## Chapter 7:

## Batch Distillation

In the previous chapters, we have learned the distillation operation in the continuous mode, meaning that

- the feed(s) is(are) fed continuously into the distillation column
- the distillation products [e.g., distillate, bottom, side stream(s)] are continuously withdrawn from the column

In the continuous operation, after the column has been operated for a certain period of time, the system reaches a steady state

At steady state, the properties of the system, such as

- the feed flow rate
- the flow rates of, e.g., the distillate and the bottom
- the feed composition
- the compositions of the distillate and the bottom
- reflux ratio $\left(L_{o} / D\right)$
- system's pressure are constant

With these characteristics, a continuous distillation is the thermodynamically and economically efficient method for producing large amounts of material of constant composition

However, when small amounts of products of varying compositions are required, a batch distillation provides several advantages over the continuous distillation (the details of the batch distillation will be discussed later in this chapter)

Batch distillation is versatile and commonly employed for producing biochemical, biomedical, and/or pharmaceutical products, in which the production amounts are small but a very high purity and/or an ultra clean product is needed

The equipment for batch distillation can be arranged in a wide variety of configurations

In a simple batch distillation (Figure 7.1), vapour (i.e. the product) is withdrawn from the top of the re-boiler (which is also called the "still pot") continuously, and by doing so, the liquid level in the still pot is decreasing continuously


## Still Pot

Figure 7.1: A simple batch distillation
(from "Separation Process Engineering" by Wankat, 2007)

Note that the distillation system shown in Figure 7.1 is similar to the flash distillation

However, there are a number of differences
between the batch distillation (e.g., Figure 7.1) and the flash distillation: i.e.

- in the flash distillation, feed is continuous-
$l y$ fed into the column, whereas there is
no continuous feed input into the still pot for the batch distillation
- in the flash distillation, the products (i.e. vapour and liquid products) are withdrawn continuously from the system, whereas, for the batch distillation, the remaining liquid in the still pot is drained out of the pot (or the re-boiler) only at the end of the distillation

Another configuration of batch distillation is a constant-level batch distillation, which is similar to the simple batch distillation, as illustrated in Figure 7.1; however, in this configuration, the liquid (i.e. the feed) is continuously fed into the still pot (or the re-boiler) to keep the liquid level in the pot constant

The more complex batch distillation (than the simple and the constant-level batch distillation) is the multi-stage batch distillation

In this distillation system, a staged or packed distillation column is placed on top of the reboiler (or the still pot), as shown in Figure 7.2


Figure 7.2: A multi-stage batch distillation
(from "Separation Process Engineering" by Wankat, 2007)

In the usual operation of the multi-stage distillation system, the distillate is withdrawn continuously from the system, until the distillation is ended

Another way of operating the multi-stage
batch distillation is that the system is operated such that there is no distillate withdrawn from the column (or system), thus resulting in a continuous change in the concentration or composition of liquid in the pot (or the re-boiler)

Additionally, when a pure bottom product is required, an inverted batch distillation is employed

In this technique (i.e. the inverted batch distillation), the bottom product or the liquid in the re-boiler is withdrawn continuously while the distillate is withdrawn only at the end of the distillation

# 7.1 Binary-mixture Batch Distillation: Rayleigh 

 EquationThe material balances for the batch distillation are different from those for continuous distillation

In the batch distillation, the main focus is at the total amounts of input(s) [i.e. feed(s)] and outputs (e.g., distillate or bottom) collected at the end of the distillation, rather than the rates of such inputs and outputs

The material balances around the batch distillation system for the entire operating time are as follows

Overall:

$$
\begin{equation*}
F=W_{\text {final }}+D_{\text {total }} \tag{7.1}
\end{equation*}
$$

where
$F \quad=$ the total amount of feed fed into the distillation column for the entire operating period
$W_{\text {final }}=$ the final amount of liquid in the re-boiler (the notation $W$ is used because the remaining liquid in the still pot is normally a waste)
$D_{\text {total }}=$ the total amount of the distillate withdrawn from the distillation column (in some textbooks, the notation $D_{\text {final }}$ may be used)

Species balance (for a more volatile component: MVC):

$$
\begin{equation*}
x_{F} F=x_{w, \text { final }} W_{\text {final }}+x_{D, \text { avg }} D_{\text {total }} \tag{7.2}
\end{equation*}
$$

## where

$x_{F} \quad=$ mole fraction of a more volatile species in the feed
$x_{w, \text { final }}=$ the mole fraction of an MVC of the
remaining liquid in the re-boiler
$x_{D, \text { avg }}=$ an average concentration of an MVC in the distillate
(in some textbooks the notation $x_{D, \text { final }}$ may be used)

Normally, $F$ and $x_{F}$ are specified (or given in the problem statement), and the value of either $x_{w, \text { final }}$ or $x_{D, \text { avg }}$ is also specified (or given)

Thus, there are $\mathbf{3}$ unknowns for the binarymixture batch distillation system:

- $W_{\text {final }}$
- $D_{\text {total }}$
- either $x_{D, \text { avg }}$ or $x_{w, \text { final }}$

Problematically, however, by just performing material balances, we have only 2 equations (i.e. Eqs. 7.1 and 7.2)

Hence, another or additional equation is required

The additional equation for solving batch distillation problems is commonly known as the Rayleigh equation

To derive this equation, Lord Rayleigh (1902) employed the facts that (see Figure 7.1), at any instant of time,

1) the rate of the distillate flowing out of
the batch distillation system, $d D$, is equal
to the decreasing rate of the liquid in the
still pot, $-d W$
2) the rate of species $i$ in the distillate flowing out of the batch distillation system,
$x_{D} d D$, is equal to the decreasing rate of species $i$ the liquid in the still pot
$-d\left(W x_{w}\right)$

Thus, the following equations can be formu-
lated:

$$
\begin{gather*}
d D=-d W  \tag{7.3}\\
x_{D} d D=-d\left(W x_{w}\right) \tag{7.4}
\end{gather*}
$$

Note that it is assumed that, at any instant of time, the concentration or the composition of the distillate $\left(x_{D}\right)$ is constant

Combining Eq. 7.3 with Eq. 7.4 and re-arranging gives

$$
\begin{gathered}
-x_{D} d W=-d\left(W x_{w}\right) \\
-x_{D} d W=-W d x_{w}-x_{w} d W
\end{gathered}
$$

(7.5)

Re-arranging Eq. 7.5 and integrating the resulting equation yields

$$
\begin{gathered}
W d x_{w}=x_{D} d W-x_{w} d W \\
W d x_{w}=\left(x_{D}-x_{w}\right) d W \\
\frac{d x_{w}}{\left(x_{D}-x_{w}\right)}=\frac{d W}{W} \\
\int_{W=F}^{W=W_{\text {final }}} \frac{d W}{W}=\int_{x_{w}=x_{F}}^{x_{w}=x_{w, \text { final }}} \frac{d x_{w}}{x_{D}-x_{w}}
\end{gathered}
$$

which results in

$$
\begin{equation*}
\ln \left(\frac{W_{\text {final }}}{F}\right)=\int_{x_{F}}^{x_{w, \text { final }}} \frac{d x_{w}}{x_{D}-x_{w}} \tag{7.6a}
\end{equation*}
$$

or

$$
\ln \left(\frac{W_{\text {final }}}{F}\right)=-\int_{x_{w, \text { final }}}^{x_{F}} \frac{d x_{w}}{x_{D}-x_{w}} \quad(7.6 \mathrm{~b})
$$

In order to perform an integration of the right hand side (RHS) of Eq. 7.6 (a \& b), $x_{D}$ must be a function of $x_{w}$ :

$$
x_{D}=y=f\left(x_{w}\right)
$$

For a simple batch distillation shown in Figure 7.1, it is reasonable to assume that the vapour that comes out of the top of the still pot (or the re-boiler) [note that the amount of the vapour is equal to that of the distillate] is in equilibrium with the liquid $(W)$ in the re-boiler

Thus, if the total condenser is used,

$$
y=x_{D}
$$

and $x_{D}$ and $x_{w}$ can be related to each other using an equilibrium curve or equilibrium equation

Accordingly, Eq. 7.6b can be re-written as follows

$$
\begin{equation*}
\ln \left(\frac{W_{\text {final }}}{F}\right)=-\int_{x_{w, \text { frial }}}^{x_{F}} \frac{d x}{y-x}=-\int_{x_{w, \text { final }}}^{x_{F}} \frac{d x}{f(x)-x} \tag{7.7}
\end{equation*}
$$

Note that $y=f(x)=x_{D}$ and $x=x_{w}$

The integration of the RHS of Eq. 7.7 can be done sequentially (ตามขั้นตอน) as follows

1) Plot an equilibrium curve
2) At each value of $x$ (from $x_{F}$ to $\left.x_{w, \text { final }}\right)$,
determine the value of $y$ (or $x_{D}$ ) from the equilibrium curve/equation
3) Plot $\frac{1}{y-x}$ (Y-axis) against $x$ (X-axis) or
fit it to an equation
4) Graphically determine the area under the curve from $x_{F}$ to $x_{w, \text { final }}$ or perform the integration analytically or numerically from $x_{F}$ to $x_{w, \text { final }}$; the graphical integration is as illustrated below


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

After the numerical value of the integration is obtained, the value of $W_{\text {final }}$ (i.e. the amount of liquid remained in the still pot) can be obtained from manipulating Eq. 7.7 as follows

$$
W_{\text {final }}=F \exp \left(-\int_{x_{u, \text { final }}}^{x_{F}} \frac{d x}{y-x}\right)
$$

(7.8a)
or

$$
\begin{array}{r}
W_{\text {final }}=F \exp (- \text { area under the curve }) \\
(7.8 \mathrm{~b})
\end{array}
$$

Finally, the value of the average distillate concentration, $x_{D, \text { avg }}$, and the total amount of the distillate, $D_{\text {total }}$, can be obtained by solving Eqs. 7.1 and 7.2:

$$
\begin{gather*}
F=W_{\text {final }}+D_{\text {total }}  \tag{7.1}\\
x_{F} F=x_{w, \text { final }} W_{\text {final }}+x_{D, \text { avg }} D_{\text {total }}
\end{gather*}
$$

(7.2)
simultaneously, which results in

$$
x_{D, a v g}=\frac{x_{F} F-x_{w, \text { final }} W_{\text {final }}}{F-W_{\text {final }}}
$$

(7.9)
and

$$
\begin{equation*}
D_{\text {total }}=F-W_{\text {final }} \tag{7.10}
\end{equation*}
$$

In the case that the equilibrium relationship
between $y\left(x_{D}\right)$ and $x\left(x_{w}\right)$ is given as

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

the RHS of Eq. 7.7 can be integrated analytically as follows

$$
\begin{aligned}
\ln \left(\frac{W_{\text {final }}}{F}\right)= & -\int_{x_{w, \text { final }}}^{x_{F}} \frac{d x}{y-x} \\
= & \frac{1}{(\alpha-1)} \ln \left[\frac{x_{w, \text { final }}\left(1-x_{F}\right)}{x_{F}\left(1-x_{w, \text { final }}\right)}\right] \\
& +\ln \left[\frac{\left(1-x_{F}\right)}{\left(1-x_{w, \text { final }}\right)}\right]
\end{aligned}
$$

(7.11)

For the problem that the value of $x_{D}$ is specified, and the value of $x_{w, \text { final }}$ is to be determined, a trial $\mathcal{\xi}$ error technique must be employed as follows

1) Make a first ( $\left.1^{\text {st }}\right)$ guess for the value of $x_{w, \text { final }}$ and calculate the value of the integration of Eq. 7.8a or determine the area
under the curve for Eq. 7.8b, according
to the guessed value of $x_{w, \text { final }}$
2) Then, the value of $W_{\text {final }}$ can be calculated from Eq. 7.8 (either a or b)
3) Use the value of $W_{\text {final }}$ obtained from 2 and the guessed value of $x_{w, \text { final }}$ made in 1 , combined with the given values of $F$ and $x_{F}$, to compute the values of $D_{\text {calc }}$ and $x_{D, \text { calc }}$ using the following equations:

$$
\begin{equation*}
D_{\text {calc }}=F-W_{\text {final }} \tag{7.12}
\end{equation*}
$$

and

$$
x_{D, \text { calc }}=\frac{x_{F} F-x_{w, \text { final }} W_{\text {final }}}{D_{\text {calc }}}
$$

(7.13)
4) Compare the value of $x_{D, \text { calc }}$ obtained from 3 with the given value of $x_{D}$ : if $x_{D, \text { calc }}=x_{D}$, the trial \& error procedure is finished; however, if $x_{D, \text { calc }} \neq x_{D}$, the new trial $\mathfrak{E}$ error has to be repeated, until we obtain the guessed value of $x_{w, \text { final }}$ that makes $x_{D, \text { calc }}=x_{D}$

The following Example illustrates the employment of the trial $\mathfrak{G}$ error technique to solve the batch distillation problem

Example Use the given equilibrium data of methanol ( MeOH ) and water for solving the simple batch distillation problem with the following description:

A single-equilibrium-stage (or a simple) batch still pot is used to separate MeOH from water The feed with the total amount of 50 moles of an $80 \mathrm{~mol} \% \mathrm{MeOH}$ is charged into the still pot operated at 1 atm

The desired distillate concentration $\left(x_{D}\right)$ is $89.2 \mathrm{~mol} \% \mathrm{MeOH}$

Determine:
a) the total amount of the distillate collected $\left(D_{\text {total }}\right)$
b) the amount of material (liquid or waste) remained in the pot after the distillation has ended $\left(W_{\text {final }}\right)$ and its corresponding concentration $\left(x_{w, \text { final }}\right)$

It is given that

- $F=50$ moles
- $x_{F}=0.80$
- $x_{D, \text { avg }}=0.892$

The equilibrium $(y-x)$ data of MeOH is as summarised in the following Table

Methanol liquid $\left(x_{\mathrm{MeOH}}\right) \quad$ Methanol vapour $\left(\boldsymbol{y}_{\mathrm{MeOH}}\right)$

## (mol\%)

0 (mol\%)

0 0
2.0
13.4
4.0
23.0
6.0
8.0
10.0
15.0
20.0
30.0
40.0
72.9
50.0
60.0
$70.0 \quad 87.0$
$80.0 \quad 91.5$
$90.0 \quad 95.8$
$95.0 \quad 97.9$
100.0
100.0

In this Example, the unknowns are

- $D_{\text {total }}$
- $W_{\text {final }}$
- $x_{w, \text { final }}$

Since $x_{w, \text { final }}$, one of the integral boundaries, is
NOT known, a trial $\mathcal{G}$ error technique must be employed to compute the integral $\int_{x_{w, \text { final }}}^{x_{F}} \frac{d x}{y-x}$

To start the calculations, the $1^{\text {st }}$ guess with $x_{w, \text { final }}$ of 0.70 is used

From the given equilibrium data, the value of 1 $y-x$
the following Table (note that interpolations are needed to obtain the values of $y$ and $\frac{1}{y-x}$ when the values of $x$ are, e.g., $0.75,0.65)$ :

| $\boldsymbol{x}$ | $\boldsymbol{y}$ | $\boldsymbol{y}-\boldsymbol{x}$ | $\frac{\boldsymbol{1}}{\boldsymbol{y}-\boldsymbol{x}}$ |
| :---: | :---: | :---: | :---: |
| 0.80 | 0.915 | 0.115 | 8.69 |
| 0.75 | 0.895 | 0.145 | 6.89 |
| 0.70 | 0.871 | 0.171 | 5.85 |
| 0.65 | 0.845 | 0.195 | 5.13 |
| 0.60 | 0.825 | 0.225 | 4.44 |
| 0.50 | 0.780 | 0.280 | 3.57 |

Plotting a graph between $x$ (X-axis) and $\frac{1}{y-x}$ (Y-axis) using the data in the Table on the previous Page, from $x_{w, \text { final }}=0.70$ (the dashed lines) to $x_{F}=0.80$, yields the following graph


## $x$

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

From the resulting graph, $\int_{0.70}^{0.80} \frac{d x}{y-x}$ is, in fact, the area under the curve from $x=0.70$ to $x=$ 0.80

For this Example, the area under the curve is found to be $\mathbf{0 . 7 0 4 4}$

The value of $W_{\text {final }}$ (i.e. the liquid remained in the still pot) can then be calculated, using Eq. 7.8b, as follows

$$
\begin{gathered}
W_{\text {final }}=F \exp (- \text { area under the curve }) \\
=(50) \exp (-0.7044) \\
\\
W_{\text {final }}=24.72 \mathrm{~mol}
\end{gathered}
$$

Thus, the total amount of the distillate can
be computed using from Eq. 7.12 as follows

$$
\begin{aligned}
D_{\text {calc }} & =F-W_{\text {final }} \\
& =50-24.72
\end{aligned}
$$

$$
D_{\text {calc }}=25.28
$$

The value of $x_{D, \text { calc }}$ can be calculated using Eq.
7.13 as follows

$$
\begin{gathered}
x_{D, \text { calc }}=\frac{x_{F} F-x_{w, \text { final }} W_{\text {final }}}{D_{\text {calc }}} \\
=\frac{[(0.80)(50)]-[(0.70)(24.72)]}{(25.28)} \\
x_{D, \text { calc }}=0.898
\end{gathered}
$$

However, the desired value of $x_{D}$ or $x_{D, \text { avg }}$ is
0.892 - the calculated $\boldsymbol{x}_{\boldsymbol{D}}$ value is too high!

Thus, a new guess of $x_{w, \text { final }}$ is needed

With the new guess of $x_{w, \text { final }}$ of 0.60 , we obtain the following (by performing similar calculations as above):

- area under the curve from $x_{w \text {,inal }}=0.60$ to

$$
x_{F}=0.80 \text { is } 1.2084
$$

- $W_{\text {final }}=50 \exp (-1.2084)=14.93$
- $D_{\text {calc }}=F-W_{\text {final }}=50-14.93=35.07$
- $x_{D, \text { calc }}=0.885$ (too low!)

Hence, we need to make a new (the $3^{\text {rd }}$ ) guess for $x_{w, \text { final }}$

With the $3^{\text {rd }}$ guess of $x_{w, \text { final }}$ of 0.65 , we obtain the following (try doing the detailed calculations yourself):

- area under the curve (from $x=0.65$ to

$$
x=0.80)=0.9710
$$

- $W_{\text {final }}=18.94$
- $D_{\text {calc }}=31.06$
- $x_{D, \text { calc }}=0.891$ (O.K. - close enough!)


### 7.2 Constant-level Batch Distillation

The recent Example is the simple batch distillation problem in which the amount of liquid in the still pot is decreasing as the distillation proceeds (while the distillate is being collected)

In a constant-level batch distillation, which is another configuration of a batch distillation, a solvent (or the feed) is added to the re-boiler (or the still pot) to keep the level of the liquid in the pot constant

Note that, during the addition of the solvent, the total number of moles of all species in the still pot is kept constant

The total mole balance is

$$
[\mathrm{In}]-[\mathrm{Out}]=\left[\begin{array}{l}
\text { Accumulation }  \tag{7.14}\\
\text { in the still pot }
\end{array}\right]
$$

Since, in this kind of batch distillation, the total number of moles is constant, Eq. 7.14 becomes

$$
\begin{equation*}
[\mathrm{In}]-[\mathrm{Out}]=0 \tag{7.15}
\end{equation*}
$$

Also, since this is a constant-level batch distillation, the amount of solvent evaporated $(-d V)$ must be equal to the amount of solvent added to the still pot $(+d S)$ - note that the solvent added into the still pot is called "the second solvent"

$$
\begin{aligned}
-d V & =d S \\
(\text { or } d V & =-d S)
\end{aligned}
$$

Performing a species (or component) balance on the evaporated solvent (called "the original solvent") gives

$$
\begin{aligned}
-y d V & =-d\left(W x_{w}\right) \\
y d V & =d\left(W x_{w}\right)
\end{aligned}
$$

[note that, at any instant of time, the concentration of the vapour evaporated from the liquid $(y)$ is constant; thus, it is drawn from the differentiation)

$$
\begin{equation*}
y d V=W d x_{w}+x_{w} d W \tag{7.17}
\end{equation*}
$$

but $W$ is kept constant

Thus, Eq. 7.17 becomes

$$
\begin{equation*}
y d V=W d x_{w} \tag{7.18}
\end{equation*}
$$

Substituting Eq. 7.16 into Eq. 7.18 yields

$$
\begin{equation*}
-y d S=W d x_{w} \tag{7.19}
\end{equation*}
$$

Re-arranging Eq. 7.19 and integrating the resulting equation gives

$$
\begin{gather*}
-y d S=W d x_{w} \\
\frac{d S}{W}=-\frac{d x_{w}}{y} \\
\int \frac{d S}{W}=-\int_{x_{w, \text { initial }}}^{x_{w, \text { final }}} \frac{d x_{w}}{y} \\
\frac{S}{W}=\int_{x_{w, \text { final }}}^{x_{w, \text { initial }}} \frac{d x_{w}}{y} \tag{7.20}
\end{gather*}
$$

In this kind of batch distillation, the vapour phase and the liquid phase in the system (i.e. the still pot) are assumed to be in equilibrium with each other

Hence, the value of $y$ (the concentration of the vapour phase) can be related to the value of $x_{w}$ (the concentration of the liquid phase in the still pot) using either equilibrium curve or equation

If the relationship between $y$ and $x_{w}$ can be expressed in the following form:

$$
\begin{equation*}
y=\frac{\alpha x_{w}}{1+(\alpha-1) x_{w}} \tag{7.21}
\end{equation*}
$$

the integral of the RHS of Eq. 7.20 is

$$
\frac{S}{W}=\frac{1}{\alpha} \ln \left[\frac{x_{w, \text { initial }}}{x_{w, \text { final }}}\right]+\frac{\alpha-1}{\alpha}\left(x_{w, \text { initial }}-x_{w, \text { final }}\right)
$$

(7.22)

Alternatively, the graphical solution (for the value of $\frac{S}{W}$ ) to this kind of problem can be obtained as follows

For each value of $x_{w}$, the value of $y$ can be read from the equilibrium curve, and the graph between $x_{w}\left(\mathrm{X}\right.$-axis) and $\frac{1}{y}$ (Y-axis) is plotted

The area under the curve from $x_{w, \text { final }}$ to
$x_{w, \text { intitial }}$ is, in fact, $\frac{S}{W}$

Generally, the value of $W$ is given; thus, Eq.
7.20 (or 7.22 ) is normally used to compute the value of $S$ (i.e. the amount of solvent required to keep the liquid level in the still pot constant)

### 7.3 Batch Steam Distillation

In the batch steam distillation, steam is purged directly into the still pot, as shown in Figure 7.3


Figure 7.3: A batch steam distillation
(from "Separation Process Engineering" by Wankat, 2007)

Normally, the direct addition of steam into the still pot is done for the system that is NOT miscible (ละลาย) with water

Like the steam distillation in the continuous operation, the principal purpose of adding steam directly into the still pot is to keep the temperature of the system below the boiling point of water, while eliminating the need of a heat transfer device (as steam can provide heat/ energy to the system)

By solving Eqs 7.1:

$$
\begin{equation*}
F=W_{\text {final }}+D_{\text {total }} \tag{7.1}
\end{equation*}
$$

and 7.2 :

$$
\begin{equation*}
x_{F} F=x_{w, \text { final }} W_{\text {final }}+x_{D, \text { avg }} D_{\text {total }} \tag{7.2}
\end{equation*}
$$

simultaneously, we obtain the following equation:

$$
W_{\text {final }}=F\left(\frac{x_{D}-x_{F}}{x_{D}-x_{w, \text { final }}}\right)
$$

(7.23)

Since the more volatile component (e.g., the volatile organic components: VOCs) is much more volatile than water, its concentration (i.e. $x_{D}$ ) in the vapour phase can be considered pure (i.e. the distillate contains only the VOCs, or $x_{D}$ for the VOCs is 1.0 )

Thus, Eq. 7.23 becomes

$$
\begin{equation*}
W_{\text {final }}=F\left(\frac{1-x_{F}}{1-x_{w, \text { final }}}\right) \tag{7.24}
\end{equation*}
$$

The amount of the distillate, $D$, can, thus, be calculated using the following equation:

$$
\begin{equation*}
D=F-W_{\text {final }} \tag{7.25}
\end{equation*}
$$

The amount of water in the separating tank $\left(n_{w}\right)$ (see Figure 7.3), which is, in fact, the amount of steam condensed at the condenser, can be computed using the following equation:

$$
n_{w}=\int_{0}^{D} \frac{P_{\text {total }}-x_{\mathrm{VOC} \text { in the remaining liquid }} P_{\mathrm{VOC}}^{*}}{x_{\mathrm{VOC} \text { in the remaining liquid }} P_{\mathrm{VOC}}^{*}} d n_{\text {org }}
$$

(7.26)

The total amount of steam required is $n_{w}$ plus with the amount of steam that is condensed and remained in the still pot (note that this steam is used to heat up the feed and vaporise the VOCs)

$$
\begin{equation*}
\left(n_{\text {steam }}\right)_{\text {total }}=n_{w}+\left(n_{\text {steam }}\right)_{\text {used to heat up the feed }} \tag{7.27}
\end{equation*}
$$

### 7.4 Multi-stage Batch Distillation

If very high purity of a product (either distillate or bottom) is needed, a multi-stage column is added to the batch distillation system, as illustrated in Figure 7.2 (Page 7)

The complexity with the multi-stage equilibrium is attributed to the fact that $x_{D}$ and $x_{w}$ are no longer in equilibrium with each other as per the case of the simple batch distillation

## In other words, the relationship between $x_{D}$

 and $x_{w}$ can no longer be expressed using the equilibrium curve or equationAccordingly, the Rayleigh equation (i.e. Eq.
7.6a or 7.6 b ), cannot be integrated until the relationship between $x_{D}$ and $x_{w}$ is established

Generally, this relationship between $x_{D}$ and $x_{w}$ for the multi-stage distillation can be obtained by performing stage-by-stage calculations

The following is how to formulate the relationship between $x_{D}$ and $x_{w}$ for the multi-stage distillation system

Performing material and energy balances for Figure 7.2 from stage $j$ to the top of the column, with the assumption that the accumulation at any where except the re-boiler is negligible, yields

$$
\begin{gather*}
V_{j+1}=L_{j}+D  \tag{7.28}\\
y_{j+1} V_{j+1}=x_{j} L_{j}+x_{D} D  \tag{7.29}\\
Q_{C}+V_{j+1} H_{j+1}=L_{j} h_{j}+D h_{D}
\end{gather*}
$$

In order to simplify the calculations, CMO is assumed, and the operating equation of the multistage batch distillation can be written, by rearranging Eq. 7.29, as follows

$$
\begin{equation*}
y_{j+1}=\frac{L}{V} x_{j}+\left(1-\frac{L}{V}\right) x_{D} \tag{7.31}
\end{equation*}
$$

However, the difficulty of using Eq. 7.31 in the batch distillation is that either $x_{D}$ or $\frac{L}{V}$ is varying during the operation, thus resulting in the fact that the operating line will continuously be changing (or it is not constant) throughout the operation

Normally, the batch distillation operation can be divided into 2 modes:

- Constant reflux ratio $\left(\frac{L}{D}\right)$
- Constant distillate concentration $\left(x_{D}\right)$

The following is the details of each mode

### 7.4.1 Constant reflux ratio

One of the most common multi-stage batch distillation modes is the operation in which the reflux ratio $\left(\frac{\boldsymbol{L}}{\boldsymbol{D}}\right)$ is kept constant throughout the distillation

In this kind of operation, the concentration of the distillate $\left(x_{D}\right)$ is varied (changed), while the values of $L$ and $V$ are kept constant (by fixing the reflux ratio)

Accordingly, we obtain the operating lines
with the same slope (i.e. the same $\frac{L}{V}$ ) but various Y-intercepts; note that the points where $y=x=$ $x_{D}$ are also varied

In other words, in this kind of distillation operation (i.e. batch distillation), there are several operating lines, which is in contrast to the case of continuous distillation, in which there is only one operating line

After an appropriate number of operating lines are plotted for each value of $x_{D}$, we step off stages (for a given number of equilibrium stages) to find the value of $x_{w}$ for each $x_{D}$

Once $x_{w}$ value for each value of $x_{D}$ is obtained, we can perform the integration for the Rayleigh equation (Eq. 7.6), and the values of

- $W_{\text {final }}$
- $D_{\text {total }}$
- $x_{D, \text { avg }}$
will subsequently be obtained

Note, once again, that if the value of $x_{D, \text { avg }}$ is specified, the trial $\mathfrak{G}$ error technique is to be employed to calculate the value of $x_{w, \text { final }}$

Let's examine the following Example, which illustrates how to solve the constant reflux ratio distillation problem

Example The $50-\mathrm{mol}$ feed comprising $32 \% \mathrm{EtOH}$ and $68 \%$ water is to be distilled in the multi-stage batch distillation with the additional 2 equilibrium stages on top of the re-boiler (still pot)

Reflux is returned to the column as a saturated liquid with the constant reflux ratio $\left(\frac{L}{D}\right)$ of $2 / 3$

It is desired that the solvent remained in the still pot has the concentration of EtOH of 4.5 mol\%

Determine the average distillate composition $\left(x_{D, \text { avg }}\right)$, the final amount (in moles) of liquid in the still pot $\left(W_{\text {final }}\right)$, and the total amount of the distillate collected $\left(D_{\text {total }}\right)$

The operation is at 1 atm

The schematic diagram of the batch distillation in this Example is as shown below

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

Since the operation is at 1 atm , the equilibrium curve of EtOH can be obtained from the $x-y$ equilibrium data of the EtOH -water mixture at 1 atm

A value of $x_{D}$ is selected (specified), and the corresponding value of $x_{w}$ for each selected value of $x_{D}$ (or for each operating line) for the number of stages of $\mathbf{3}$ (why " 3 "? ) is obtained, as shown in the following McCabe-Thiele diagram

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

From each pair of $x_{D}$ and $x_{w}$, the value of $\underline{1}$ is calculated
$x_{D}-x_{w}$

Eventually, a graph between $\frac{1}{x_{D}-x_{w}}$ and $x_{w}$ is plotted from $x_{w, \text { final }}=0.045(4.5 \%)$ to $x_{F}=$ $0.32(32 \%)$, as shown on the next Page, and the area under the curve is found to be $\mathbf{0 . 6 0 8}$

Hence,

$$
\begin{gathered}
W_{\text {final }}=F \exp (- \text { area under the curve }) \\
=(50) \exp (-0.608) \\
W_{\text {final }}=27.21
\end{gathered}
$$

and

$$
D_{\text {total }}=F-W_{\text {final }}=50-27.21=22.79
$$


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

The value of $x_{D, \text { avg }}$ can, thus, be calculated as follows

$$
\begin{aligned}
& x_{D, \mathrm{avg}}= \frac{x_{F} F-x_{w, \text { final }} W_{\text {final }}}{D_{\text {total }}} \\
&= \frac{[(0.32)(50)]-[(0.045)(27.21)]}{(22.79)} \\
& x_{D, \mathrm{avg}}=0.648
\end{aligned}
$$

### 7.4.2 Variable reflux ratio (Constant $x_{D}$ )

In this batch distillation mode, the distillation is carried out such that the value of $x_{D}$ is fixed, while the reflux ratio $\left(\frac{L}{D}\right)$ is varied

As the reflux ratio is varied, the slope of the operating line keeps changing as illustrated in Figure 7.4

Note that the point where the operating line intersects with the $y=x$ line at $x_{D}$ is fixed (as the value of $x_{D}$ is kept constant)

Similar to the constant reflux ratio batch distillation (but not exactly the same), to solve this
problem, we step off stages (for a given number of equilibrium stages) from the point where $y=x$ $=x_{D}$ for each operating line, and the corresponding value of $x_{w}$ is obtained; then, the graph between $\frac{1}{x_{D}-x_{w}}$ and $x_{w}$ is plotted


## Figure 7.4: The batch distillation with varying

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

### 7.5 Operating Time for Batch Distillation

The overall operating time for batch distillation includes the operating time and the down time:

$$
\begin{equation*}
t_{\text {batch }}=t_{\text {operating }}+t_{\text {down }} \tag{7.32}
\end{equation*}
$$

The actual operating time for batch distillation $\left(t_{\text {operating }}\right)$ can be computed using the following equation:

$$
\begin{equation*}
t_{\text {operating }}=\frac{D_{\text {total }}}{\dot{D}} \tag{7.33}
\end{equation*}
$$

where $\dot{D}=$ the flow rate of the distillate

The value of $\dot{D}$ cannot be set arbitrarily (อย่างไรก็ได้ or ตามใจชอบ), as the column must be
designed to accommodate a limited amount of vapour flow rate $\left(V_{\max }\right)$, to avoid the flooding phenomenon

The maximum distillate flow rate $\left(D_{\max }\right)$ can be computed as follows

$$
\begin{equation*}
\dot{D}_{\max }=\frac{V_{\max }}{1+\frac{L}{D}} \tag{7.34}
\end{equation*}
$$

Normally, the optimal or operating value of $\dot{D}$ is $0.75 \dot{D}$
max

The down time $\left(t_{\text {down }}\right)$ includes

1) the time required for dumping (draining out) the remaining bottom product
2) the clean-up time (to clean up the column)
3) the loading time for the next batch
4) the heat-up time until the reflux starts to appear

Note that ehe energy requirements for a condenser and a re-boiler can be calculated from the energy balance equations around the condenser and the whole system, respectively

If the reflux is a saturated liquid,

$$
\begin{equation*}
Q_{C}=-V_{1}\left(H_{1}-h_{D}\right)=-V_{1} \lambda \tag{7.35}
\end{equation*}
$$

where $\lambda$ is the latent heat of vaporisation

The energy balance around the entire system yields the heating load $\left(Q_{R}\right)$ as follows

$$
\begin{equation*}
Q_{R}=-Q_{C}+D h_{D} \tag{7.36}
\end{equation*}
$$

