *more than one substance* (*i.e.* the liquid and vapour streams are **not pure**)

In order to obtain a *single-species* product for each of liquid and vapour phases, a separation technique called the ***distillation cascade*** is employed; the details of this technique are as follows

Let's consider the separation of such a binary mixture as ethanol (EtOH)-water system, in which **EtOH** is ***more volatile*** (*i.e.* has a *higher vapour pressure* or a *lower boiling temperature*); thus, for this system, *water* a *less volatile* species

From the flash distillation, it is evident that the *vapour* product contains a high concentration of *more volatile species*; on the other hand, the *liquid* product has a high concentration of *less volatile component(s)*

In order to produce a *vapour* product with a higher concentration of *more volatile* species, the vapour product must be condensed to liquid and, subsequently, passed through the flash column for another flash distillation

Alternatively, the *vapour* stream is pressurised (to yield a *higher-pressure* vapour stream) and then sent to another flash tank; note that the pressure of the second flash tank is *higher* than that of the first one

At the other end (*i.e.* the *liquid* product), in order to obtain a *liquid* product with a higher (or pure) concentration of *less volatile* species, the liquid stream is passed through an expansion valve (to reduce its pressure) and, then, sent to another flash column, operating at a *lower* pressure (than the first one)

Figure 3.1 illustrates the configuration of the above description, which is, in fact, the *simple* distillation cascade; note that

- the *first* column (or the feeding flash column) is denoted as “3”
- the *higher-pressure* column is denoted as “2”
- the *lower-pressure* column is denoted as “4”

Note that a pump or a compressor is used to increase the pressure of the vapour product

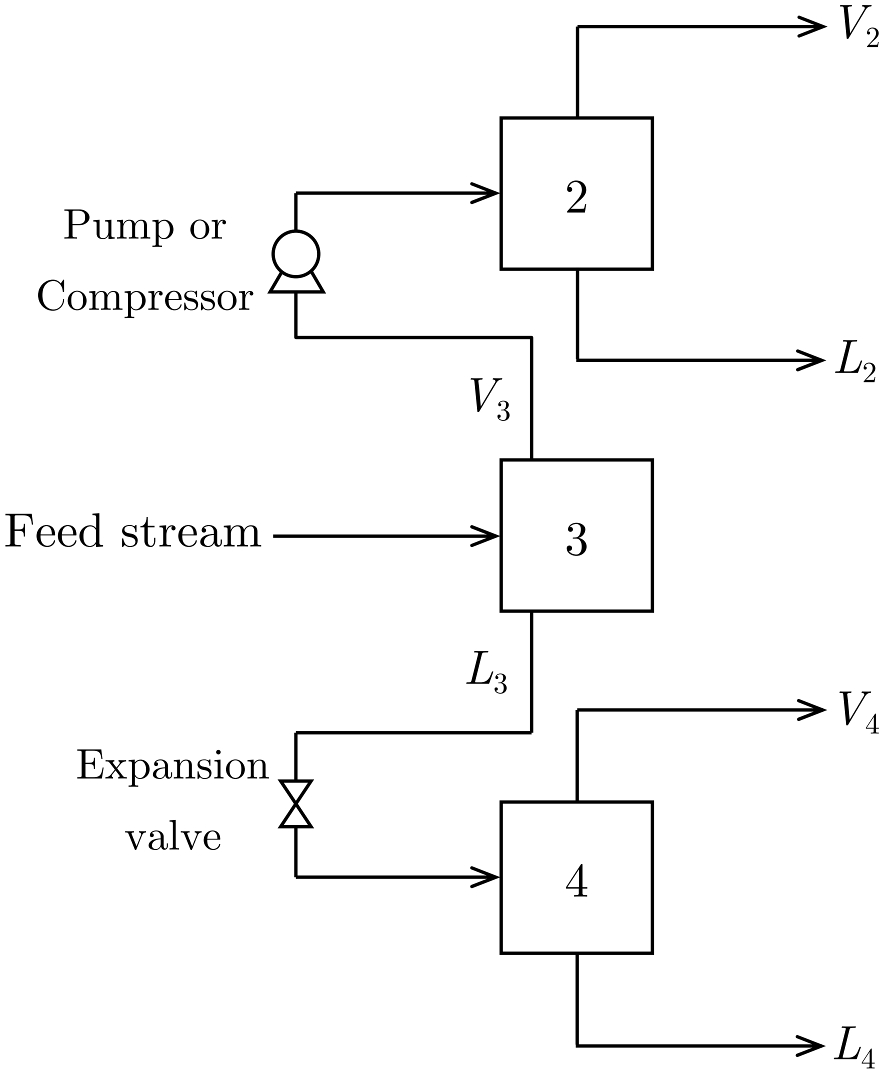

Figure 3.1: The configuration of a *simple* distillation cascade

The *vapour* product from the flash column **# 2** contains an even higher concentration of *more volatile* component(s)

At the other end, the *liquid* product from the flash column **# 4** has an even higher concentration of *less volatile* component(s)

To obtain a higher concentration of *more volatile* species in the *vapour* phase, more *higher-pressure* flash columns and the accompanying pumps or compressors are added on top of the flash column # 2

Likewise, to obtain a higher concentration of *less volatile* species in the *liquid* phase, more *lower-pressure* flash columns and the accompanying expansion valves are added to the bottom of the flash column # 4

The system that comprises the *multiple flash columns* with an *increasing pressure* from the *lowest* column *to* the *highest* one (or with a *decreasing* pressure from the *top-most* column *to* the *lowest* one) is called the ***"distillation cascade"***, which can be illustrated by Figure 3.2; note that, in Figure 3.2, $P_1 > P_2 > P_3 > P_4 > P_5$

By using this distillation cascade, the ***final vapour*** product, *e.g.*, $\boldsymbol{V_1}$, is very *rich* (or even pure) in *more volatile* component(s), while the ***final liquid*** product, *e.g.*, $\boldsymbol{L_5}$, is very *rich* (or pure) in *less volatile* species

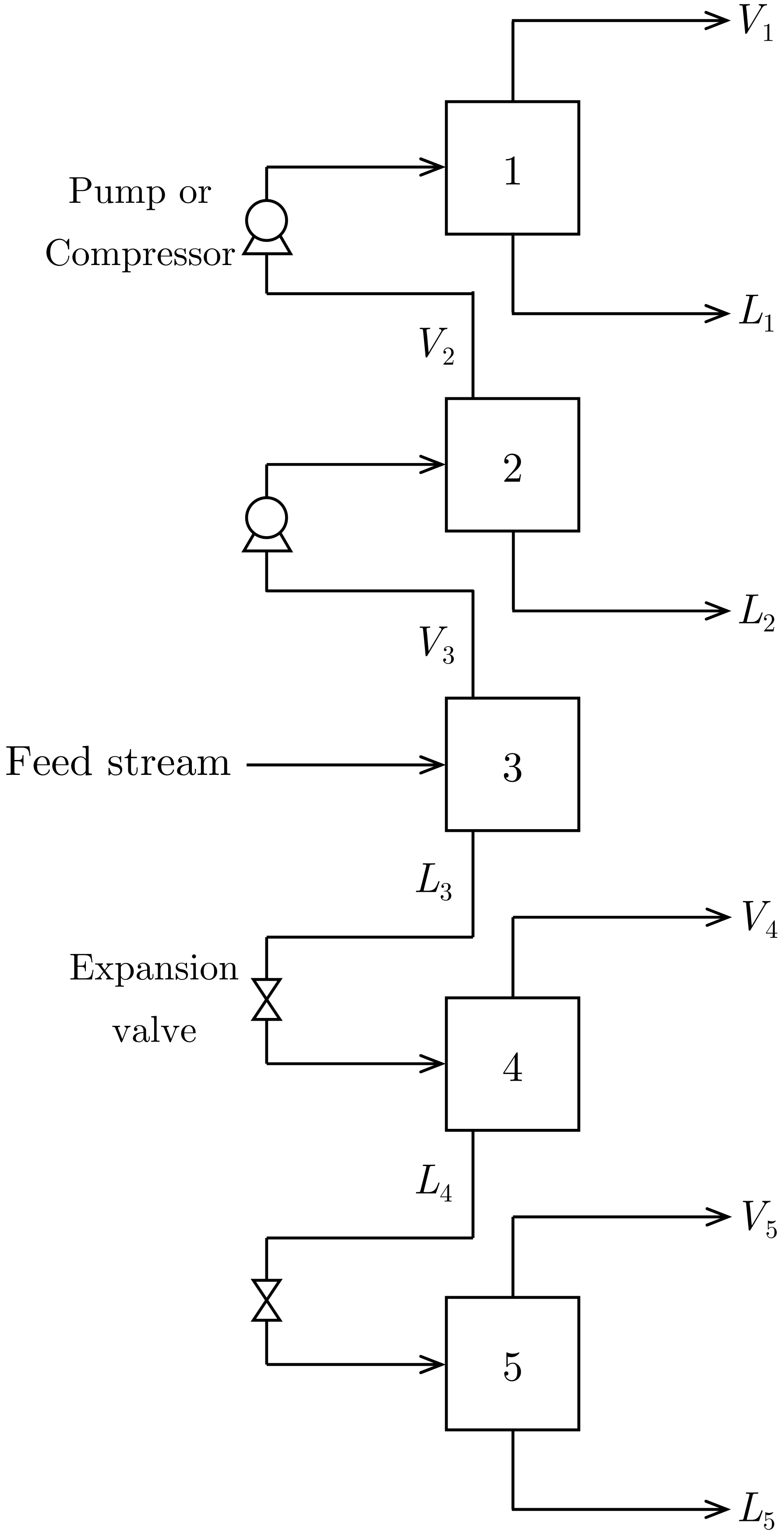

Figure 3.3: The distillation cascade

It should be noted, however, that, although we obtain the **final** *vapour product* with a very *high concentration* of the *more volatile* species and the **final** *liquid stream* containing a very *high concentration* of the *low volatile* component, we also get the *intermediate* products/streams (*e.g.*, L_1, L_2, V_4, and V_5,) with the *intermediate* concentrations (*i.e. not pure* or contain both more and less volatile species)

How could we deal/how should we do with these intermediate streams?

Let's start with the stream L_2

Since the stream L_2 is the *liquid* product of the column # 2, which, in turn, comes from the *vapour* stream of the column # 3 (or the feeding column), its *concentration* should be *similar* (but NOT equal) to that of the *feed stream* (as it is vaporised once and condensed once)

Thus, the stream L_2 can be fed back into the flash column # 3

Next, consider the stream V_4, the *vapour* product of the column # 4, as it is condensed once (in the flash tank # 3) and vaporised once (in the flash tank # 4), once again, its *concentration* should be close to that of the *feed stream*

Hence, this stream can also be fed back to the column/tank # 3

The ways to deal with the streams L_1 and V_5 are in the similar manner as *per* the streams L_2 and V_4, respectively

The resulting *new configuration* of the distillation cascade, which is commonly called the ***"counter-current distillation cascade"***, is shown in Figure 3.3

Note that there are **no intermediate products** in the *"counter-current distillation cascade"*

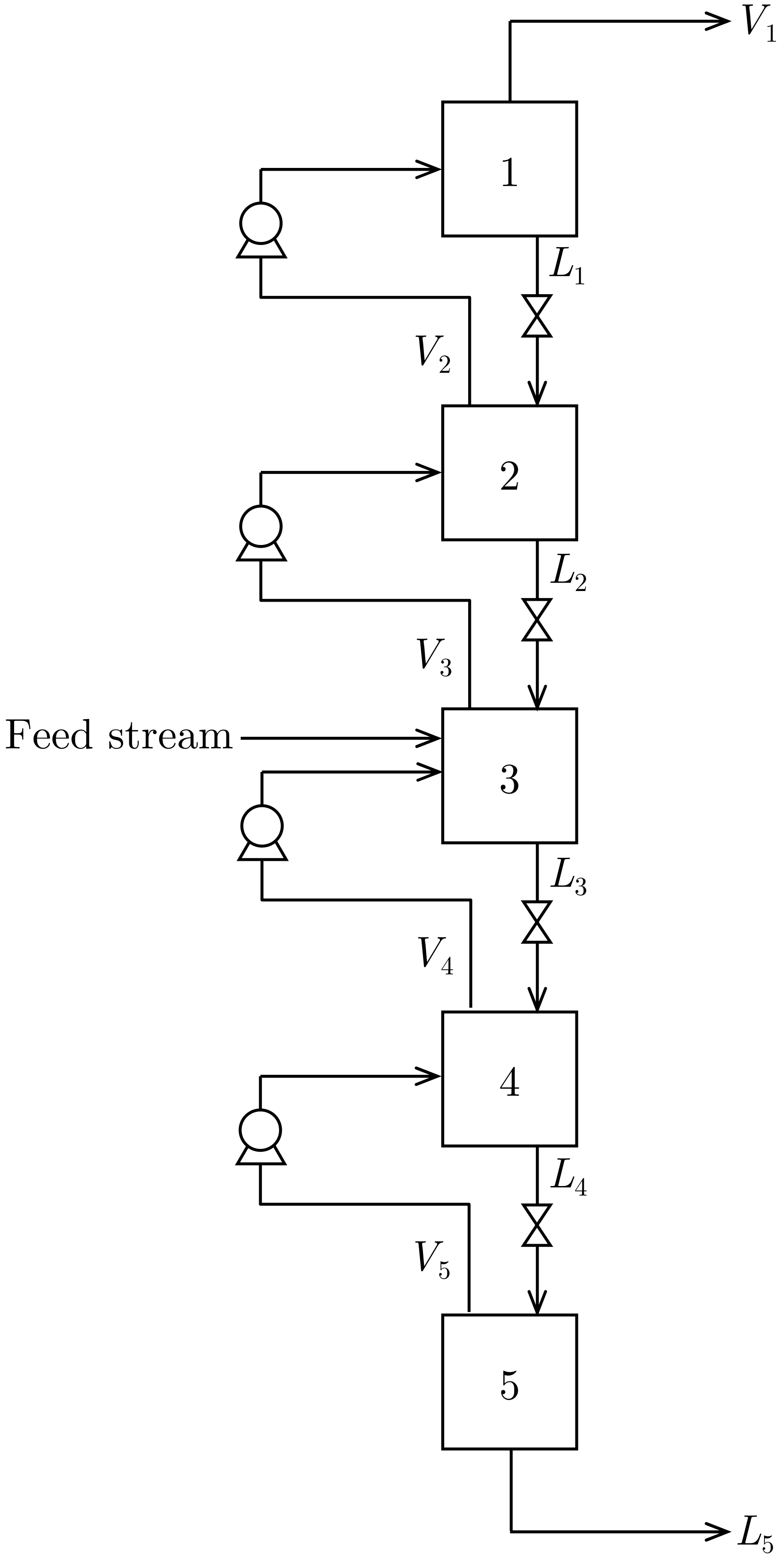

Figure 3.3: A counter-current distillation cascade

By using the counter-current distillation cascade, the *vapour* and *liquid* products with *very high concentrations* of (or even *pure*) *more volatile* and *less volatile* components, respectively, can be obtained

Unfortunately, however, the **distillation cascade** with each cascade operating at **different pressure** (but the whole system is at **nearly constant temperature**) is **NOT** ***practical***, as it requires a number of compressors, which are rather expensive; additionally, the *effect* of *pressure* on the vapour-liquid *equilibrium* is *not so significant* (on the contrary, the *effect* of *temperature* on the equilibrium is rather *strong*)

Hence, *to be more practical*, the distillation cascade that operates such that the **temperature** of each cascade is **varied**, while the *pressure* of the whole system is kept *constant* (or nearly constant), is proposed

The *principal* idea of this configuration of distillation cascade is that the *top (upper)* part of the cascade contains high concentrations of *more volatile* components, which boils at low temperatures, whereas the *bottom (lower)* part is concentrated with *less volatile* species, which boils at higher temperature

Additionally, we have learned previously that the *phase* changes in which

- liquid becomes vapour is an *endothermic* process
  (*i.e.* heat/energy must be provided to the system)

and

- vapour is condensed to liquid is an *exothermic* process
  (*i.e.* heat/energy is released from the system)

Accordingly, the operation or the design of the *temperature-varying* distillation cascade is as follows:

- The *top* or *vapour* product is totally *condensed*, using a condenser, to *liquid*, and then split into 2 streams: one is the *product stream*, which is called the *distillate* and denoted as D, another one is the stream that is returned to the cascade, which is called the reflux and denoted as L_o
- The *bottom* product is partially *vaporised*, using a re-boiler, and then fed back (as *vapour*) to the cascade (*i.e.* stream V_6 in Figure 3.4)

Note that the *bottom product* that is *not vaporised* is the *end product*, which is denoted as B; the *end product* is normally *liquid*

The configuration of the *temperature-varying* counter-current distillation cascade is illustrated in Figure 3.4

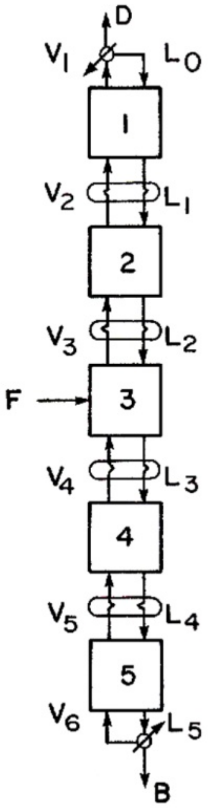

Figure 3.4: The *temperature-varying* counter-current distillation cascade

(from "Separation Process Engineering" by Wankat, 2007)

The ratio between the reflux $\left(L_o\right)$ and the distillate $\left(D\right)$ is the **reflux ratio**

From Figure 3.4, it is evident that, between each stage, the *vapour* and the *liquid* phases are *in contact* with *each other*

During the contact of the liquid and the vapour phases, the *vapour* phase, especially the *less volatile* species (whose *boiling temperature* is relatively high), is partially *condensed*, while the *liquid* phase, particularly the *more volatile* components, is partially *vaporised*

The *condensation* of the *vapour phase* (to liquid) *provides heat* required for *vaporising* the contacting *liquid phase*

With reference to the *more volatile* species, since the section ***above*** **the feeding point** is ***enriched*** **with** ***more volatile*** **components**, it is called the **“enriching”** or **“rectifying”** section

Meanwhile, the section ***below*** **the feeding point** is ***concentrated*** **with** ***less volatile*** **species**, resulting from the fact that the ***more volatile*** **components** are ***stripped out*** of the liquid phase by the rising vapour phase; hence, this section is called the **“stripping”** section

The practical distillation column widely used in chemical process industry is exemplified in Figure 3.5

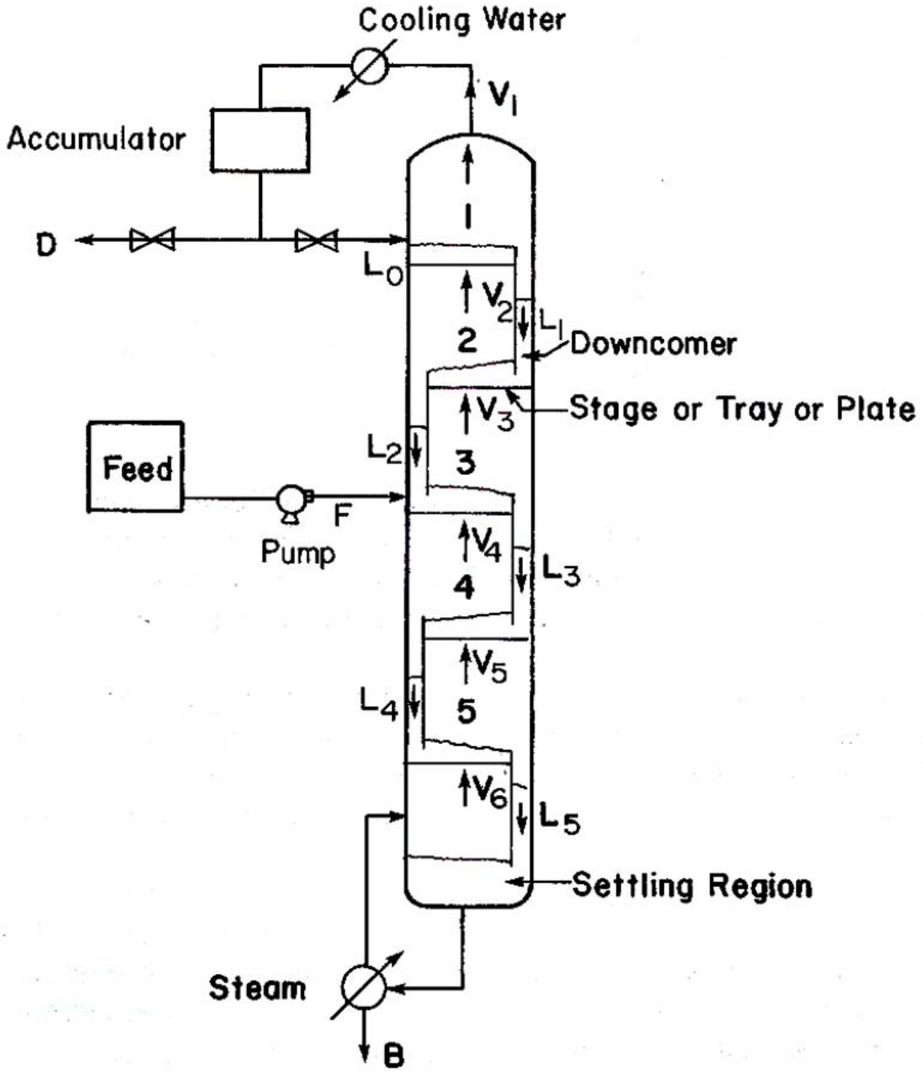

Figure 3.5: An illustration of the distillation column

(from "Separation Process Engineering" by Wankat, 2007)

The contact between the liquid and the vapour phases within each stage (which is practically called **"tray"**) is illustrated in Figure 3.6

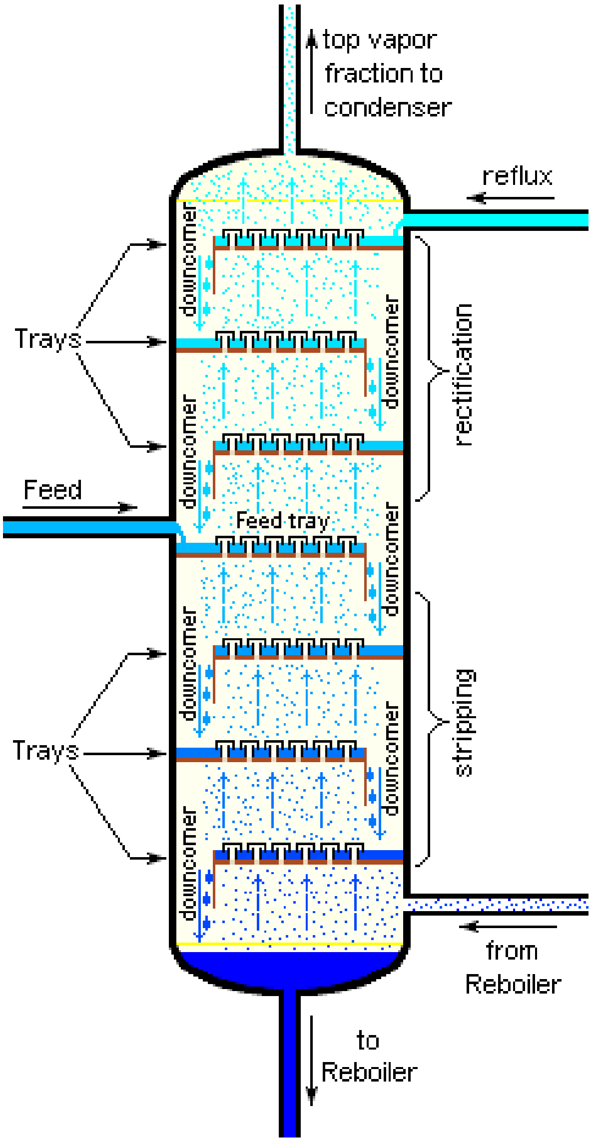

Figure 3.6: An illustration of the contact between the liquid and the vapour phases within each stage of the distillation column

(from http://wikipedia.org)

The tray shown in Figure 3.6 is called a *bubble-cap* tray, which is one among many types of tray of the distillation column

The simplest type of tray is the *sieve* tray, which is a metal plate with several holes, as illustrated in Figure 3.7

Note that a *weir (ที่กั้น)* is used to control the flow rate of the liquid phase by detaining liquid to a certain level, in order to *enhance* the *contact time* between the liquid and the vapour phases

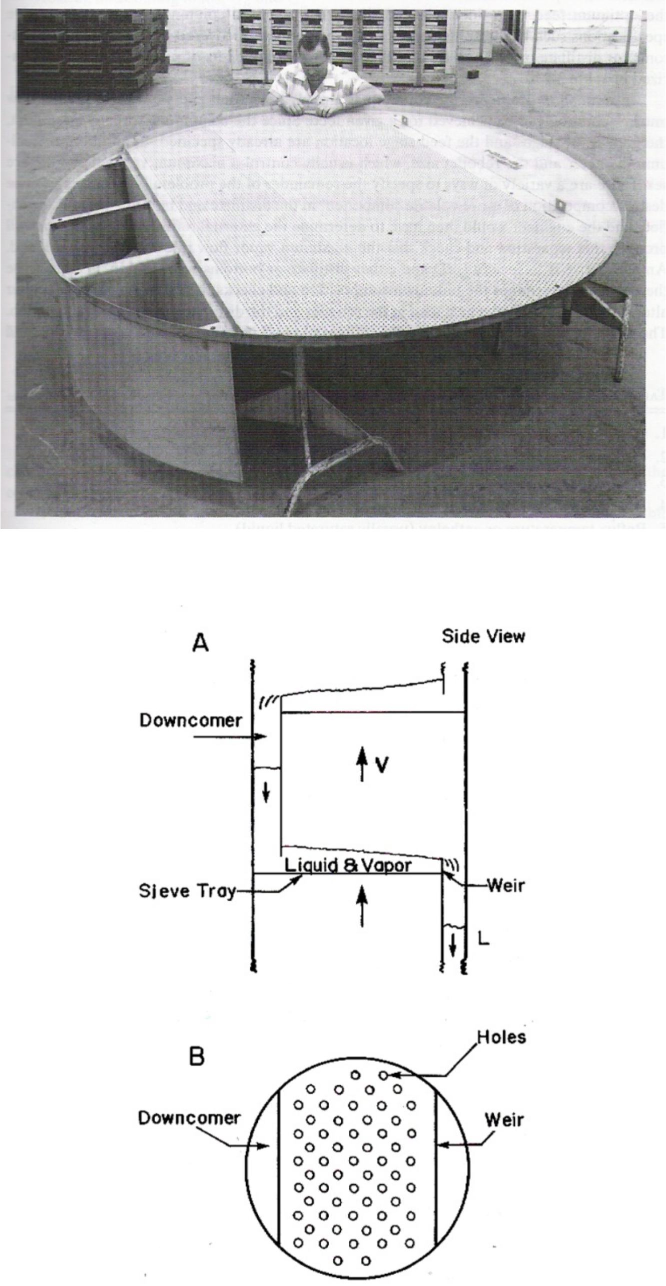

Figure 3.7: An illustration of a sieve tray

(from “Separation Process Engineering” by Wankat, 2007)

Normally, the space between trays (or the height of each stage) varies from 2 to 48 inch (~5-135 cm)

Within the distillation column, there are **4 *flow* regimes**, *depending on* the *flow rate* of the rising *vapour* stream (*gas* phase):

- ***Bubble* regime:** the *vapour flow rate* is *low*; thus, the *liquid phase* is close to being *stagnant (นิ่ง)* with only small bubbles rising through it

  In this regime, the *contact* between the vapour and the liquid phases is *low*, thus resulting in a *low stage efficiency*
- ***Foam* regime:** the *vapour flow rate* is *higher* (than the bubble one); hence, the *vapour phase* is *rapidly rising* through the liquid phase, which causes a *good mixing* between the vapour and the liquid phases, thus yielding a relatively high stage efficiency

It should be noted, however, that *if the foam is too stable* or *if there is too much foam*, the foam may fill the gap or space between each tray, and this phenolmenon is called ***"flooding"***

The *foam regime* in the distillation column can be exemplified by the foam at the top of a glass of beer in Figures 3.8 and 3.9

Figure 3.8: An illustration of the foam regime, in which the bubbles (or the vapour phase) are rising through the stagnant foam

(a) When the foam is at normal level

(b) When the foam is too thick (flooding)

Figure 3.9: An illustration of the thickness of the foam layer

- ***Forth* regime:** the *vapour flow rate* is *even higher* (than the foam one), thus causing the *surface* of the *liquid phase* to is *boil violently*, and some splashing may also be observed

In this regime, the contact or the mixing between the liquid and the vapour phases is excellent, thus resulting in a very good column efficiency

Accordingly, most of the commercial distillation columns are operated in this regime

- ***Spray* regime:** the *vapour flow rate* is *very fast* (*too fast*) until the liquid phase, through which the vapour is passed, becomes small droplets; thus lowering the contact time between the liquid and the vapour phase, which, in turn, yields a low column efficiency

Let’s get back to Figure 3.5 or 3.6

Between each stage, the *rising* vapour phase $\left(V_k\right)$ exchanges both mass and energy (or heat) with the *down-coming* liquid phase $\left(L_{k-1}\right)$

As described previously, the *liquid phase* flowing down *from the top* is a result of the *condensation* of the rising *vapour phase*; hence, it *contains* a *high amount* of *more volatile* components

On the other way around, the *rising vapour phase* is from the *vaporisation* of the *liquid phase* from the bottom part, it contains very *higher amounts* of *less volatile* species

The *more volatile* species in the down-coming *liquid phase* boil at low temperatures

In order for liquid to boil, heat or energy is required (*i.e.* the boiling is an *endothermic* process)

The *heat* for boiling liquid is *provided by* the *rising vapour phase*

When the heat comes out of the rising vapour phase (*i.e.* an exothermic process), the *less volatile components* in the vapour phase *condense*

Normally, as the latent heat of vaporisation (ความร้อนแฝงของการกลายเป็นไอ) *per* mole is approximately constant with reference to tempearture, the condensation of 1 mole of a less volatile component essentially provides the heat just enough for vaporising 1 mole of a more volatile component

This implies that, at each stage, it is reasonable to assume that the *process* is *adiabatic* (*i.e.* the net Q is 0)

In addition to an exchange in heat or energy between the liquid and the vapour phases, mass transfer of the species also takes place

The *more volatile* components in the *down-coming liquid phase* that boil and thus become vapour *migrate* (ຍ້າຍໄປ) *into* the *vapour phase*

Meanwhile, the *less volatile* species in the *rising vapour phase* that condense and thus become liquid *transfer* into the *liquid phase*

Eventually, after passing, stage-by-stage, through the whole distillation column, the ***top*** **product** contains very **high concentrations** of ***more volatile*** **components**, and the ***bottom*** **product** comprises very **high concentrations** of ***less volatile*** **species**

For example, for a **EtOH-water** binary mixture, since **EtOH** is ***more volatile*** than water (*i.e.* the boiling point of EtOH is lower than that of water), the **top product** contains a high concentration of **EtOH**, while the **bottom product** consists of a high concentration of **water**

In addition to the boiling point, in order to determine which component would tend to be vapour when it enters the distillation column, the following technique is employed

From the flash distillation, we have learned that, if the component (species) is more volatile, when it enters the column, the fraction of such species that becomes vapour is higher than the fraction that becomes liquid, which can be implied *mathematically* as follows

$$y_i V > x_i L \tag{3.1}$$

Re-arranging Eq. 3.1 gives

$$\left(\frac{y_i}{x_i}\right)\frac{V}{L} > 1 \tag{3.2}$$

but, from Eq. 2.26, $\frac{y_i}{x_i} = K_i$

Thus, Eq. 3.2 becomes

$$K_i \frac{V}{L} > 1 \tag{3.3}$$

Eq. 3.3 implies that, for the same V and L, and the same given temperature and pressure, the components with higher values of K_i tend to be more vapour than liquid when they enter the distillation column, given that $K_i \frac{V}{L} > 1$

3.2 Specifications of the Distillation Column

For both *design* (to design a new distillation column) and *operation* or *simulation* (to operates the existing column) problems, the following variables are usually specified:

- Column pressure (which sets the *equilibrium data*)
- Feed flow rate $\left(F\right)$
- Feed composition $\left(z_i\right)$
- Feed temperature or enthalpy or quality (*i.e.* the ratio between the mass of the vapour phase and the total mass of the feed)
- Reflux temperature or enthalpy (usually the *reflux* is a *saturated liquid*)

For the *design* problems, the specified (given) variables and the variables that engineers have to calculate for each case of problem (A–D) are summarised as follows

Case	**The specified variables**	**The variables that engineers have to calculate**
A	• Mole fraction of more volatile component in distillate, x_D • Mole fraction of more volatile component in bottom, x_B • Reflux ratio, L_o/D • Optimal feed plate	• Distillate and bottom flow rates, D and B • Heating and cooling load, Q_R and Q_C, respectively • Number of stages (plates), N • Column diameter
B	• D or B • x_D or x_B • Reflux ratio • Optimal feed plate	• B or D • x_B or x_D • Q_R and Q_C • N & N_{feed} • Column diameter

Case	**The specified variables**	**The variables that engineers have to calculate**
C	• x_D and x_B • Boil-up ratio, V/B • Optimal feed plate	• D and B • Q_R and Q_C • N & N_{feed} • Column diameter
D	• Fractional recoveries of a component in distillate $(FR_i)_D$ • Fractional recoveries of a component in bottom $(FR_i)_B$ • Reflux ratio • Optimal feed plate	• x_D and x_B • Q_R and Q_C • N & N_{feed}

Note that the *optimal feed plate* is the feed location/stage that gives the *fewest number* of total *stages* (*plates*) of the distillation column

For the *simulation* problems, the specified (given) variables and the variables that engineers have to calculate for each case (A–D) are summarised as follows

Case	**The specified variables**	**The variables that engineers have to calculate**
A	• N & N_{feed} • x_D and x_B • Column diameter	• Reflux ratio • B and D • Q_R and Q_C • Check if $V > V_{\max}$
B	• N & N_{feed} • Reflux ratio • x_D or x_B • Column diameter	• x_B or x_D • B and D • Q_R and Q_C • Check if $V > V_{\max}$
C	• N & N_{feed} • x_D or x_B • Column diameter (set $V =$ a fraction × $V_{\max}$)	• Reflux ratio • x_B or x_D • B and D • Q_R and Q_C

Case	The specified variables	The variables that engineers have to calculate
D	• N & N_{feed}	• x_B or x_D
	• Q_R	• B and D
	• x_D or x_B	• Q_R and Q_C
	• Column diameter	• Check if $V > V_{\max}$

Note that $V_{\max}$ is the maximum allowable amount of the volume of the vapour phase in the feed for the given temperature and pressure

3.3 External (Mass & Energy) Balances of the Distillation Column

When the *pressure* of the distillation column, shown in Figure 3.10, is *specified*, and with the assumption that the *column* is *well insulated* (thus, the column is *adiabatic*), the *external* material balances can be formulated as follows

<u>Overall mass (mole) balance</u>

$$F = B + D \tag{3.4}$$

Note that the *bottom* (or the *bottom product*: B) and the *distillate* $\left(D\right)$ are both *liquid*

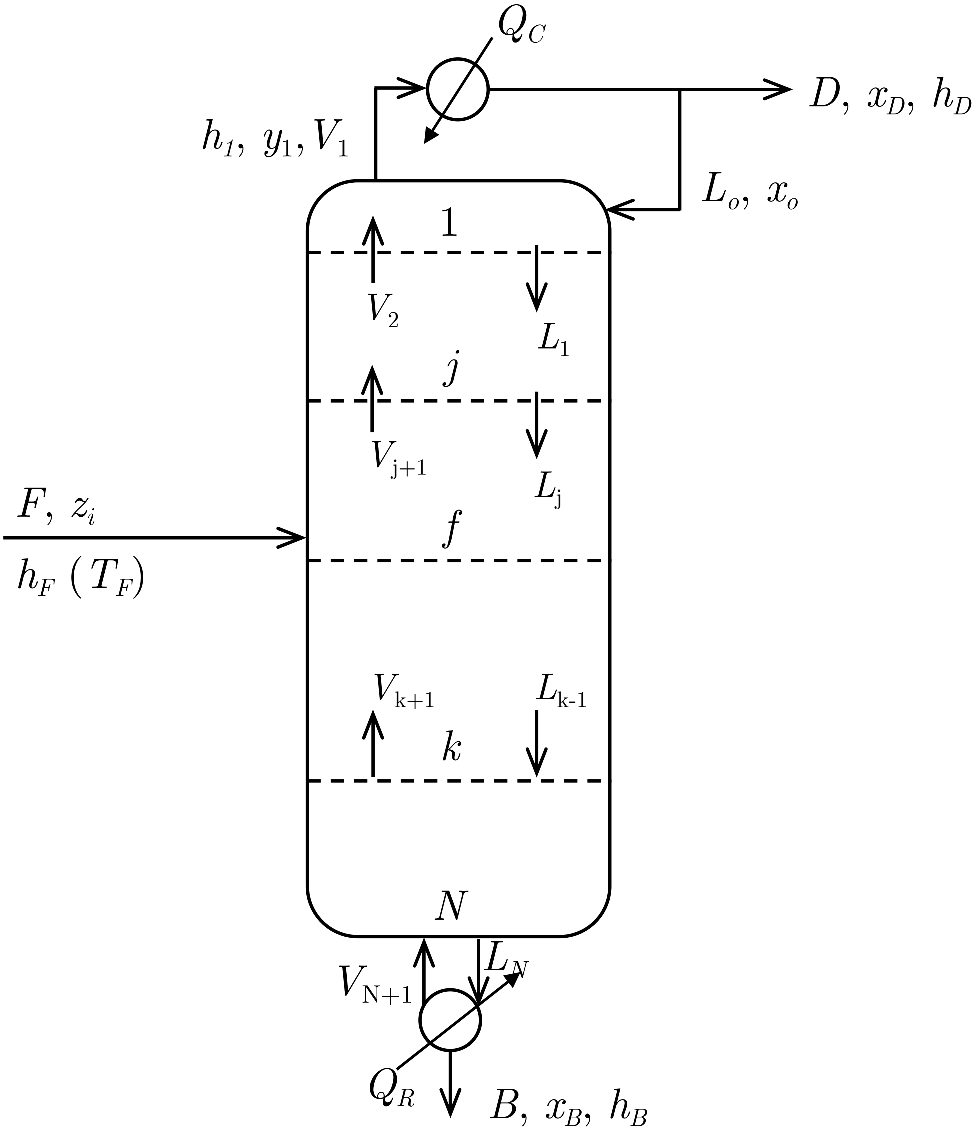

Figure 3.10: A distillation column

Species balance (for more volatile species)

$$z_i F = B x_B + D x_D \tag{3.5}$$

When x_B and x_D, as well as z_i and F, are specified, Eqs 3.4 and 3.5 can be solved simultaneously for B and D

Once B and D are obtained, the energy balance can be carried out as follows

$$h_F F + Q_R + Q_C = h_D D + h_B B \qquad (3.6)$$

Note that Q_C and Q_R are treated as the heat input; the resulting *sign* (+ or –) indicates the *direction* of the heat transfer

Normally, the enthalpy of each stream can be specified as follows

$$h_F = h_F\left(z_i, T_F, P\right) \qquad (3.7a)$$

$$h_D = h_D\left(x_D, T_{\text{reflux}}, P\right) \qquad (3.7b)$$

$$h_B = h_B\left(x_B, \text{sat. liq.}, P\right) \qquad (3.7c)$$

In Figure 3.10, the stream V_1 enters the *condenser* with the composition of y_1 and is then condensed and separated into 2 streams: the streams D (the *distillate*) and L_o (the *reflux*) with the compositions of x_D and x_o, respectively

Since there is no chemical reaction at the condenser, the composition does not change (only phase change is taken place), it results in the fact that

$$y_1 = x_D = x_o \tag{3.8}$$

Performing a mass (mole) balance *around the condenser* yields

$$V_1 = D + L_o \tag{3.9}$$

Dividing Eq. 3.9 with D results in

$$\frac{V_1}{D} = 1 + \frac{L_o}{D} \tag{3.10}$$

Note that $\frac{L_o}{D}$ = *reflux ratio*

Re-arranging Eq. 3.10 gives

$$V_1 = \left(1 + \frac{L_o}{D}\right) D \tag{3.11}$$

Normally, the *reflux ratio* is known (given), and D (the amount of the distillate) is obtained from solving Eqs. 3.4 and 3.5

Thus, the value of V_1 can be computed using Eq. 3.11

Once V_1 is obtained, an *energy balance around the condenser* can be performed as follows

$$h_1 V_1 + Q_C = h_D D + h_o L_o \tag{3.12}$$

Since the streams D and L_o are both *liquid* and has the same composition (*i.e.* $x_D = x_o$), it results in the fact that

$$h_D = h_o \tag{3.13}$$

Eq. 3.12, then, becomes

$$h_1 V_1 + Q_C = h_D \left(D + L_o \right) \tag{3.14}$$

but, from Eq. 3.9, $V_1 = D + L_o$

Hence, Eq. 3.14 can be re-arranged as follows

$$\begin{gathered} h_1 V_1 + Q_C = h_D V_1 \\ Q_C = h_D V_1 - h_1 V_1 \end{gathered}$$

$$Q_C = V_1\left(h_D - h_1\right) \tag{3.15}$$

Normally,

$$h_1 = h_1\left(y_1, \text{sat. vap.}, P\right)$$
$$h_D = h_D\left(x_D, \text{sat. liq.}, P\right)$$

and $y_1 = x_D$

Accordingly, $h_1 - h_D$ is, in fact, the latent heat of vaporisation of the liquid with the composition of x_D at a given pressure

Note that, since $h_D < h_1$, Q_C is *negative*, meaning that it is an *exothermic* process (at the *condenser*)

Once Q_C is obtained, the value of Q_R can then be calculated using Eq. 3.6:

$$h_F F + Q_R + Q_C = h_D D + h_B B \qquad (3.6)$$

Note that, by performing similar material and energy balances *around the re-boiler*, the equation that can be used to compute Q_R is obtained as follows

$$Q_R = V_{N+1}\left(h_{N+1} - h_B\right) \qquad (3.16)$$

where h_{N+1} and h_B are the enthalpies of the streams V_{N+1} are B, respectively, and $h_{N+1} - h_B$ is the latent heat of vaporisation of the liquid with the composition of x_B at a given pressure

Note also that Q_R is positive (*i.e.* the heat transfer at the *re-boiler* is *endothermic*)

Example A steady-state, counter-current, *staged* distillation column, operated at 1 atm, is to be used to separate EtOH from water

The feed comprises 30 wt% EtOH and the remaining water at 40 °C and has a flow rate of 10,000 kg/h

The reflux ratio $\left(L_o / D\right)$ is 3.0, and the desired bottom composition $\left(x_B\right)$ and distillate composition $\left(x_D\right)$ are 0.05 (weight fraction of EtOH) and 0.80, respectively

Find D, B, Q_C, and Q_R

Given h_D = 60 kcal/kg, h_B = 90 kcal/kg, h_F = 30 kcal/kg, and h_1 = 330 kcal/kg

Overall balance

$$F = B + D$$

$$10,000 = B + D \tag{3.17}$$

Species balance (for EtOH)

$$z_i F = Bx_B + Dx_D$$

$$\left(0.3\right)\left(10,000\right) = \left(0.05\right)B + \left(0.8\right)D \tag{3.18}$$

Solving Eqs. 3.17 and 3.18 simultaneously yields

$$B = 6,667 \text{ kg/h}$$

$$D = 3,333 \text{ kg/h}$$

Performing the *mass balance* around the *condenser* gives

$$V_1 = D + L_o \qquad (3.9)$$

It is given that the reflux ratio, $\frac{L_o}{D}$ is 3.0

Thus,

$$L_o = 3.0D \qquad (3.19)$$

Combining Eqs. 3.9 and 3.19 together yields

$$\begin{aligned} V_1 &= D + \left(3.0D\right) \\ &= 4.0D \\ &= 4.0\left(3,333\right) \end{aligned}$$

$$V_1 = 13,332 \text{ kg/h}$$

Thus, Q_C can then be computed using Eq. 3.15 as follows

$$\begin{aligned} Q_C &= V_1\left(h_D - h_1\right) \\ &= \left(13,332\right)\left(60 - 330\right) \end{aligned}$$

$$Q_C = -3,599,640 \approx -3,600,000 \text{ kcal/h}$$

Note that the latent heat of vaporisation of liquid with $x_{\text{EtOH}} = 0.8$ is $\left|60 - 330\right| = 270$ kcal/h

Eventually, Q_R can be calculated using Eq. 3.6:

$$h_F F + Q_R + Q_C = h_D D + h_B B \tag{3.6}$$

as follows

$$\begin{aligned} Q_R &= h_D D + h_B B - h_F F - Q_C \\ &= \left(60\right)\left(3,333\right) + \left(90\right)\left(6,667\right) \\ &\quad - \left(30\right)\left(10,000\right) - \left(-3,599,640\right) \end{aligned}$$

$$Q_R = 4,099,650 \text{ kcal/h} \approx 4{,}100{,}000 \text{ kcal/h}$$