Distillation Column
Distillation:

“Process in which a liquid or vapour mixture of two or more substances is separated into its component fractions of desired purity, by the application and removal of heat”
The choice between use of tray column or a packed column for a given mass transfer operation should, theoretically, be based on a detailed cost analysis for the two types of contactors. However, the decision can be made on the basis of a qualitative analysis of relative advantages and disadvantages, eliminating the need for a detailed cost comparison.

Which are as follows
- Liquid dispersion difficulties
- Capable of handling wide ranges liquid rates
- Cleaning
- Non-foaming systems
- Periodic cleaning
- Weight of the column
- Design information
- Inter stage cooling
- Temperature change
- Diameters
As my system is **non foaming** and **diameter** calculated is larger than 0.67 m so I am going to use **Tray column**. Also as **average temperature** calculated for my distillation column is higher that is approximately equal to **98°C**. So I prefer **Tray column**.
PLATE CONTACTORS:

Cross flow plate are the most commonly used plate contactor in distillation. In which liquid flows downward and vapours flow upward. The liquid move from plate to plate via down comer. A certain level of liquid is maintained on the plates by weir.
Three basic types of cross flow trays used are:

- Sieve Plate (Perforated Plate)
- Bubble Cap Plates
- Valve plates (floating cap plates)
Selection of Trays:

1. **Bubble-caps, Valves or Sieves...**

   - **Bubble-cap tray**
   - **Valve tray**
   - **Sieve tray**

<table>
<thead>
<tr>
<th></th>
<th>Bubble-caps</th>
<th>Valves</th>
<th>Sieves</th>
</tr>
</thead>
<tbody>
<tr>
<td>Relative cost</td>
<td>2.0</td>
<td>1.2</td>
<td>1.0</td>
</tr>
<tr>
<td>Pressure drop</td>
<td>Highest</td>
<td>Intermediate</td>
<td>Lowest</td>
</tr>
<tr>
<td>Efficiency</td>
<td>Highest</td>
<td>Highest</td>
<td>Lowest</td>
</tr>
<tr>
<td>Vapor capacity</td>
<td>Lowest</td>
<td>Highest</td>
<td>Highest</td>
</tr>
<tr>
<td>Typical turndown ratio</td>
<td>5</td>
<td>4</td>
<td>2</td>
</tr>
</tbody>
</table>
I prefer **Sieve Plate** because:

- **Pressure drop** is low as compared to bubble cap trays.
- Their **fundamentals** are well established, entailing low risk.
- The trays are **low in cost** relative to many other types of trays.
- They can easily handle **wide variations in flow rates**.
- They are **lighter in weight**. It is easier and cheaper to **install**.
- **Maintenance cost** is reduced due to the ease of cleaning.
Sieve Tray
Label Diagram (sieve tray)

- Downcomer and Weir
- Major Beam
- Plate Support Ring
- Calming Zone
- Man Way
FACTORS AFFECTING DISTILLATION COLUMN OPERATION

Adverse vapour flow conditions can cause:

- Blowing
- Coning
- Dumping
- Raining
- Weeping
- Flooding
FLOW SHEET

Condenser

REFLUX DRUM

Pump

Reboiler

(1) Methyl Iodide = 0.07
(2) Acetic Acid = 0.65
(3) Methyl Acetate = 0.22
(4) Water = 0.065

(1) Acetic Acid = 0.99
(2) Water = 0.01
From Material Balance:

<table>
<thead>
<tr>
<th>Component</th>
<th>Feed Fraction $x_f$</th>
<th>Bottom Fraction $x_b$</th>
<th>Top Fraction $x_d$</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1) Methyl Iodide</td>
<td>0.07</td>
<td>0</td>
<td>0.21</td>
</tr>
<tr>
<td>(2) Acetic Acid</td>
<td>0.65</td>
<td>0.99</td>
<td>0.0005</td>
</tr>
<tr>
<td>(3) Methyl Acetate</td>
<td>0.22</td>
<td>0</td>
<td>0.62</td>
</tr>
<tr>
<td>(4) Water</td>
<td>0.07</td>
<td>0.01</td>
<td>0.17</td>
</tr>
</tbody>
</table>

- **Heavy Key Component = Acetic Acid**
- **Light Key Component = Water**
DESIGNING STEPS OF DISTILLATION COLUMN

- Calculation of Minimum number of stages $N_{\text{min}}$
- Calculation of Minimum Reflux Ratio $R_m$.
- Calculation of Actual Reflux Ratio.
- Calculation of theoretical number of stages.
- Calculation of actual number of stages.
- Calculation of diameter of the column.
- Calculation of weeping point, entrainment.
- Calculation of pressure drop
- Calculation of the height of the column.
Calculation of Minimum no. of Plates:

The minimum no. of stages $N_{\text{min}}$ is obtained from Fenske equation which is,

$$N_{\text{min}} = \frac{\ln\left[(x_{\text{LK}}/x_{\text{HK}})_D(x_{\text{HK}}/x_{\text{LK}})_B\right]}{\ln\left(\alpha_{\text{LK}/\text{HK}}\right)_{\text{average}}}$$

Average geometric relative volatility = 1.53

So,

$$N_{\text{min}} = 24$$
Calculation of Minimum Reflux Ratio $R_m$:

Using Underwood equations

\[
\frac{\alpha_A x_{DA}}{\alpha_A - \theta} + \frac{\alpha_B x_{DB}}{\alpha_B - \theta} = R_m + 1
\]

As feed is entering as saturated vapors so,

\[q = 0\]

By trial, \[\theta = 1.68\]

Using equation of minimum reflux ratio,

\[
\frac{\alpha_A x_{fA}}{\alpha_A - \theta} + \frac{\alpha_B x_{fB}}{\alpha_B - \theta} = 1 - q
\]

Putting all values we get,

\[R_m = 4.154\]
Actual Reflux Ratio:

The rule of thumb is:

$$R = (1.2 \text{ ------} 1.5) \ R_{\text{min}}$$
$$R = 1.5 \ R_{\text{min}}$$
$$R = 6.23$$
Gilliland related the number of equilibrium stages and the minimum reflux ratio and the no. of equilibrium stages with a plot that was transformed by Eduljee into the relation:

\[
\frac{N - N_{\text{min}}}{N+1} = 0.75 \left[ 1 - \left( \frac{R - R_{\text{min}}}{R+1} \right)^{0.566} \right]
\]

From which the theoretical no. of stages to be,

\[ N = 39 \]
Calculation of actual number of stages:

Overall Tray Efficiency:

\[ E_o = 51 - 32.5 \log \left( \mu_{avg} \cdot \alpha_{avg} \right) \]

\( \alpha_{avg} \) = average relative volatility of light key component

\( \mu_{avg} \) = molar average liquid viscosity of feed evaluated at average temperature of column

\( \mu_{avg} \) = 1.75
Average temperature of column = \((118+71)/2\)
= 95 °C

Feed viscosity at average temperature = \(\mu_{avg}\)
= 0.39 mNs/m²

So,

\(E_o = 56.60\%\)

So,

No. of actual trays = \(39/0.566 = 68\)
Location of feed Plate:

The Kirk bride method is used to determine the ratio of trays above and below the feed point.

\[
\log\left(\frac{N_D}{N_B}\right) = 0.206 \log \left[ \frac{B}{D} \left( \frac{x_{HK}}{x_{LK}} \right) \left( \frac{(x_{LK})_B}{(x_{HK})_D} \right)^2 \right]
\]

From which,
Number of Plates above the feed tray = \( ND = 47 \)
Number of Plates below the feed tray = \( NB = 21 \)
Determination of the Column Diameter:

Flow Parameter:

\[ F_{LV} = \left( \frac{L}{V} \right)_n \left( \frac{\rho_v}{\rho_L} \right)^{0.5} \]

\[ F_{LV} = \text{Liquid Vapor Factor} = 0.056 \]
Capacity Parameter:

Assumed tray spacing = 18 inch (0.5 m)
From Fig (15-5) Plant Design and Economics for Chemical Engineering, sieve tray flooding capacity,

\[ C_{sb} = 0.0760 \text{ m/Sec} \]

Surface tension of Mixture = \( \sigma = 18.35 \text{ dynes/Cm} \)

\[ V_{nf} = C_{sb} \left( \frac{\sigma}{20} \right)^{0.2} \left( \frac{\rho l - \rho v}{\rho v} \right)^{0.5} \]

\[ V_{nf} = 1.67 \text{ m/sec} \]

Assume 90% of flooding then

\[ V_n = 0.9V_{nf} \]

So, actual vapor velocity,

\[ V_n = 1.51 \text{ m/sec} \]
Net column area used in separation is

\[ A_n = \frac{m_v}{V_n} \]

Volumetric flow rate of vapors = \( m_v \)

\[ m_v = \frac{\text{mass vapor flow rate}}{(3600) \text{ vapor density}} \]

\[ m_v = 2.1184m^3/\text{sec} \]

Now, net area \( A_n = \frac{m_v}{V_n} = 1.41m^2 \)

Assume that downcommer occupies 15% of cross sectional Area \( (A_c) \) of column thus:

\[ A_c = A_n + A_d \]

Where, \( A_d = \text{downcommer area} \)
$$A_c = A_n + 0.15(A_c)$$
$$A_c = A_n / 0.85$$
$$A_c = 1.65 \text{ m}^2$$

So Diameter of Column Is
$$A_c = (\pi/4)D^2$$
$$D = (4A_c/\pi)$$
$$D = 1.45 \text{ meter} = 5\text{ft}$$
(based upon bottom conditions)
Liquid flow arrangement:

In order to find liquid flow arrangement first find maximum liquid volumetric flow rate

So liquid flow rate =

(Liquid mass rate)/ (3600) (Liquid density)

Max Liquid Rate Is At the bottom of column so using "L_m" values

So Maximum liquid flow rate = 0.005 m³/sec

So from fig11.28 Coulson & Richardson 6th volume 3rd edition cross flow single pass plate is selected
Provisional Plate Design:

Column Diameter $D_c = 1.4513$ m
Column Cross-sectional Area ($A_c$) = 1.65 m$^2$
Down comer area $A_d = 0.15A_c = 0.25$ m$^2$
Net Area ($A_n$) = $A_c - A_d = 1.41$ m$^2$
Active area $A_a = A_c - 2A_d = 1.16$ m$^2$
Hole area $A_h$, take 10% $A_a = 0.1 \times 1.16$
  
  $= 0.0462$ m$^2$

Weir length
$A_d / A_c = 0.248 / 1.654 = 0.15$
From figure 11.31 Coulson & Richardson 6th volume 3rd edition

\[
\frac{L_w}{d_c} = 0.80 \\
L_w = 1.452 \times 0.80 \\
= 0.733 \text{ m}
\]

Weir length should be 60 to 85% of column diameter which is satisfactory.

Take weir height, \( h_w = 50 \text{ mm} \)

Hole diameter, \( d_h = 5 \text{ mm} \)

Plate thickness = 5 mm
Check Weeping:

\[ \tilde{U}_{\text{min}} = \frac{K_2 - 0.9(25.4 - d_h)}{(\rho \nu)^{1/2}} \]

where \( U_{\text{min}} \) is the minimum design vapor velocity.

The vapor velocity at weeping point is the minimum velocity for the stable operation.

In order to have \( K_2 \) value from fig11.30 Coulson & Richardson 6th volume 3rd edition we have to 1st find \( h_{ow} \) (depth of the crest of liquid over the weir)

where \( h_{ow} \) is calculated by following formula:
\[ h_{ow} = 750 \left[ \left( \frac{L_m}{l_w} \cdot \rho \right)^{2/3} \right] \]

Maximum liquid rate “\( L_m \)“ = 4.7 kg/sec
Minimum Liquid Rate At 70% turn down ratio = 3.3 Kg/sec

At Maximum rate (\( h_{ow} \)) = 20 mm Liquid
At Minimum rate (\( h_{ow} \)) = 16 mm Liquid
\[ h_w + h_{ow} = 50 + 16 = 66 \text{ mm Liquid} \]

from fig 11.30, Coulson and Richardson Vol.6
\[ K_2 = 30.50 \]
So,
\[ U_{(\text{min})} = 9 \text{ m/sec} \]
Now maximum volumetric flow rate (vapors)

Base = 2.12 m$^3$/sec
Top = 1.14 m$^3$/sec

At 70% turn down ratio

Actual minimum vapor velocity

$= \frac{\text{minimum vapor rate}}{A_h}$

$= 12.81$ m/sec

So minimum vapor rate will be well above the weep point.
Plate Pressure Drop (P.D):

Consist of **dry plate P.D** (orifice loss), **P.D due to static head of liquid** and **residual P.D** (bubbles formation result in energy loss)

**Dry Plate Drop:**
Max. Vapor velocity through holes \((U_h) = \) Maximum Volumetric Flow Rate / Hole Area \(= 18.30 \text{ m/sec}\)
Perforated area \(A_p\) (active area) \(= 1.16 \text{ m}^2\)

\[
Ah/Ap = 0.100
\]
From fig. 11.34 (Coulson & Richardson 6th volume 3rd edition) for plate thickness/hole diameter = 1.00
We get, \( C_0 = 0.84 \)

\[
h_d = 51 \left[ \frac{\hat{U}_h}{C_0} \right]^2 \frac{\rho_v}{\rho_L}
\]

This equation is derived for orifice meter pressure drop.

\( h_d = 48 \text{ mm Liquid} \)

**Residual Head (h_r):**

\[
hr = \left( 12.5 \times 10^3 / \rho_L \right)
\]

\( = 13.3 \text{ mm Liquid} \)
So,

\[ h_t = h_d + (h_w + h_{ow}) + h_r \]

Total pressure drop
\[ = 48 + (50 + 20) + 13.32 \]
\[ h_t = 131.35 \text{ mm liquid} \]

Total column pressure drop \( \text{Pa, (N/m}^2) \)
\[ = (9.81 \times 10^{-3}) h_t \rho_L N \]
\[ = 82771.6 \text{ Pa} = 82 \text{ kPa} \]
Down comer Liquid Backup: 
Caused by Pressure Drop over the plate and resistance to flow in the downcomer it self.

\[ h_b = (h_w + h_{ow}) + h_t + h_{dc} \]

The main resistance to flow in downcomer will be caused by constriction in the downcomer outlet, and head loss in the down comer can be estimated using the equation given as,

\[ h_{dc} = 166 \left( \frac{l_{wd}}{\rho LA_{ap}} \right)^2 \]

where \( L_{wd} \) is the liquid flow rate in downcomer, kg/sec and \( A_{ap} \) is the clearance area under the downcomer, m\(^2\)

\[ A_{ap} = h_{ap} L_w \]
Where \( h_{ap} \) the height of bottom edge of apron above the plate.

\[
h_{ap} = h_w - (5 \text{ to } 10 \text{ mm})
\]

\[
h_{ap} = 40 \text{ mm}
\]

so,

Area under apron “\( A_{ap} \)” = 0.05 m\(^2\)

As this is less than area of downcomer \( A_d \) so using \( A_{ap} \) values in above formula.

So,

\[
h_{dc} = 1.95 \text{ mm}
\]
As a result,

\[ h_b = 203.24 \text{ mm} \]
\[ = 0.203 \text{ m} \]

\[ h_b < \frac{1}{2} \text{ (Tray spacing + weir height)} \]

0.20 < 0.25

So tray spacing is acceptable
Check Residence Time:

Sufficient residence time should be allowed in the downcomer for the entrained vapors to disengage from liquid stream to prevent aerated liquid being carried under the downcomer.

\[ t_r = A_d \ h_{bc} \ \rho_L / L_{(max)} \]

\[ t_r = 10 \text{ sec} \]

It should be > 3 sec. so, result is satisfactory.
Check Entrainment:

\[(u_n) \text{ actual velocity} = \left( \frac{\text{maximum volumetric flow rate at base } V_m}{\text{net area } A_n} \right)\]

\[(u_n) \text{ actual velocity} = 1.51 \text{ m/sec}\]

Velocity at flooding condition \( U_f = 1.67 \text{ m/sec}\)

So Percent flooding \( = u_n / u_f = 0.90 = 90\% \)
Liquid flow factor $F_{LV} = 0.056$

From fig. 11.29 Coulson & Richardson 6th volume 3rd edition fractional entrainment $\psi$ can be found out.

**Fractional entrainment ($\psi$) = 0.0750**

Well below the upper limit of ($\psi$) which is 0.1. Below this the effect of entrainment on efficiency is small.
Area of 1 Hole = \( \frac{\pi}{4} D_{\text{hole}}^2 \)
\[ = 0.00002 \text{ m}^2 \]

Area of \( N \) Holes = 0.1158 m\(^2\)

So,

Number OF Holes = 5900
Height of Distillation Column

Height of column $H_c = (N_{act} - 1) H_s + \Delta H + \text{plates thickness}$

No. of plates = 68
Tray spacing $H_s = 0.50 \text{ m}$
$\Delta H = 0.5 \text{ meter each for liquid hold up and vapor disengagement}$
$\Delta H = 1 \text{ m}$
Total thickness of trays = $0.005 \times 68 = 0.34 \text{ m}$
So,

Height of column = $(68-1) \times 0.50 + 1 + 0.34 = 35 \text{ meters}$
Plate Specifications

- Hole diameter = 5mm
- No. of holes = 5900
- $h_{ow}$ = Weir crust
- $h_{ap}$ = 40 mm
- $h_{w}$ = 50 mm
- Height = 35m
- Length = 1.45m
## Specification Sheet Of Distillation Column:

### Identification:
- **Item**: Distillation column
- **No. required**: 1
- **Tray type**: Sieve tray

### Function:
- Separation of **Acetic Acid** from **iodo methane** and Reaction **by products**.

### Operation:
- **Continuous**
**Material handled:**

<table>
<thead>
<tr>
<th></th>
<th>Feed</th>
<th>Top</th>
<th>Bottom</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Amount</strong></td>
<td>4755 Kg/hr</td>
<td>1968 Kg/hr</td>
<td>2786 Kg/hr</td>
</tr>
<tr>
<td><strong>Composition of Acetic Acid</strong></td>
<td>0.64</td>
<td>0.005</td>
<td>0.99</td>
</tr>
<tr>
<td><strong>Temp.</strong></td>
<td>119°C</td>
<td>71°C</td>
<td>118°C</td>
</tr>
</tbody>
</table>
## Design data:

<table>
<thead>
<tr>
<th></th>
<th>No. of tray = 68</th>
<th>Active holes = 5900</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure = 101.325 Kpa</td>
<td>Weir height = 50 mm</td>
<td></td>
</tr>
<tr>
<td>Height of column = 35 m</td>
<td>Weir length = 1 m</td>
<td></td>
</tr>
<tr>
<td>Diameter of column = 1.45 m</td>
<td>Reflux ratio = 6.23</td>
<td></td>
</tr>
<tr>
<td>Hole size = 5 mm</td>
<td>Tray spacing = 0.5 m</td>
<td></td>
</tr>
<tr>
<td>Pressure drop per tray = 1.2 Kpa</td>
<td>Active area = 1.16 m²</td>
<td></td>
</tr>
<tr>
<td>Tray thickness = 5 mm</td>
<td>Percent Flooding = 90%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Entrainment = 0.075</td>
<td></td>
</tr>
</tbody>
</table>
References

- Coulson & Richardson 6th volume 3rd edition
- Plant Design and Economics for Chemical Engineering
- Coulson & Richardson 2th volume 5th edition
- Perry’s Chemical engineer’s hand book
The End