Stagewise Operations
Fall 2010

Introduction to Column Distillation

Instructor: Dr. Housam Binous
KFUPM, Dhahran
Over 90% of separations are done using distillation

Over 40,000 distillation columns operate in the USA alone
Distillation Cascade

Flash distillation is simple but produces a limited amount of separation.

One can achieve more separation by cascading more flash separators.
Send vapor & liquid streams to additional flash chambers with increasing and decreasing pressures, respectively.
$V_1$ and $L_5$ have high and low concentrations of the more volatile component, respectively

**What to do with all other streams?**

**Solution:** use intermediate streams as additional feeds within the same cascade

For example $V_4$ is used to feed stage 3 after compression to higher pressure
Only two product streams obtained in high yield and high purity
Isothermal distillation requires large number of compressors

Instead one can operate at constant pressure and force temperature to vary. We can use heat exchangers to heat or cool intermediate streams
Use reflux and boilup streams to control liquid and vapor flow rates in the column.
All heat required for distillation is applied to the bottom reboiler

All required cooling is done in the top condenser
Intermediate heat exchange can be achieved efficiently with direct liquid-vapor contact on each stage ⇒ results in cheaper and simpler device
Enriching section is section above feed stage

Stripping section is section below feed stage

Light components are stripped out by the rising vapor from the liquid phase

If the relative volatility is close to unity or if there is an azeotrope, little separation will take place
If thermo-sensitive components are present, one has to use vacuum or steam distillation

\[ y_i V > L x_i \quad \text{or} \quad K_i \frac{V}{L} > 1 \]

Component \( i \) tends to exit in the distillate
Distillation Equipment

Columns are built in metal and have circular cross-sections.

Trays are built so that liquid-vapor contact occurs.

Sieve trays are sheets of metal with holes punched into them to allow vapor to pass through.
Liquid flow down from tray above in a downcomer

Liquid is contacted with vapor as it flows across the sieve tray

Rising vapor prevents liquid from dripping downward

Metal weir allows a sufficient liquid level on each stage

Frothy mixture flows over the weir and the vapor disengages in the downcomer
Space above plate designed so that it avoids excessive entrainment

- Bubble regime
- Foam regime
- Froth regime
- Spray regime

Decreasing vapor flow rates

Increasing vapor flow rates
Bubble regime

Low gas flow rates

Liquid pool with rising gas bubbles

Poor mixing $\iff$ Low efficiency

Regime undesirable in commercial applications
Foam regime

Higher gas flow rates

Liquid phase is continuous with rapidly rising distinct gas bubbles

There is a distinct foam, which has a large surface area

↓

Large efficiency

Foam can fill entire space between stages leading to excessive entrainment and column may flood

Regime at gas flow rates too low for most industrial applications
Froth regime

At even higher gas flow rates

Liquid is continuous with large pulsating voids of vapor rapidly passing through

Violent boiling at liquid surface and considerable splashing

Thoroughly mixed liquid phase $\implies$ good efficiency if mass transfer is controlled by liquid phase resistance

Usual regime in commercial applications
Spray regime

At even higher gas flow rates

Vapor phase is continuous

Liquid occurs as discontinuous spray of droplets

Vapor is very well mixed but liquid droplets are not

↓

Low efficiency

Regime undesirable in commercial applications

One should be cautious sine he can go from froth to spray regimes only because of a small increase in the gas flow rate
Figure 6.1  Flow patterns on trays. (a) Froth regime (liquid phase is continuous); (b) spray regime (gas phase is continuous). (Henry Z. Kister, excerpted by special permission from Chemical Engineering, September 8, 1980; copyright ©, by McGraw-Hill, Inc., New York, NY 10020.)
Other popular equipments

Valve trays & bubble-cap trays

Downcomers can be circular pipes

Partial condensers

Multiple feeds

Side-stream withdrawals

Measurement (e.g., flow meters) and control devices
Column with bubble-cap trays
Usual specified variables for binary distillation

1/ column pressure

2/ feed flow rate

3/ feed composition

4/ feed temperature or enthalpy or quality

5/ reflux temperature or enthalpy
(usually reflux is saturated liquid)

4 additional variables must be set
Two types of problems are considered

**Design problems**
- Desired separation is set
- Column is designed in order to achieve this separation

**Simulation problems**
- Column is already built
- Predict how much separation can be achieved
Design problems

For binary distillation:

- **Specify** mole fraction of light component both in distillate and bottom

- **Specify** external reflux ratio

- **Specify** that feed location is optimum

- **Compute** $D, B, Q_R, Q_C, N$, optimum feed plate location and column diameter
<table>
<thead>
<tr>
<th>Specified Variables</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. 1. Mole fraction more volatile component in distillate, ( x_B )</td>
</tr>
<tr>
<td>2. Mole fraction more volatile component in bottoms, ( x_B )</td>
</tr>
<tr>
<td>3. External reflux ratio, ( L_v/D )</td>
</tr>
<tr>
<td>4. Use optimum feed plate</td>
</tr>
<tr>
<td>B. 1.2. Fractional recoveries of components in distillate and bottoms, ( (FR_A)<em>{dist}, (FR_B)</em>{bott} )</td>
</tr>
<tr>
<td>3. External reflux ratio, ( L_v/D )</td>
</tr>
<tr>
<td>4. Use optimum feed plate</td>
</tr>
<tr>
<td>C. 1. D or B</td>
</tr>
<tr>
<td>2. ( x_D ) or ( x_B )</td>
</tr>
<tr>
<td>3. External reflux ratio, ( L_v/D )</td>
</tr>
<tr>
<td>4. Use optimum feed plate</td>
</tr>
<tr>
<td>D. 1.2. ( x_D ) and ( x_B )</td>
</tr>
<tr>
<td>3. Boilup ratio, ( V/B )</td>
</tr>
<tr>
<td>4. Use optimum feed plate</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Designer Calculates</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Distillate and bottoms flow rates, D and B</td>
</tr>
<tr>
<td>Heating and cooling loads, ( Q_R ) and ( Q_c )</td>
</tr>
<tr>
<td>Number of stages, ( N )</td>
</tr>
<tr>
<td>Optimum feed plate</td>
</tr>
<tr>
<td>Column diameter</td>
</tr>
<tr>
<td>B. ( x_B, x_D, D, B )</td>
</tr>
<tr>
<td>( Q_R, Q_c )</td>
</tr>
<tr>
<td>( N )</td>
</tr>
<tr>
<td>( N_{feed} )</td>
</tr>
<tr>
<td>Column diameter</td>
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<tr>
<td>C. B or D</td>
</tr>
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<td>D. D and B. ( Q_R ) and ( Q_c )</td>
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<tr>
<td>( N, N_{feed} )</td>
</tr>
<tr>
<td>Column diameter</td>
</tr>
</tbody>
</table>
A. 1. Mole fraction more volatile component in distillate, $x_D$
2. Mole fraction more volatile component in bottoms, $x_B$
3. External reflux ratio, $L_0/D$
4. Use optimum feed plate

A. Distillate and bottoms flow rates, $D$ and $B$
Heating and cooling loads, $Q_R$ and $Q_c$
Number of stages, $N$
Optimum feed plate
Column diameter
Simulation problems

Given are feed flow rate and composition, feed location, number of stages, column diameter and reboiler size, which controls $V_{max}$

Specify mole fractions of light component in both product streams

Compute external reflux ratio and check that $V < V_{max}$
<table>
<thead>
<tr>
<th>Specified Variables</th>
<th>Designer Calculates</th>
</tr>
</thead>
</table>
| A. 1, 2. N, N_{feed}  
3.4. x_D and x_B  
Column diameter | A. \( \frac{L_d}{D} \)  
B. D, \( Q_c \), \( Q_R \)  
Check \( V < V_{max} \) |
| B. 1, 2. N, N_{feed}  
3.4. \( \frac{L_0}{D} \), x_D (or x_B)  
Column diameter | B. x_B (or x_D)  
B. D, \( Q_c \), \( Q_R \)  
Check \( V < V_{max} \) |
| C. 1, 2. N, N_{feed}  
3. x_D (or x_B)  
4. Column diameter  
(set \( V = \text{fraction} \times V_{max} \)) | C. \( \frac{L_0}{D} \), x_B (or x_D)  
B. D, \( Q_c \), \( Q_R \)  
D. B, D, \( Q_c \), x_B (or x_D), \( \frac{L_0}{D} \)  
Check \( V < V_{max} \) |
| D. 1, 2. N, N_{feed}  
3. \( Q_R \)  
4. x_D (or x_B)  
Column diameter | |

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External Column Balances

We will derive mass and energy balances around entire column in order to compute: $D$, $B$, $Q_R$ and $Q_C$

For binary systems, one can solve these equations without doing stage-by-stage calculations.
Column is adiabatic and operates at constant pressure

Design problem: solve for $D$ and $B$

\[
F = D + B
\]
\[
F_Z = B x_B + D x_D
\]
Write energy balance neglecting potential and kinetic energies as well as work terms

\[ F h_F + Q_C + Q_R = D h_D + B h_B \]

\[ Q_C < 0 \quad \text{and} \quad Q_R > 0 \]
\[ Q_C = - \left( 1 + \frac{L_0}{D} \right) \left( \frac{Z - x_B}{x_D - x_B} \right) F \lambda \]

\[ Q_R = Dh_D + Bh_B - Fh_F + \left( 1 + \frac{L_0}{D} \right) D \lambda \]
CHE 306
Stagewise Operations
Fall 2010

Column Distillation: Internal Stage-by-Stage Balances

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For binary systems, one needs to determine $N$ by performing stage-by-stage balances

1/ Start at the top
2/ Write balances and equilibrium relationships for first stage
3/ Determine unknown variables for first stage
4/ Write balances and equilibrium relationships for second stage utilizing variables that were just computed
5/ Proceed down the column in a stage-by-stage fashion until you reach the bottom
Figure 4-1. Enriching section balance envelopes; (A) stage 1, (B) stage 2, (C) stage j
Balance equations

\[ V_2 = L_1 + D \]
\[ V_2 y_2 = L_1 x_1 + Dx_D \]
\[ V_2 H_2 + Q_C = L_1 h_1 + Dh_D \]

Equilibrium relationships

\[ h_1 = h_1(x_1) \]
\[ H_2 = H_2(y_2) \]
\[ x_1 = x_1(y_1) \]
6 unknowns 6 equations

Determine: $L_1, V_2, x_1, y_2, H_2, h_1$
Proceed to second stage:

Balance equations

\[
V_3 = L_2 + D
\]
\[
V_3 y_3 = L_2 x_2 + D x_D
\]
\[
V_3 H_3 + Q_c = L_2 h_2 + D h_D
\]

Equilibrium relationships

\[
h_2 = h_2(x_2)
\]
\[
H_3 = H_3(y_3)
\]
\[
x_2 = x_2(y_2)
\]
6 unknowns

6 equations

Determine: \( L_2, V_3, x_2, y_3, H_3, h_2 \)
For a general stage above feed stage

\[ V_{j+1} = L_j + D \]

\[ V_{j+1} y_{j+1} = L_j x_j + D x_D \]

\[ V_{j+1} H_{j+1} + Q_C = L_j h_j + D h_D \]

\[ h_j = h_j(x_j) \]

\[ H_{j+1} = H_{j+1}(y_{j+1}) \]

\[ x_j = x_j(y_j) \]
Determine: $L_j$, $V_{j+1}$, $x_j$, $y_{j+1}$, $H_{j+1}$, $h_j$
Figure 4-2. Stripping section balance envelopes: (A) below feed stage (stage $f+1$), (B) stage $k$. (C) partial reboiler.
Stage immediately below feed plate: \( \text{stage } f+1 \)

**Balance equations**

- \( \overline{V}_{f+1} = \overline{L}_f - B \)
- \( \overline{V}_{f+1} y_{f+1} = \overline{L}_f x_f - B x_B \)
- \( \overline{V}_{f+1} H_{f+1} = \overline{L}_f h_f - B h_B + Q_R \)

**Equilibrium relationships**

- \( h_f = h_f(x_f) \)
- \( H_{f+1} = H_{f+1}(y_{f+1}) \)
- \( x_f = x_f(y_f) \)
6 unknowns

6 equations

Determine: \( L_f, V_{f+1}, x_f, y_{f+1}, H_{f+1}, h_f \)

\( x_B \) specified

\( B \) and \( Q_R \) are determined from column balances
For a general stage below feed stage: stage \( k \)

### Balance equations

\[
\begin{align*}
\bar{V}_k &= \bar{L}_{k-1} - B \\
\bar{V}_k y_k &= \bar{L}_{k-1} x_{k-1} - B x_B \\
\bar{V}_k H_k &= \bar{L}_{k-1} h_{k-1} - B h_B + Q_B
\end{align*}
\]

### Equilibrium relationships

\[
\begin{align*}
h_{k-1} &= h_{k-1}(x_{k-1}) \\
H_k &= H_k(y_k) \\
x_{k-1} &= x_{k-1}(y_{k-1})
\end{align*}
\]
A partial reboiler acts as an equilibrium stage

Problem is finished when:

\[ x_{N+1} < x_B \]
Binary stage-by-stage solution methods

Solve 6 equations (3 balance equations + 3 equilibrium relationships) simultaneously

\[ \downarrow \]

- Sorel (1893): trial and error
- Ponchon (1921) & Savarit (1922): graphical resolution
Lewis (1922)

Molar vapor & liquid flow rates are constant in each column section

\[
\begin{align*}
L_1 &= L_2 = \cdots = L_{f-1} = L \\
V_2 &= V_3 = \cdots = V_f = V \\
\overline{L}_f &= \overline{L}_{f+1} = \cdots = \overline{L}_N = \overline{L} \\
\overline{V}_{f+1} &= \overline{V}_{f+2} = \cdots = \overline{V}_{N+1} = \overline{V}
\end{align*}
\]

When 1 mole of vapor is condensed then 1 mole of liquid is vaporized
CMO assumption = Constant Molal Overflow

Column is adiabatic

Specific heat changes << latent heat changes

\[ |H_{j+1} - H_j| \ll \lambda \quad \text{and} \quad |h_{j+1} - h_j| \ll \lambda \]

Latent heat of vaporization, \( \lambda \), is independent of composition
$H(y)$

$h(x)$

$\lambda = \text{constant}$
CMO assumption

\[
H_{j+1} \approx H_j \quad \text{and} \quad h_{j+1} \approx h_j
\]

energy balance automatically satisfied
Lewis method: CMO valid

Operating line in the enriching section

Relationship between compositions of 2 passing streams

\[ y_{j+1} = \frac{L}{V} x_j + \left( 1 - \frac{L}{V} \right) x_D \]
Column balance $\Rightarrow$ B and D $\Rightarrow$ L and V

$L = RD$ and $V = L + D$

$y_1$ known $\Rightarrow$ x$_1$ known $\Rightarrow$ y$_2$ known

Equilibrium relationship $\Rightarrow$ Operating line

Proceed until the feed stage
If slope of this operating line is known then one can alternate between equilibrium relationship and operating line of the stripping section.
Feed quality

\[ q = \frac{\bar{L} - L}{F} \sim \frac{H - h_F}{H - h} \]

Feed can be saturated vapor (q=0), saturated liquid (q=1) or a two-phase mixture (1>q>0)

\[ \bar{L} = L + qF \quad \text{and} \quad \bar{V} = V - (1 - q)F \]
McCabe & Thiele Method (1925)

Graphical method based on Lewis’s method and the fact that operating lines are straight lines on the y-x diagram.

Solve the equilibrium relationship from the y-x equilibrium curve and the mass balance from the operating lines.
Top operating line

Straight line with $L/V$ as slope and $(1-L/V) \times D$ as $y$-intercept

$$y = \frac{L}{V} x + \left(1 - \frac{L}{V}\right) \times D$$
Top of the column = total condenser

\[ y_1 = x_D = x_0 \]

Equilibrium curve

Operating line
\[(1 - L/V)x_D\]

**Top operating line**

Slope = \(L/V\)
Knowing $y_2$ one can proceed down the column

We step off stages

We can continue as long as we are in the rectifying section
The above procedure is called the McCabe & Thiele method.

It produces a staircase construction.

In the stripping section, one has to use a different operating line.

\[ y = \left( \frac{L}{V} \right) x - \left( \frac{L}{V} - 1 \right) x_B \]

Applies to passing streams in the stripping section.
Start with liquid leaving the reboiler $x_B = x_{N+1}$

Use equilibrium curve

\[ y_{N+1} \]

Use operating line

\[ x_N \]

We can continue this alternating process between equilibrium curve and operating line as long as we are in the stripping section.
Figure 4-7. Stepping off stages in stripping section
At feed stage one has to switch between top operating line and bottom operating line
How to compute slope of bottom operating line

\[ \bar{L} = L + qF \quad \text{and} \quad \bar{V} = V - (1 - q)F \]

\[ L = RD \quad \text{and} \quad V = L + D \]

Obtain \( D \) and \( B \) from mass balances around entire column
Useful for computer calculations

\[
\frac{\bar{L}}{\bar{V}} = \frac{\frac{L}{D} (z - x_B) + q (x_D - x_B)}{\frac{L}{D} (z - x_B) + q (x_D - x_B) - (x_D - z)}
\]
Feed line

\[ y = \frac{q}{q-1} x + \frac{1}{1-q} Z_F \]

\[ y = \frac{f-1}{f} x + \frac{1}{f} Z_F \]
Determine feed quality

\[ q = \frac{\bar{L} - L}{F} \sim \frac{H - h_F}{H - h} \]

Get slope of feed line

\[ \frac{q}{q - 1} \]

Feed line goes through point \((x=z, y=z)\)
<table>
<thead>
<tr>
<th>Type feed</th>
<th>T</th>
<th>hF</th>
<th>q</th>
<th>f</th>
<th>Slope</th>
</tr>
</thead>
<tbody>
<tr>
<td>Subcooled liquid</td>
<td>TF&lt;TBP</td>
<td>hF&lt;h</td>
<td>q&gt;1</td>
<td>f&lt;0</td>
<td>&gt;1.0</td>
</tr>
<tr>
<td>Saturated liquid</td>
<td>TF=TBP</td>
<td>h</td>
<td>1</td>
<td>0</td>
<td>Infinite</td>
</tr>
<tr>
<td>Biphasic feed</td>
<td>TDP&gt;TF&gt;TBP</td>
<td>H&gt;hF&gt;h</td>
<td>1&gt;q&gt;0</td>
<td>0&lt;f&lt;1</td>
<td>Negative</td>
</tr>
<tr>
<td>Saturated vapor</td>
<td>TF=TDP</td>
<td>H</td>
<td>0</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>Superheated vapor</td>
<td>TF&gt;TDP</td>
<td>hF&gt;H</td>
<td>q&lt;0</td>
<td>f&gt;1</td>
<td>0&lt;slope&lt;1</td>
</tr>
</tbody>
</table>
Figure 4-11. Operating line intersection: (A) changing reflux ratio with constant $q$; (B) changing $q$ with fixed reflux ratio
Intersection of two operating lines \((x_I, y_I)\)

\[
x_I = \frac{-(q - 1) \left(1 - \frac{L}{V}\right) x_D - z_F}{(q - 1) \frac{L}{V} - q}
\]

\[
y_I = \frac{z_F + \frac{x_D q}{L/D}}{1 + q \frac{L}{V}}
\]
Profiles for binary distillation

![Graph showing profiles for binary distillation](image-url)

**Figure 4-14. Profiles for Example 4-3**
Composition profile is obtained from fig 4-13
To obtain temperature at each stage: use Txy diagram and known x-values
CMO assumption: flow rates will be constant in each section

Value of \( q \) (feed quality) determine changes in flow rates at feed stage