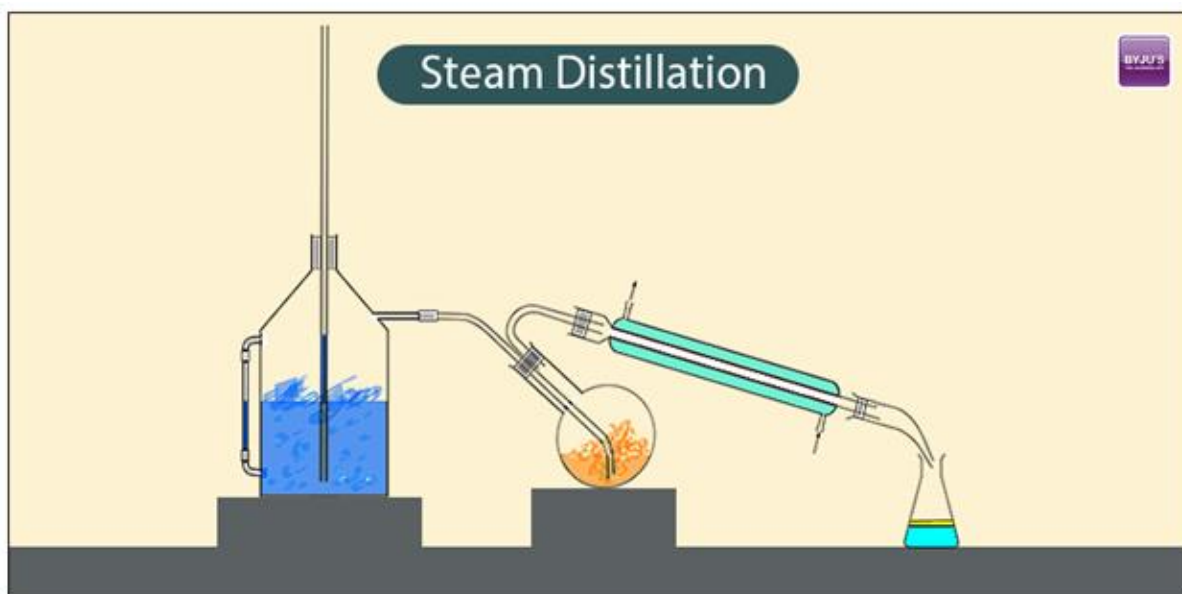


## Chapter 2 –Distillation

### 2.14 Steam distillation and its application

**Steam distillation** is used to separate temperature-sensitive organic compounds such as aromatic substances.



At a very high sustained temperature, few of **organic compounds** may decompose. It would be difficult to separate by a distillation process at a certain boiling point. Steam will be introduced into an apparatus ( distillation).

Condensed liquid phase separates that are vaporized a water vapor carries compounds and they are placed in a condensation flask that is placed near by. Now distillation takes place at a lower temperature. Steam distillation can be applied in case if the substance is very sensitive to heat. Vapours are condensed after distillation process.

#### **Applications**

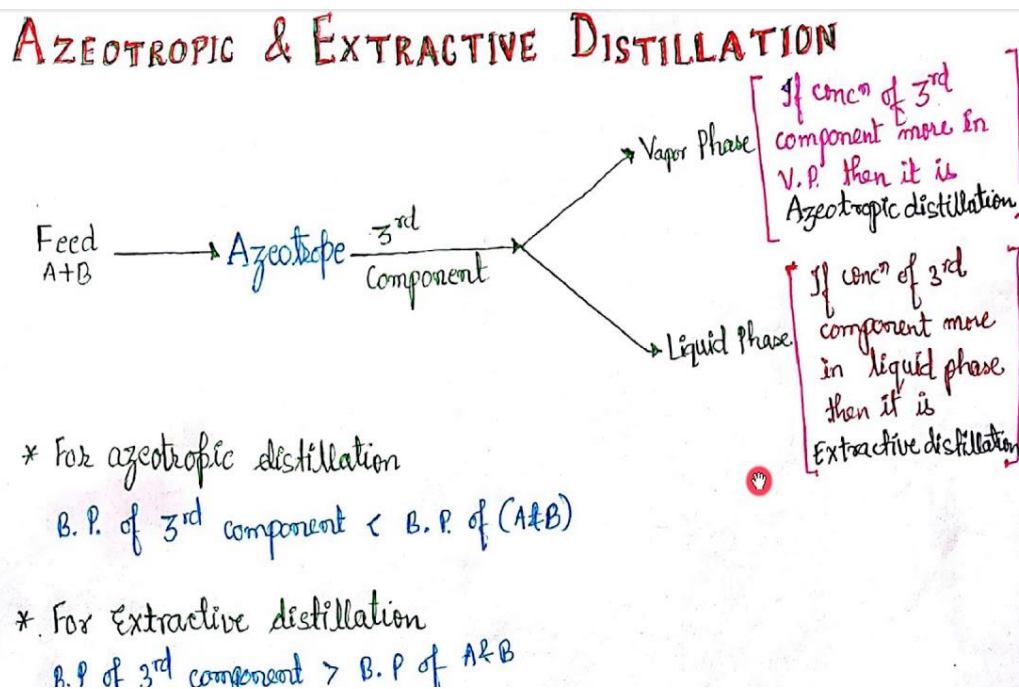
- Steam distillation are widely used in the manufacturing of essential oils, for instance perfumes.
- This method uses a plant material that consists of essential oils. Mainly orange oil is extracted on a large scale in industries using this method.

- Application of steam distillation can be found in the production of consumer food products and petroleum industries. They are used in separation of fatty acids from mixtures.
- It can be used to extract oils from natural products, such as eucalyptus oil, citrus oils, or other natural substances derived from organic matter.

## 2.15 Azeotropic and extractive distillation

**Azeotropic distillation (AD)** is a process to break **azeotrope** where another volatile component, called the entrainer, the solvent, or the mass separating agent (MSA), is added to form a new lower-boiling **azeotrope** that is **HETEROGENEOUS** (diverse in character or content).

In the same way extractive distillation also works. Find the mechanism below.



**What is difference between azeotropic distillation and extractive distillation?**

1. In azeotropic distillation (AC) the third component added is referred to as entrainer while in extractive distillation (EC) the third component added is referred to as solvent.
2. Product obtained from AC is from the bottom of the column while in EC product is obtained from the top of the column.
3. Third component added in AC forms a low boiling azeotrope while in EC the third component added has low volatility (high boiling point).

## **Chapter 3- ABSORPTION**

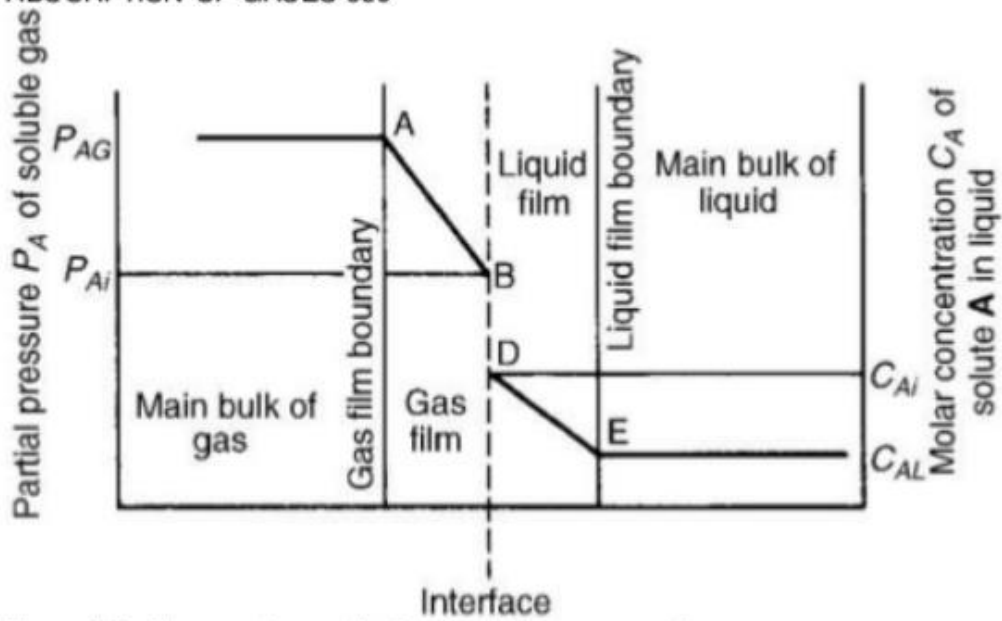
### **CHAPTER – III ABSORPTION**

#### **3.1 Principles of absorption and factors affecting rates of absorption**

**Absorption** : Absorption, in chemical technology, is a process in which atoms or molecules transfer from a gas phase into a liquid phase

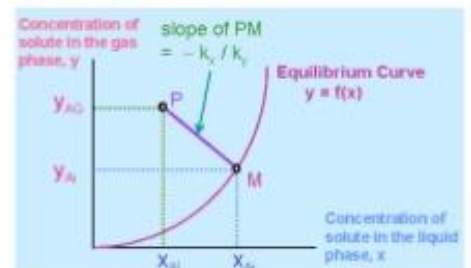
Theory of Absorption:

#### **-Two film Theory**



## Two-Film Theory and Equilibrium Solubility Curve

- The concentrations at the interface:
  - in the gas (  $y_{Ai}$  )
  - in the liquid (  $x_{Ai}$  )
- are represented as a point M on the equilibrium solubility curve.
- Point M thus has the coordinates (  $y_{Ai}$ ,  $x_{Ai}$  ).
- As we move along the column along the continuous interface, we can trace out an equilibrium curve.
- The “best case scenario” will be equilibrium
  - From P to M



### Type of Absorption

**1. Chemical Absorption :** Chemical absorption or reactive **absorption** involves a **chemical** reaction between the substance being **absorbed** and the **absorbing** medium

## 2. Physical Absorption: No chemical reaction involved.

# Chemical vs. Physical Absorption

Chemical	Physical
The capacity or solubility shows in practice limited sensitivity to the partial pressure of the gas to be removed	The capacity or solubility is sensitive to the partial pressure of the gas to be removed
High heat of absorption	Low heat of absorption
Desorption achieved mainly by temperature increase and in high pressure cases, also by pressure reduction (flashing)	Desorption achieved mainly by pressure reduction (flashing), and sometimes with additional heating of the solvent
Potential to reduce level of acid gas to a very low level	Extent of acid gas removal limited

### FACTORS AFFECTING ABSORPTION::

#### ➤ Operating Pressure ::

Raising the pressure may increase the separation effectiveness considerably.

#### ➤ Mass Transfer Coefficient of Liquid Side:

More the MTC, More the absorption

#### ➤ Temperature and Humidity of the Rich Gas ::

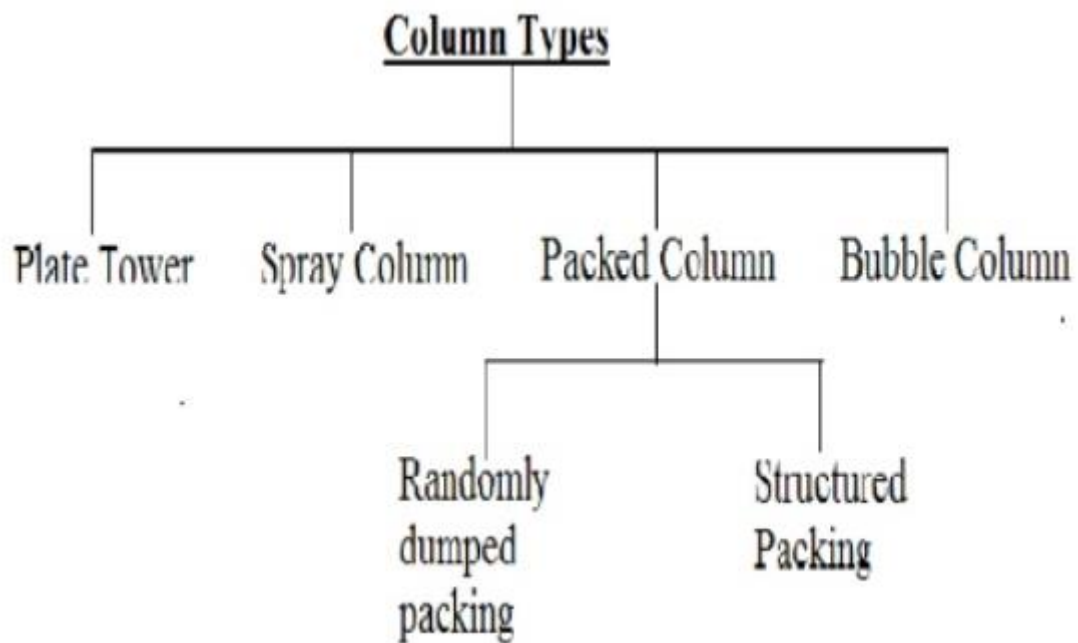
Cooling and consequent dehumidification of the feed gas to an absorption tower can be very beneficial. A high humidity (or relative saturation with the solvent) limits the capacity of the gas to take up latent heat and hence is unfavorable to absorption. Thus dehumidification of the inlet gas is worth considering in the design of absorbers with large heat effects.

#### ➤ Liquid-to-Gas Ratio (L/G Ratio) :

More gas flow rate leads to more absorption rate.

### 3.2 Different equipments used for absorption

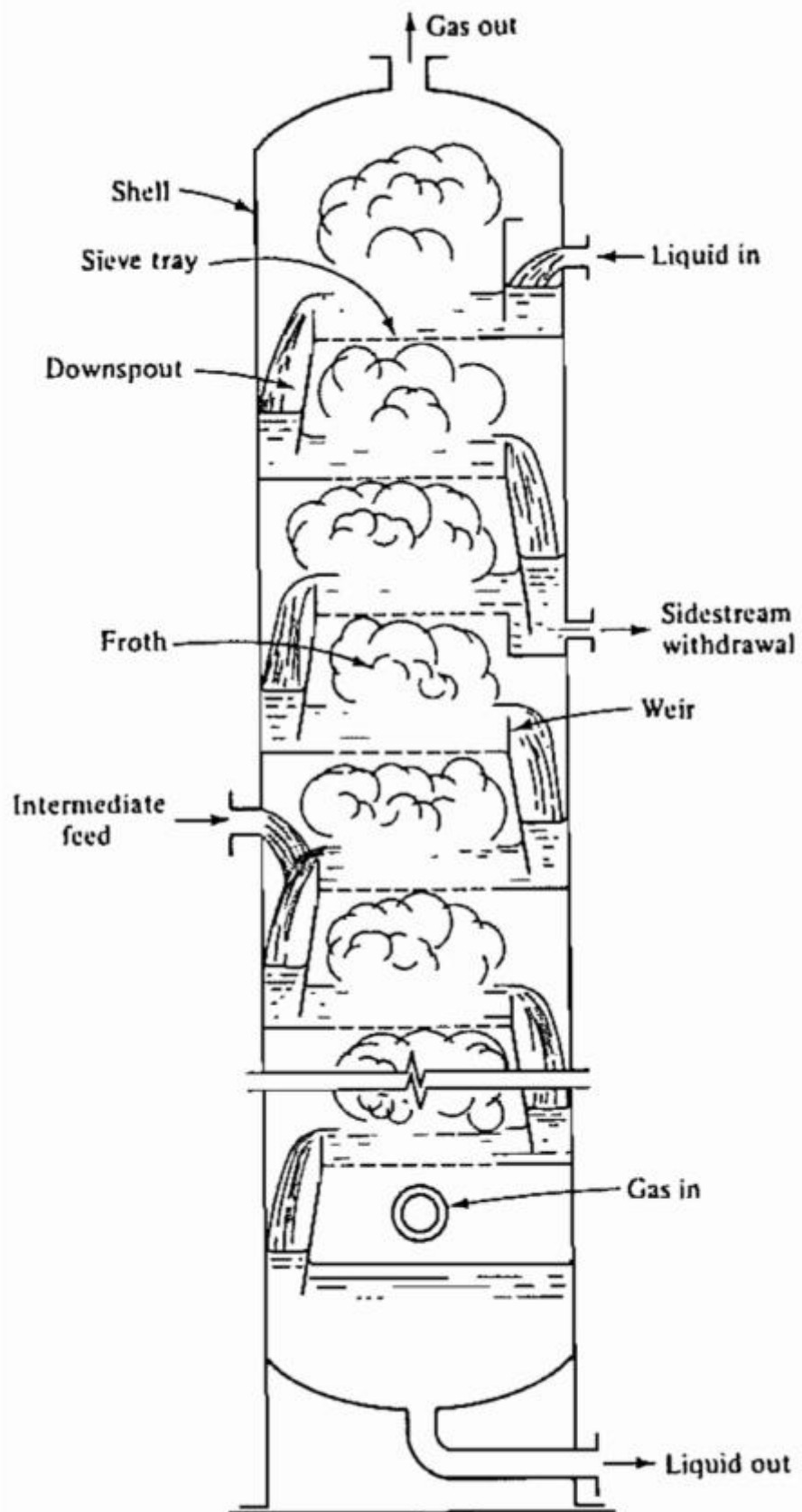
Absorption Tower:



*ASSIGNMENT for the above diagrams given to students.*

## Equipments Used in Absorption process.

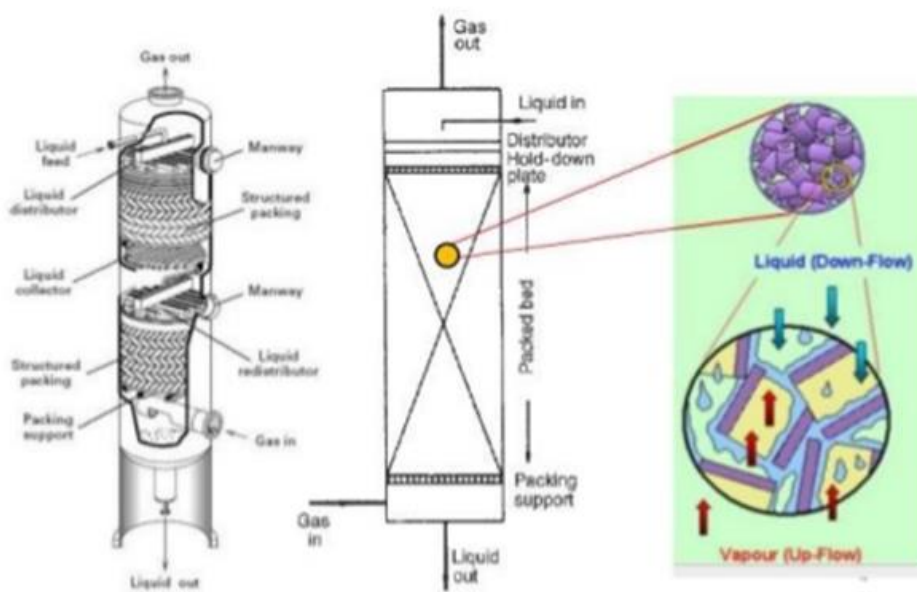
### 1. Tray Tower



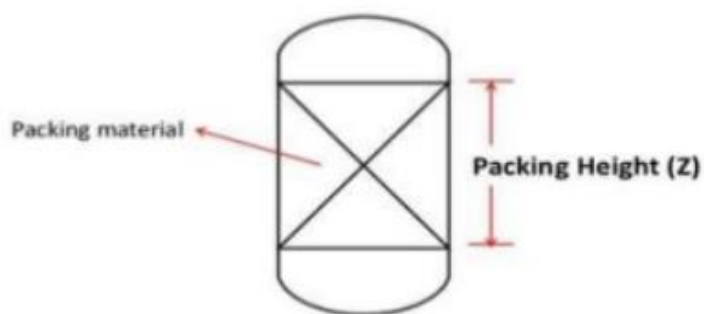
## 2. Packed Tower

Component of a Packed bed/Tower

### ***Component of a packed column:***

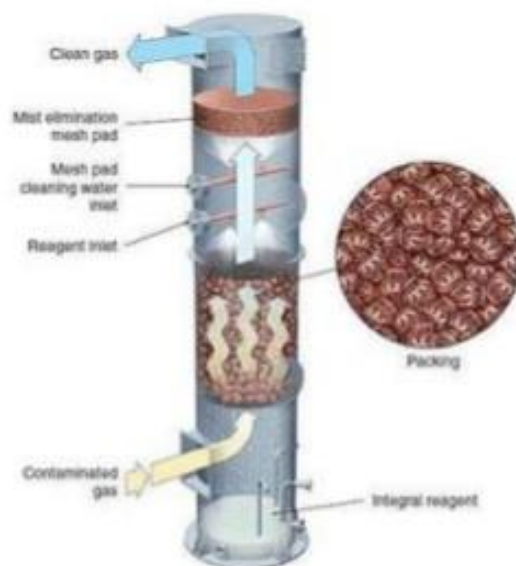
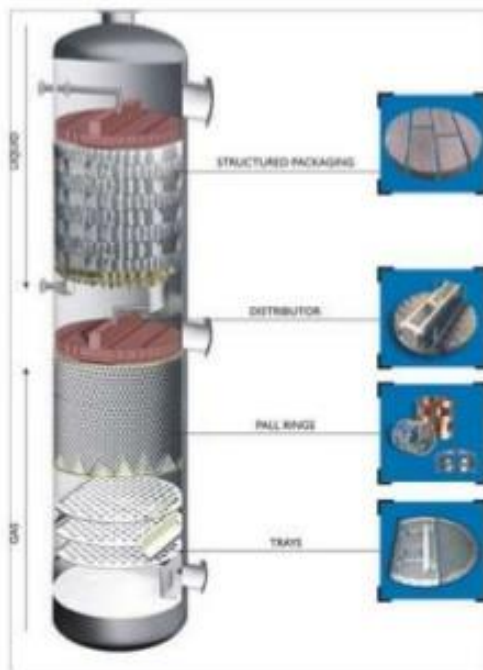


### ***Representation of packed column:***





## Cross sectional/internal view of a packed absorption column

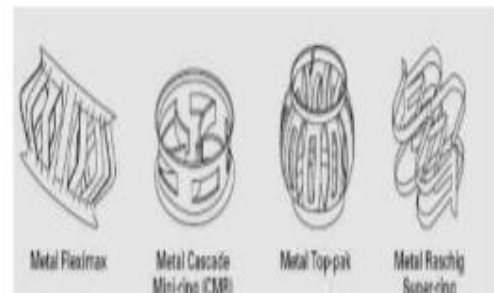
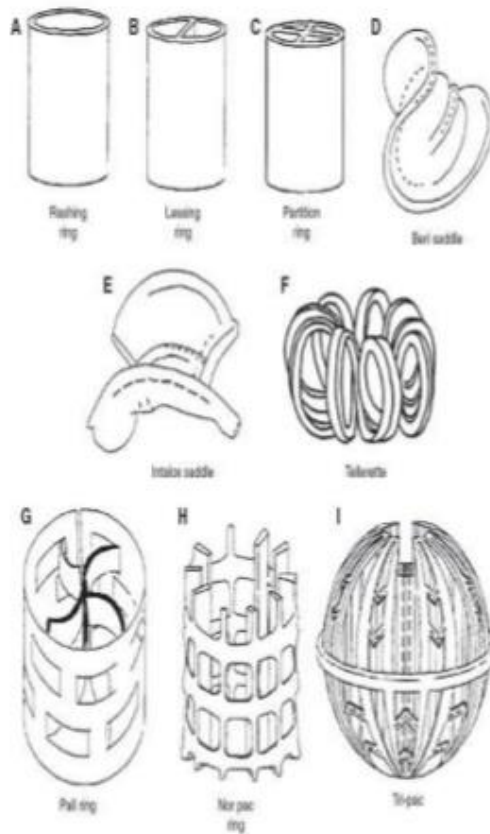


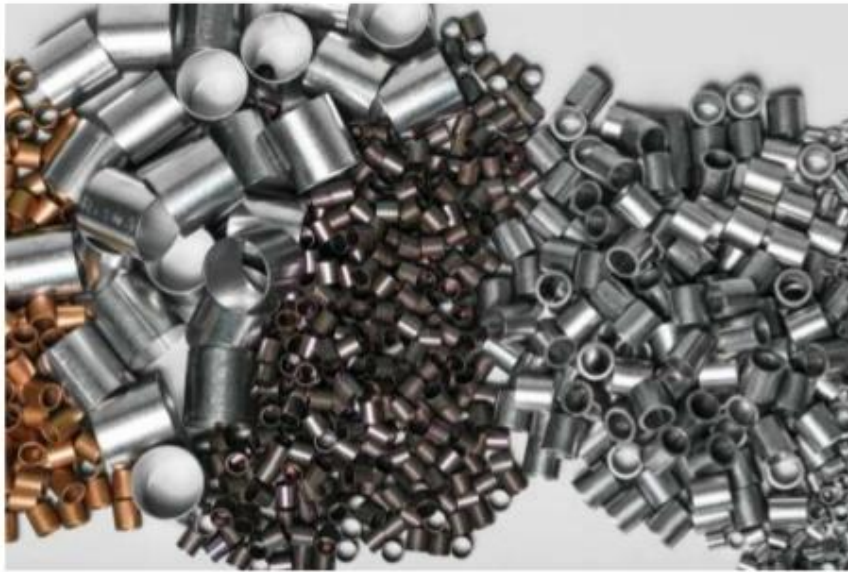
### **Packing materials:**

1-*Ceramic*: superior wettability ,corrosion resistance at elevated temperature ,bad strength.

2-*metal*: superior strength and good wettability.

3-*plastic*:inexpensive, good strength but may have poor wettability at low liquid rate.





## Types of packing in a absorption tower

1. Random Packing ( Irregular)
2. Structured packing( Regular)

## ***Type of packed column:***

### ***1. Random Packed Column:***

Random packing is packing of specific geometrical shapes which are dumped into the tower and orient themselves randomly. Random packing has more risk than structured packing and less ability to handle maldistributed liquid.



### ***2. Structured Packed Column :***

is crimped layers or corrugated sheets which is stacked in the column. Each layer is oriented at 70° to 90° to the layer below. Structured packed offers 30% capacities higher than random packed for equal efficiency up to 50% higher at the same capacity.



## Cases of operation for both types of towers::

**Priming:** Forth formation between trays ( Mainly in tray towers)

**Coning:** In low gas velocity, the gas will make channel and move in the opening of the trays by pushing the liquids away.

**Weeping or Dumping:** If the gas velocity is too low, the liquids will move down ( Leaks) through the openings provided for gas flow

**Flooding:** At very high gas velocity, it won't allow the liquids to come through the openings provided. High pressure drop occurs. Liquids will overflow the tower. Some times returns back through the inlet.

**Loading:** At a velocity where none of the above happens . Optimum Pressure drop

### 3.3

#### HETP (Height Equivalent to a Theoretical Plate)

As we have noted, instead of a tray (plate) column, a packed column can be used for various unit operations such as continuous or batch distillation, or gas absorption.

With a tray column, the vapours leaving an ideal plate will be richer in the more volatile component than the vapour entering the plate by one equilibrium "step".

When **packings** are used instead of trays, the same enrichment of the vapour will occur over a **certain height of packings**, and this height is termed the height equivalent to a theoretical plate (HETP). As all sections of the packings are physically the same, it is assumed that **one equilibrium (theoretical) plate is represented by a given height of packings**. Thus the required height of packings for any desired separation is given by (HETP x No. of ideal trays required).

HETP values are complex functions of temperature, pressure, composition, density, viscosity, diffusivity, pressure drop, vapour and/or liquid flowrates, packing characteristics, etc. Empirical correlations, though available to calculate the values of HETP, are restricted to limited applications. The main difficulty lies in the failure to account for the fundamentally different action of tray and packed columns

## 3.4

# Wetted wall Column OR Falling-film column

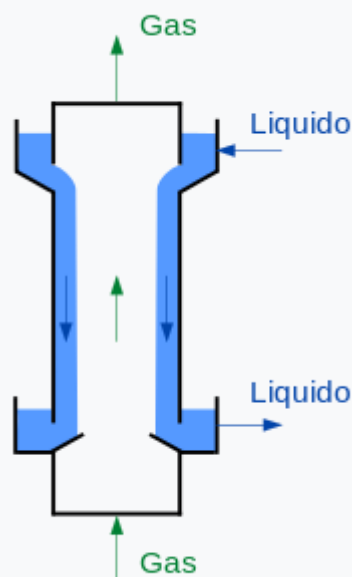
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A **falling-film column** (or **wetted-wall column**) is a particular chemical equipment used to achieve mass and heat transfer between two fluid phases (in general one gas phase and one liquid phase).

It is essentially formed by a vertical tube-shaped vessel: the liquid stream flows through the inner wall of the tube and the gas stream flows in correspondence of the centre of the tube.

## Description

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Schematics of a typical falling-film column.

In the most common case, the column contains one liquid stream and one gas stream. The liquid forms a thin film that covers the inner surface of the vessel,<sup>[1]</sup> instead the gas stream is normally injected from the bottom of the column, so the two fluids are subjected to a counter-current exchange of matter and heat, that happens in correspondence of the gas-liquid interface.

Sometimes, the same equipment is used to achieve the co-current mass and heat transfer between two immiscible liquids.

## Applications

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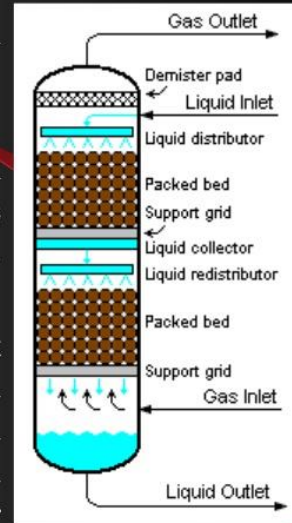
Because of its easiness of modelling, falling-film column is generally used as laboratory equipment, for example to measure experimentally the values of transport coefficients.<sup>[1]</sup> A significant experiment was carried out in 1934 by Edwin R. Gilliland and Thomas Kilgore Sherwood that used a falling-film column to study the mass transfer phenomenon between a liquid phase and a gas phase, obtaining an experimental correlation between Sherwood number, Reynolds number and Schmidt number.<sup>[3]</sup>

It is not used at an industrial scale, because it has low surface area and liquid hold-up compared to other gas-liquid contactors (e.g. a packed column or a plate column).



## Design of Absorption equipments

- Gas absorption is usually carried out in vertical counter current columns.
- The solvent is fed at the top of the absorber , whereas the gas mixture enters from the bottom .The absorbed substance is washed out by the solvent and leaves the absorber at the bottom as a liquid solution .
- The solvent is often recovered in a subsequent stripping or desorption operation . This second step is essentially the reverse of absorption and involves counter current contacting of the liquid loaded with solute using and inert gas or water vapor .



### Choice Of Solvent for Absorption

- If the principal purpose of the absorption operation is to produce a specific solution, as in the manufacture of hydrochloric acid, for example, the solvent is specified by the nature of the product, i.e. water is to be the solvent. If the principal purpose is to remove some components (e.g. impurities) from the gas, some choice is frequently possible.
- The factors to be considered are:



- **GAS SOLUBILITY :**

The gas solubility should be high, thus increasing the rate of absorption and decreasing the quantity of solvent required.

Solvent with a chemical nature similar to the solute to be absorbed will provide good solubility.

- **VOLATILITY :**

The solvent should have a low vapour pressure to reduce loss of solvent in the gas leaving an absorption column.

- **CORROSIVENESS :**

The materials of construction required for the equipment should not be unusual or expensive

- **COST :**

The materials of construction required for the equipment should not be unusual or expensive.

- **VISCOSITY :**

Low viscosity is preferred for reasons of rapid absorption rates, improved flooding characteristics in packed column, low pressure drops on pumping, and good heat transfer characteristics.

- The solvent should be non-toxic, non-flammable and chemically stable.

## COUNTERCURRENT FLOW

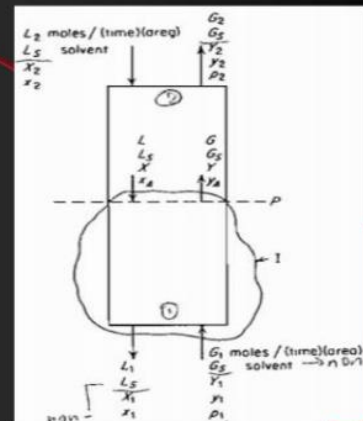
Clip slide

- Countercurrent tower which may be either a packed or spray tower, filled with bubble cap trays or of any internal construction to bring about liquid gas contact.
- Mole ratio may be define as

$$Y = y/(1-y)$$

$$X = x/(1-x)$$

- Here y for gas stream and x for liquid stream.



Material balance

$$G_s = G(1-y) = G/(1+Y)$$

$$L_s = L(1-x) = L/(1+X)$$

NOW

$$G_s(Y_1 - Y) = L_s(X_s - X)$$

AND

$$G_s(y_1/(1-y_1) - y/(1-y)) = G_s(p_1/(1-p_1) - p/(1-p)) = G_s(x_1/(1-x_1) - x/(1-x))$$

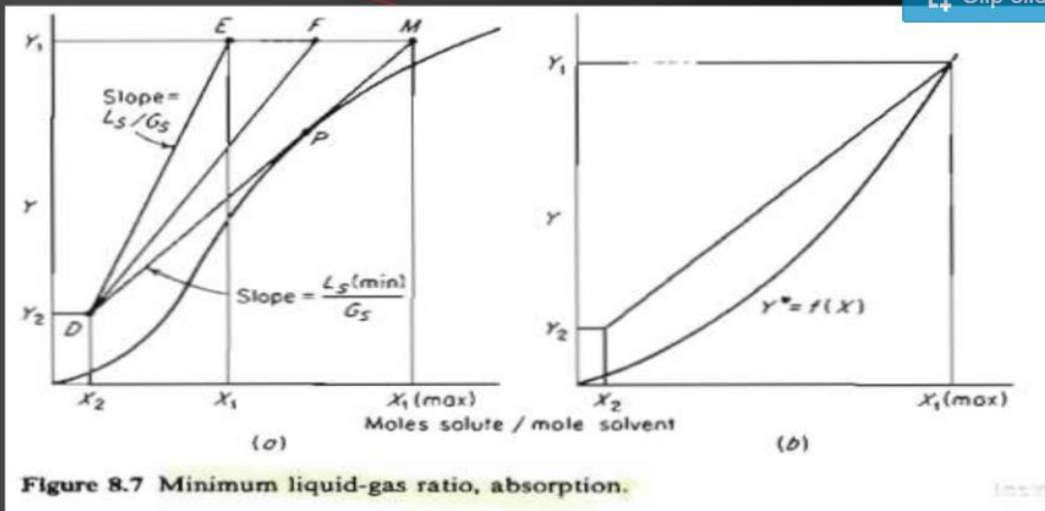


Figure 8.7 Minimum liquid-gas ratio, absorption.

## MINIMUM LIQUID-GAS RATIO

- In the design of absorbers, the quantity of gas to be treated  $G$  or  $G_S$ , the terminal concentrations  $Y_1$  and  $Y_2$ , and the composition of the entering liquid  $X_2$  are ordinarily fixed by process requirements.
- The operating line must pass through point  $D$  and must end at the ordinate  $Y$ .
- Liquid gas ratio is defined by
 
$$L/G$$
- Graph of minimum liquid-gas ratio can be shown as behind slide.

## CO-CURRENT FLOW

- When gas and liquid flow cocurrently, the operating line has a negative slope.  
-L/G
- There is no limit of this ratio, but an infinitely tall tower would produce an exit liquid and gas in equilibrium
- In cocurrent flow required height of tower should be higher than in counter current flow.

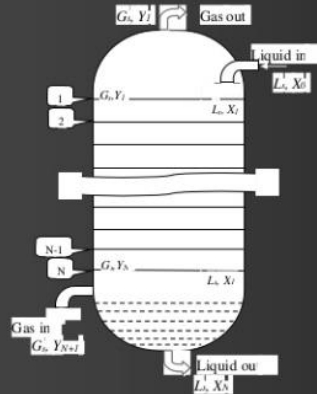
## Counter-current multi-stage absorption (Tray absorber):

- In tray absorption tower, multi-stage contact between gas and liquid takes place.
- In each tray, the liquid is brought into intimate contact of gas and equilibrium is reached thus making an ideal stage.
- In ideal stage, average composition of liquid leaving the tray is in equilibrium with liquid leaving that tray.
- The most important step in design of tray absorber is the determination of number of trays.
- The schematic of tray tower is presented in figure.
- The liquid enters from top of the column whereas gas is added from the bottom.

- The efficiency of the stages can be calculated as:

*Stage efficiency = Number of ideal stages / Number of real stages*

.....Eq.(1)



- The following parameters should be known for the determination of “number of stages”:

- (1) Gas feed rate
- (2) Concentration of gas at inlet and outlet of the tower
- (3) Minimum liquid rate; actual liquid rate is 1.2 to 2 times the minimum liquid rate.
- (4) Equilibrium data for construction of equilibrium curve

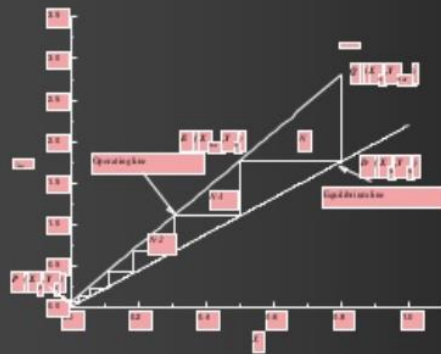
- Now, the number of theoretic stages can be obtained graphically:



### (A) Graphical Method for the Determination of Number of Ideal Stages:

- Overall material balance on tray tower:  
 $G_s(Y_{N+1} - Y_1) = L_s(X_N - X_0)$   
 This is the operating line for tray tower
- If the stage (plate) is ideal,  $(X_n, Y_n)$  must lie on the equilibrium line,  $Y^* = f(X)$ .
- Top plate is located at  $P(X_0, Y_1)$  and bottom plate is marked as  $Q(X_N, Y_{N+1})$  in X-Y plane.
- A vertical line is drawn from Q point to D point in equilibrium line at  $(X_N, Y_N)$ .
- From point D in equilibrium line, a horizontal line is extended up to operating line at E  $(X_{N-1}, Y_N)$ .

- The region QDE stands for N-th plate. We may get fraction of plates.
- In that situation, the next whole number will be the actual number of ideal plates.
- If the overall stage efficiency is known, the number of real plates can be obtained from Equation A.



**Example:**

It is desired to absorb 95% of acetone by water from a mixture of acetone and nitrogen containing 1.5% of the component in a countercurrent tray tower. Total gas input is 30 kmol/hr and water enters the tower at a rate of 90 kmol/hr. The tower operates at 27°C and 1 atm. The equilibrium relation is  $Y=2.53X$ . Determine the number of ideal stages necessary for the separation using graphical method.

**Solution:**

- Basis: 1 hour
- $G(N+1)=30$  kmol
- $Y(N+1)=0.015$
- $L_0=90$  kmol
- Moles acetone in =  $30 \times 0.015$  moles = 0.45 moles
- Moles nitrogen in =  $(30-0.45)$  moles = 29.55 moles
- Moles acetone leaving (95% absorbed) =  $0.45 \times (1-0.95)$  moles = 0.0225 moles
- $G_s=29.55$  moles
- $L_s=90$  moles
- $\alpha=2.53$  [as,  $Y=2.53X$ ]

- $Y_1 = 0.0225/29.55 = 7.61 \times 10^{-4}$
- $Y(N+1) = 0.015$
- Equation..  $GsY(N+1) - Y_1 = Ls(XN - X_0)$   
 $29.55 \times 0.015 - 7.61 \times 10^{-4} = 90(XN - 0)$
- $XN = 4.68 \times 10^{-3}$
- Solution by graphical method, Construction of operating line PQ:  
 $P(X_0, Y_1) = P(0, 7.61 \times 10^{-4})$   
 $Q(XN, Y_{N+1}) = Q(4.68 \times 10^{-3}, 0.015)$   
 Construction of equilibrium line ( $Y = 2.53X$ ):

X	0	0.001	0.002	0.003	0.004	0.005
Y	0	0.00253	0.00506	0.00759	0.01012	0.01265

From graphical construction in Figure , the number of triangles obtained is more than 7. Hence number of ideal stages is 8.

