FLOW OF FLUID

Fluid includes both liquids and gases.

- Fluids may be defined as a substance that does not permanently resist distortion. an attempt to change the shape of a mass of fluid will result in layers of fluids sliding over one another until a new shape is attained.

During the change of shape *shear stresses* will exist, the magnitude of which depends upon the viscosity of the fluid and the rate of sliding. But when a final shape is reached, all shear stresses will disappear. A fluid at equilibrium is free from shear stresses.

- The density of a fluid changes with temperature and pressure. In case of a liquid the density is not appreciably affected by moderate change of pressure.

In case of gases, density is affected appreciably by both change of temperature and pressure.

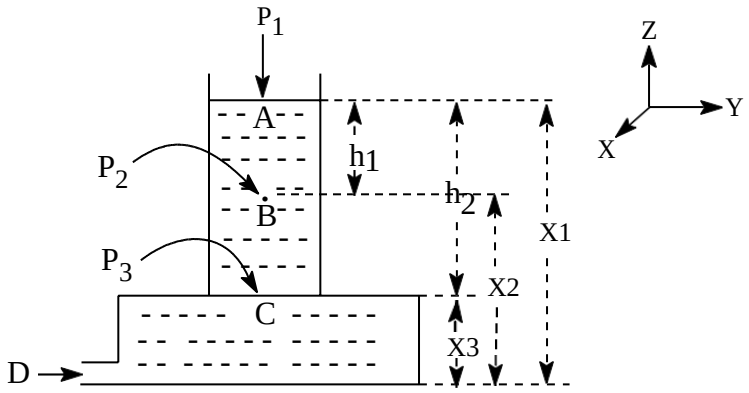
- The science of fluid mechanics includes two branches:

(i) fluid statics and (ii) fluid dynamics.

Fluid statics deals with fluids at rest in equilibrium.

Fluid dynamics deals with fluids under conditions where a portion is in motion relative to other portions.

FLUID STATICS



Hydrostatic pressure

In a stationary column of static fluid the pressure at any one point is the same in all directions. The pressure will also remain constant in any cross-section parallel to the earth's surface, but will vary from height to height.

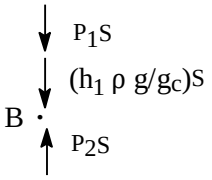
Let us consider, that the column of fluid in the figure is remaining at equilibrium. If the orifice D is open then the fluid will try to flow away. So either D is closed or a pressure is applied such that the liquid column stand at any desired height. The cross-section of the column is S (let). Now, say the pressure at the height $X_2 = P_2$ (in gravitational unit). At equilibrium all the forces acting on point B will be the same.

i.e. *Upward force* $=P_2S$

Downward forces:

Force given by atmosphere = P_1S

Force given by fluid column of height $h_1 = (h_1\rho\ g/g_c)S$



Where, ρ is the density of the fluid.

At equilibrium upward and downward forces are equal at point B.

$P_2S = P_1S + h_1 \rho S\ g/g_c$ eqn. (1)

where, each term of force is expressed in gravitational units i.e. lb_f, gm-wt, kg-wt etc.

$g/g_c \approx 1.0$ so equation (1) can be written as

$P_2S = P_1S + h_1 \rho S$

$P_2 = P_1 + h_1 \rho$ eqn. (2)

Similarly, $P_3 = P_2 + (h_2 - h_1)\rho$

$= P_1 + h_1 \rho + h_2 \rho - h_1 \rho$

$= P_1 + h_2 \rho$

$= P_1 + (X_1 - X_3)\rho$ [since $h_2 = X_1 - X_3$]

We can thus generalize for any point in the fluid, the pressure will be

$P_n = P_1 + \rho \Delta X$ where $\Delta X = X_1 - X_m$

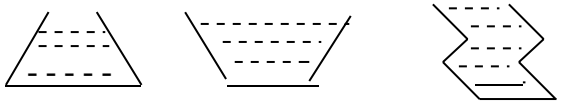
or, $P_n - P_1 = \Delta\rho X$

or, $\Delta P_n = \rho \Delta X$ eqn. (3)

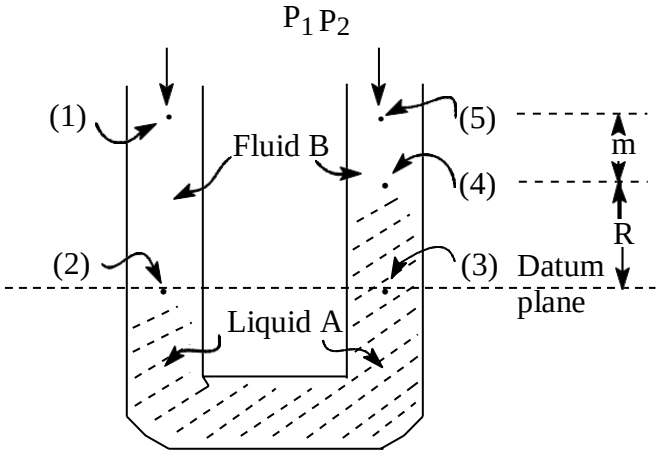
i.e. the pressure difference (ΔP_n) between any two points can be measured by the vertical distance between those two points, multiplied by the density of the fluid.

Since in equation (3) there is no term involving the cross-sectional area (S), it is not necessary that the vertical column be of uniform cross-section.

i.e. the shape may be any of the following types:



MANOMETERS



Simple Manometer

Manometers are used to measure the pressure of any fluid.

A U-tube is filled with a liquid A of density ρ_A. The arms of the U-tube above liquid A are filled with fluid B which is not miscible with liquid A and has a density of ρ_B. A pressure of P1 is exerted in one arm of the U-tube, and a pressure P2 on the other. As a result of the difference in pressure (P_1-P_2) the meniscus in one branch of the U-tube will be higher than the other branch.

The vertical distance between these two surfaces is R. *It is the purpose of the manometer to measure the difference in pressure (P_1 - P_2) by means of the reading R.*

At equilibrium the forces at the two points (2 and 3) on the datum plane will be equal.

Let the cross sectional area of the U-tube be S.

** All the forces are expressed in gravitational unit.

Total downward force at point (2) = Forces at point (1)
+ force due to column of fluid B in between points (1) and (2).
= $P_1S + (m + R)\,\rho_B\,(g / g_c)\,S$

Total downward force at point (3) = Force at point (5)
+ Force due to column of fluid B in between points (5) and (4)
+ Force due to column of liquid A in between points (4) and (3)
= $P_2S + m\,\rho_B\,(g/g_c)\,S + R\,\rho_A\,(g/g_c)\,S$

At equilibrium:

Force at point (2) = Force at point (3)

or, $P_1S + (m + R)\,\rho_B\,(g / g_c)\,S = P_2S + m\,\rho_B\,(g/g_c)\,S + R\,\rho_A\,(g/g_c)S$

or, $P_1 - P_2 = R_A\,(g/g_c) + m\,\rho_B\,(g/g_c) - m\rho_B\,(g/g_c) - R\,\rho_B\,(g/g_c)$

$= R\,(\rho_A - \rho_B)\,g/g_c$.or,

$$\Delta P = P_1 - P_2 = R\,(\rho_A - \rho_B)\,g/g_c.$$

It should be noted that this relationship is independent of the distance 'm' and cross sectional area 'S' of the U-tube, provided that P_1 and P_2 are measured from the same horizontal plane.

DIFFERENTIAL MANOMETER

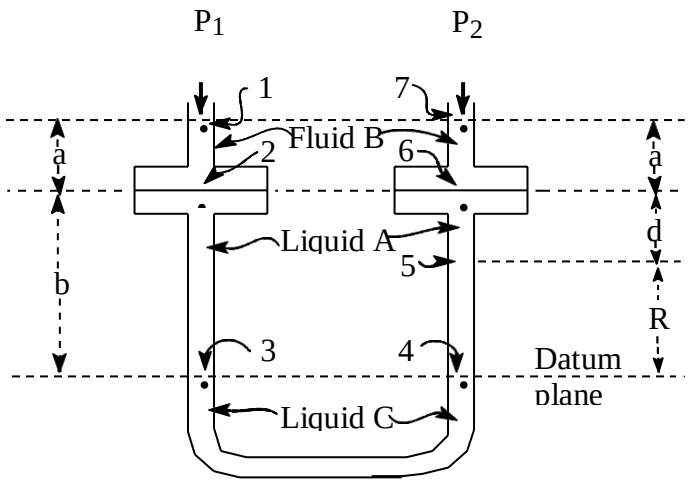


Fig. Differential manometer

For the measurement of smaller pressure differences, differential manometer is used.

The manometer contains two liquids A and C which must be immiscible.

Enlarged chambers are inserted in the manometer so that the position of the meniscus 2 and 6 do not change appreciably with the changes in reading.

So the distance between (1) and (2) = Distance between (6) and (7)

Total downward force on point (3)

$F_{left} = P_1S + a\,\rho_A\,g/g_c\,S + b\,\rho_A\,g/g_c\,S$

Total downward force on point (4)

$F_{right} = P_2S + a\,\rho_B\,g/g_c\,S + d\,\rho_A\,g/g_c\,S + R\rho_C\,g/g_c\,S$At equilibrium

$F_{left} = F_{right}$.

$\therefore \quad P_1S + a\,\rho_A\,g/g_c\,S + b_A\,g/g_c\,S = P_2S + a\,\rho_B\,g/g_c\,S + d\,\rho_A\,g/g_c\,S + R\rho_C\,g/g_c\,S$

$P_1 - P_2 = (d - b)\,\rho_A\,g/g_c + R\rho_C\,g/g_c$

$= - R\,\rho_A\,g/g_c + R\rho_C\,g/g_c.$

$= R\,(\rho_C - \rho_A)\,g/g_c$

$$\Delta P = P_1 - P_2 = R\,(\rho_C - \rho_A)\,g/g_c$$

From this it follows that the smaller the differences $\rho_C - \rho_A$,the larger will be the reading R on the manometer for a given value of ΔP.

INCLINED MANOMETER

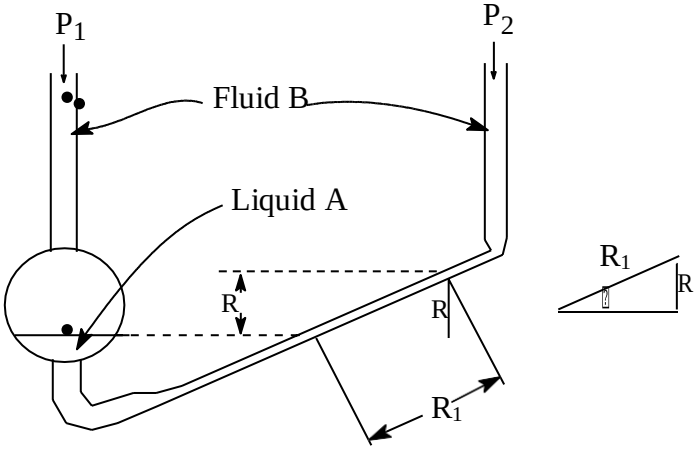


Fig. Inclined manometer

For measuring small difference in pressure this type of manometer is used.

In this type of manometer the leg containing one meniscus must move a considerable distance along the tube. Here the actual reading R is magnified many folds by R_1, where

$$R = R_1 \sin \alpha$$

where α is the angle of inclination of the inclined leg with the horizontal plane.

In this case $\Delta P = P1 - P2$

$= R\,(\rho_A - \rho_B)\,g/g_c.$

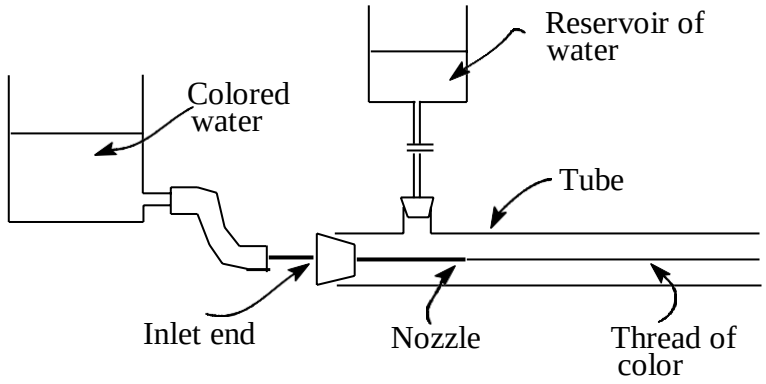
In this type of gauge it is necessary to provide an enlargement in the vertical leg so that the movement of the meniscus in this enlargement is negligible within the range of the

gauge.

By making α small the value of R is multiplied into a much larger distance R_1.

FLUID DYNAMICS

Reynolds' Experiment



This experiment was performed by Osborne Reynolds in 1883. I Reynolds experiment a glass tube was connected to a reservoir of water in such a way that the velocity of water flowing through the tube could be varied.

At the inlet end of the tube a nozzle was fitted through which a fine stream of coloured water can be introduced.

After experimentation Reynolds found that when the velocity of the water was low the thread of color maintained itself through the tube. By putting one of these jets at different points in cross section, it can be shown that in no part of the tube there was mixing, and the fluid flowed in parallel straight lines.

As the velocity was increased, it was found that at a definite velocity the thread disappeared and the entire mass of liquid was uniformly colored. In other words the individual particles of liquid, instead of flowing in an orderly manner parallel to the long axes of the tube, were now flowing in an erratic manner so that there was complete mixing.

When the fluid flowed in parallel straight lines the fluid motion is known as **Streamline flow** or **Viscous flow**.

When the fluid motion is erratic it is called turbulent flow. The velocity at which the flow changes from streamline or viscous flow to turbulent flow it is known as the critical velocity.

THE REYNOLDS NUMBER

From Reynolds' experiment it was found that critical velocity depends on

1. The internal diameter of the tube (D)
2. The average velocity of the fluid (u)
3. The density of the fluid (ρ) and
4. The viscosity of the fluid (μ)

Further, Reynolds showed that these four factors must be combined in one and only one way namely ($Du\rho/\mu$) . This function ($Du\rho/\mu$) is known as the Reynolds number. It is a dimensionless group.

it has been shown that for straight circular pipe, when the value of the Reynolds number is less than 2000 the flow will always be viscous.

i.e. NRe < 2000 ⟶ viscous flow or streamline flow

NRe > 4000 ⟶ turbulent flow

Dimensional analysis of Reynolds number

$[D] = L$ (ft)

$[u] = L/\theta$ (ft / sec)

$[\rho] = M / L^3$ (lb/ft^3)

$[\mu] = M / (L\theta)$ {lb/(ft sec)}

$$\left[\frac{Du\rho}{\mu}\right] = \frac{(L)(L/\theta)(M/L^3)}{\frac{M}{L\theta}} = \frac{L\,L\,M\,L\,\theta}{M\,\theta\,L^3}$$

$$= 1$$

BERNOULLI'S THEOREM

When the principle of conservation of energy is applied to the flow of fluids, the resulting equation is called *Bernoullis theorem*.

Let us consider the system represented in the figure, and **assume** that the temperature is uniform through out the system. This figure represents a

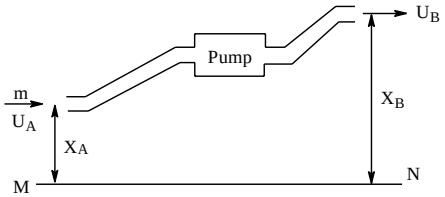


Fig. Bernoullis theorem

channel conveying a liquid from point A to point B The pump supplies the necessary energy to cause the flow. Let us consider a liquid mass **m** (lb) is entering at point A.

Let the pressure at A and b are P_A and P_B (lb-force/ft^2) respectively.

The average velocity of the liquid at A and B are u_A and u_B (ft/sec).
The specific volume of the liquid at A and B are V_A and V_B (ft^3/lb).
The height of point A and B from an arbitrary datum plane (MN) are X_A and X_B (ft) respectively.

Potential energy at point A, (W1)= mgX_A ft-poundal [absolute unit]
= $m (g/g_C)X_A$ ft-lb force = mX_A ft-lb force [gravitational unit]

Since the liquid is in motion
∴ Kinetic energy at point A, (W2) = $^1/_2 . m u_A^2$ ft-poundal
= $(^1/_2 . m u_A^2)/ g_C$ pound-force

As the liquid **m** enters the pipe it enters against pressure of P_A lb-force/ft^2 and therefore.
Work against the pressure at point A, (W3) = mP_AV_A $ft\text{-}lb_f$.

N.B. Force at point A = $P_A S$ [S = Cross-section area]
Work done against force $P_A S = P_A (S h) = P_A V$

∴ Total energy of liquid **m** entering the section at point a will be (E1) = W1 + W2 + W3

$$E1 = [\, mX_A + (^1/_2 . m u_A^2)/ g_C + mP_AV_A \,]\ ft\text{-}lb_f.$$

After the system has reached the steady state when ever **m** (lb) of liquid enters at A another **m** (lb) pound of liquid is displaced at B according to the principle of the conservation of mass. This **m** (lb) leaving at B will have energy content of

$$E2 = [\, mX_B + (^1/_2 . m u_B^2)/ g_C + mP_BV_B \,]\ ft\text{-}lb_f.$$

Energy is added by the pump. Let the pump is giving **w** $ft\text{-}lb_f$ / lb energy to the liquid

$$E3 = m w \quad ft\text{-}lb_f.$$

Some energy will be converted into heat by friction. It has been assumed that the system is at a constant temperature; hence, it must be assumed that the heat is lost by radiation or by other means. Let this loss due to friction be F $ft\text{-}lb_f$ / lb of liquid.

$$E4 = - mF \quad ft\text{-}lb_f \quad [\text{negative sign for loss}]$$

∴ The complete equation representing energy balance across the system between points A and will therefore be

$$E1 + E3 + E4 = E2$$

or, $mX_A + (^1/_2 . m u_A^2)/ g_C + mP_AV_A + m w - mF = mX_B + (^1/_2 . m u_B^2)/ g_C + mP_BV_B$ Now,

the unit of energy term is $ft\text{-}lb_f$ / lb

The BERNOULLI'S THEOREM.

$$X_A + \frac{U_A^2}{2g_C} + P_AV_A + w - F = X_B + \frac{U_B^2}{2g_C} + P_BV_B$$

The density of the liquid ρ be expressed lb_m / ft^3, then

$V_A = 1 / \rho_A$ and $V_B = 1 / \rho_B$ then Bernoulli's equation can be written in the form also

$$X_A + \frac{U_A^2}{2g_C} + \frac{P_A}{\rho_A} + w - F = X_B + \frac{U_B^2}{2g_C} + \frac{P_B}{\rho_B}$$

FLUID HEADS

All the terms in Bernoulli's theorem have unit of **ft-lb$_f$ / lb$_m$** which is numerically equal to '**ft**' only. That is each and every time terms can be expressed by height.

Dimensional Analysis

[ft] = L

[lb$_f$] = $(ML\theta^{-2}) / (L\theta^{-2})$ = M

[lb$_m$] = M

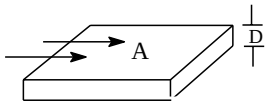
[ft-lb$_f$ / lb$_m$] = L~~M~~ / ~~M~~ = L

That is every term has a dimension of length (or height) if the terms are expressed in gravitational unit. This height are termed as **heads** in the discussions of hydraulics. Each term has different names:

Potential heads	XA , XB.
Velocity heads	UA2 / (2 gC), UB2 / (2 gC)
Pressure heads	PA VA , P A □A , PB VB , PB □B .
Friction head	F
Head added by the pump	w

FRICTION LOSSES

In Bernoulli's equation a term was included to represent the loss of energy due to friction in the system. The frictional loss of a fluid flowing through a pipe is a special case of general law of the resistance between a solid and fluid in relative motion.



Let us consider a solid body of any designed shape, immersed in a stream of fluid.

Let, the area of contact between the solid and f fluid = A

If the velocity of the fluid passing the body is small in comparison to the velocity of sound, it has been found experimentally that the resisting force depends only on the roughness, size and shape of the solid and on the velocity, density and viscosity of the fluid. Through a consideration of the dimensions of these quantities it can be shown that,

$$\frac{F}{A} = \frac{\rho u^2}{g_c}\ \phi\left[\frac{Du\rho}{\mu}\right]$$

where, F = total resisting force

A = area of solid surface in contact with fluid

u = velocity of the fluid passing the body

ρ = density of fluid

μ = viscosity of fluid

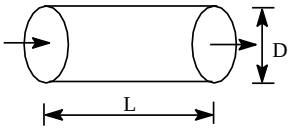
g_c = 32.2 (lb$_m$ ft)/ (lb$_f$ s^2)

ϕ = some friction whose precise form must be determined for each specific case.

The form of function ϕ depends upon the geometric shape of the solid and its roughness.

FRICTION IN PIPES

In a particular case of a fluid flowing through a circular pipe of length L, the total force resisting the flow must equal the product of the area of contact between the fluid and the pipe wall and F/A of the friction loss equation.



The pressure drop will be:

$$\Delta P_f = \frac{\text{Total force}}{\text{Cross sectional area}}$$

$$= \frac{(F/A)(L\pi D)}{\pi D^2/4}$$

$$= \frac{F}{A}\,\frac{L\pi D}{\pi D^2/4}$$

Since $\frac{F}{A} = \frac{\rho u^2}{g_c}\,\phi\left[\frac{Du\rho}{\mu}\right]$ Therefore $\Delta P_f = \frac{\rho u^2}{g_c}\,\phi\left[\frac{Du\rho}{\mu}\right]\left[\frac{4L\pi D}{\pi D^2}\right]$

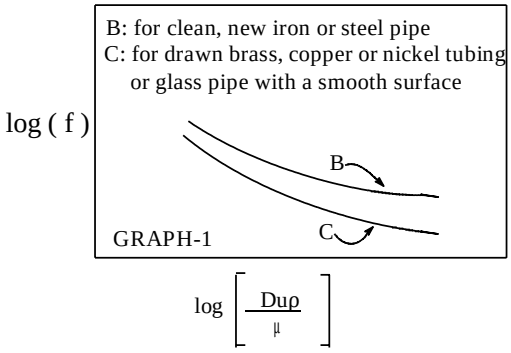
$$= \frac{4u^2 L\rho}{g_c D}\,\phi\left[\frac{Du\rho}{\mu}\right] \text{ --------- eqn (1)}$$

where ΔP_f = pressure drop due to friction (lb/ft^2)
F / A = resisting force ($ft\text{-}lb_f$ per ft^2 of contact area)
L = length of pipe (ft)
D = inside diameter of the pipe (ft)
ρ = density of fluid (lb_m / ft^3)
u = average velocity of fluid (ft / s)
μ = viscosity of fluid (lb_m / ft / s)
g_c = 32.2 (lb_m ft / lb_f s^2)

For many decades Fanning's equation was used:

$$\Delta P_f \quad \frac{2 f u^2 L \rho}{g_c D} \text{ ---------- eqn (2)}$$

In Fanning's equation the value of '**f** ' was taken from tables. This equation however has been widely used for so many years that most engineers still use the Fanning's equation, except that instead of taking values of 'f' from arbitrary tables a plot of the equation $f = (Du\rho / \mu)$ is used.
The graph (Graph-1) is not that much accurate : Error: ±5 to 10 % may be expected for <u>laminar flow</u> .



By combining Hagen Poiseulles equation a new simple form of equation can be obtained.

$$f = \frac{16}{\frac{Du\rho}{\mu}} = \frac{16}{\text{Reynolds No}}$$

MEASUREMENT OF FLUID FLOW

Methods of measuring fluids may be classified as follows:-

1) Hydrodynamic methods
- *(a)* Orifice meter
- (b) Venturimeter
- (c) Pitot tube
- (d) Rotameter
- (e) Weirs

2) *Direct displacement*
- (a) Disc meters
- (b) Current meters

3) *Dilution method and*

4) *Direct weighing or measuring*

ORIFICE METER

Objective:

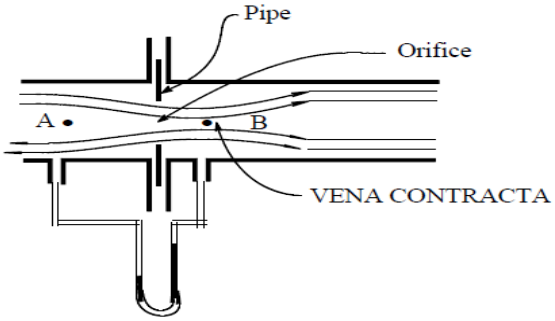
To measure the flow of fluids.

i) Velocity of fluid through a pipe (ft/sec)

ii) Volume of liquid passing per unit time(ft^3/sec, ft^3/min, ft^3/hr).

Description

An orifice meter is considered to be a thin plate containing an aperture through which a fluid issues. The plate may be placed at the side or bottom of a container or may be inserted into a pipeline.



A manometer is fitted outside the pipe. One end at point A and the other end at point B (see fig.). The pressure difference between A and B (i.e. before and after the orifice) is read, and the reading is then converted to fluid flow-rate.

Derivation

Bernoulli's equation is written between these two points, the following relationship Holds

$$X_A + \frac{U_A^2}{2g_c} + \frac{P_A}{\rho_A} - F + w = X_B + \frac{U_B^2}{2g_c} + \frac{P_B}{\rho_B} \quad \text{.....(1)}$$

Conditions	Equation (1) changes to:
i) The pipe is horizontal $\therefore X_A = X_B$.	$\frac{U_A^2}{2g_c} + \frac{P_A}{\rho_A} - F + w = \frac{U_B^2}{2g_c} + \frac{P_B}{\rho_B}$
ii) If frictional losses are assumed to be inappreciable then F = 0	$\frac{U_A^2}{2g_c} + \frac{P_A}{\rho_A} + w = \frac{U_B^2}{2g_c} + \frac{P_B}{\rho_B}$
iii) If the fluid is a liquid then $\rho A \approx \rho B = \rho$ (let)	$\frac{U_A^2}{2g_c} + \frac{P_A}{\rho} + w = \frac{U_B^2}{2g_c} + \frac{P_B}{\rho}$
iv) Since no work is done on the liquid, or by the liquid between A and B. $\therefore w = 0$	$\frac{U_A^2}{2g_c} + \frac{P_A}{\rho_A} = \frac{U_B^2}{2g_c} + \frac{P_B}{\rho_B}$ ..... (2)

Equation (2) may be written as:

$$U_B^2 - U_A^2 = \frac{2g_C}{\rho}(P_A - P_B) \quad \text{.....(3)}$$

Since, $P_A - P_B = \Delta P$, and since $\frac{\Delta P}{\rho} = \Delta H$

$\therefore$ equation (3) can be written as

$$\sqrt{U_B^2 - U_A^2} = \sqrt{2g_C\ \Delta H} \quad \text{.....(4)}$$

N.B. $P_A = H_A \rho g / g_c$
$P_B = H_B \rho g / g_c$
$P_A - P_B = (H_A - H_B)\rho g / g_c$
or, $\Delta P = \Delta H - g / g_c$.
Since, $g / g_c \approx 1.0$ hence, $\Delta H = \Delta P /$
If the pipe to the right of the orifice plate were removed so that the liquid issued as a jet from the orifice, the minimum diameter of the stream would be less than the diameter of the orifice. This point of minimum cross-section is known a vena-contracta.

Point B was chosen at the vena-contracta. In practice the diameter of the stream at the vena-contracta is not known, but the orifice diameter is known. Hence equation (4) may be written in terms of the velocity through the orifice, as a result a constant (Co) has to be inserted in the equation (4) to correct the difference between this velocity and the velocity at the vena-contracta. There may be some loss by friction and this also may be included in the constant. Equation (4) then becomes:

$$\sqrt{U_0^2 - U_A^2} = C_0 \sqrt{2g_C\ \Delta H} \quad \text{.....(5)}$$

where U_0 = velocity through the orifice.
The pressure difference ΔP between A and B is read directly from the manometer.In equation (5)

ΔH is measured from manometer $(\Delta P/\rho)g_c$ is constant
C_0 is constant and known for a particular orifice meter.
U_0 and U_A is unknown

So to solve both U_0 and U_A another equation is required. We can assume that the volume flow-rate at A and orifice are equal, we can thus deduce the following equation.

$$U_A \frac{\pi d_P^2}{4} = U_O \frac{\pi d_O^2}{4} \qquad \text{or,} \qquad \frac{U_A}{U_O} = \left(\frac{d_O}{d_P}\right)^2 \quad \text{.....(6)}$$

where, d_P = diameter of pipe
d_O = diameter of orifice
d_P and d_O are already known

Now we can solve equation (5) and (6) to get the value of both U_A and U_O.

U_A = velocity of fluid in the pipe

$$U_A \times \frac{\pi d_P^2}{4} = \text{volume flow rate of fluid in the pipe.}$$

The constant Co depends on the

- ratio of the orifice diameter to the pipe diameter

- position of the orifice taps
- value of Reynolds number for the fluid flowing in the pipe.

For values of Reynolds number (based on orifice diameter i.e. Re = $\frac{d_O u_O \rho}{\mu}$ of 30,000 or above, the value of Co may be taken as 0.61.

Advantage

It is very simple device and can be easily installed i.e. cost of installation is less.

Fluids of various viscosity can be measured just by changing the orifice diameter.

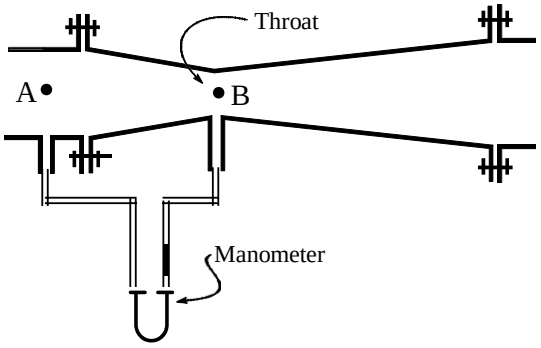
Disadvantage

The orifice always results in a permanent loss of pressure (head), which decreases as the ratio of orifice diameter to pipe, diameter increases i.e. cost of operation, particularly for long term, is considerable.

VENTURIMETER

Description

The venturimeter, as shown in the figure consists of two tapered sections inserted in the pipeline, with the tapers smooth and gradual enough so that there are no serious loss of energy. At point B the section of venturimeter has minimum diameter. This point is called the 'throat' of the venturimeter.



The venturimeter is fitted within a pipe. The pressure difference at A and B is measured by a manometer.

Derivation

If the Bernoulli's equation is written between these two points the following relationship holds.

$$X_A + \frac{U_A^2}{2g_c} + \frac{P_A}{\rho_A} - F + w = X_B + \frac{U_B^2}{2g_c} + \frac{P_B}{\rho_B} \quad \text{.....(1)}$$

Conditions	Equation (1) changes to:
i) The pipe is horizontal $\therefore X_A = X_B$	$\frac{U_A^2}{2g_c} + \frac{P_A}{\rho_A} - F + w = \frac{U_B^2}{2g_c} + \frac{P_B}{\rho_B}$
ii) If frictional losses are assumed to be inappreciable then F = 0	$\frac{U_A^2}{2g_c} + \frac{P_A}{\rho_A} + w = \frac{U_B^2}{2g_c} + \frac{P_B}{\rho_B}$
iii) If the fluid is a liquid then $\rho A \approx \rho B = \rho$ (let)	$\frac{U_A^2}{2g_c} + \frac{P_A}{\rho} + w = \frac{U_B^2}{2g_c} + \frac{P_B}{\rho}$
iv) Since no work is done on the liquid, or by the liquid between A and B. i.e. w = 0	$\frac{U_A^2}{2g_c} + \frac{P_A}{\rho_A} = \frac{U_B^2}{2g_c} + \frac{P_B}{\rho_B}$ ..... (2)

Equation (2) may be written as:

$$U_B^2 - U_A^2 = \frac{2g_c}{\rho}(P_A - P_B) \quad \text{.....(3)}$$

Since, $P_A - P_B = \Delta P$, and since $\frac{\Delta P}{\rho} = \Delta H$

equation (3) can be written as

$$\sqrt{U_B^2 - U_A^2} = \sqrt{2g_C \Delta H} \quad \text{.....(4)}$$

N.B. $P_A = H_A \rho g / g_c$

$P_B = H_B \rho g / g_c$

$P_A - P_B = (H_A - H_B) g / g_c$

or, $\Delta P = \Delta H \rho g / g_c$.

Since, $g / g_c \approx 1.0$ hence, $\Delta H = \Delta P / \rho$

If the pipe to the right of the orifice plate were removed so that the liquid issued as a jet from the orifice, the minimum diameter of the stream would be less than the diameter of the orifice. This point of minimum cross-section is known a vena-contracta.

Since there are practically no losses dude to eddies and since the cross-section of the high velocity part of the system is accurately defined hence equation (4) may be written as

$$\sqrt{U_B^2 - U_A^2} = C_V \sqrt{2g_C \Delta H} \quad \text{.....(5)}$$

where U_B = velocity at the throat of the venturimeter

In case of venturimeter the value of coefficient $C_V = 0.98$.

Comparison between orificemeter and venturimeter:

Orifice meter	Venturimeter
1. Installation is cheap and easy. 2. The power loss is considerable in long run. 3. They are best used for testing purposes or other cases where the power loss is not a factor, as in steam lines. 4. Installing a new orifice plate with a different opening is a simple matter.	1. Installation is costly. It is less easier than orifice meter. (*Disadvantage*) 2. Power loss is less in long run even negligible (*Advantage*) 3. Venturimeters are used for permanent installation. 4. Installation of a different opening require replacement of the whole venturimeter. (*Disadvantage*)

PITOT TUBE

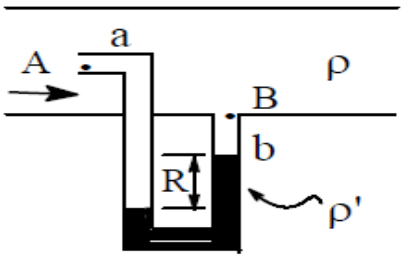


Fig 1

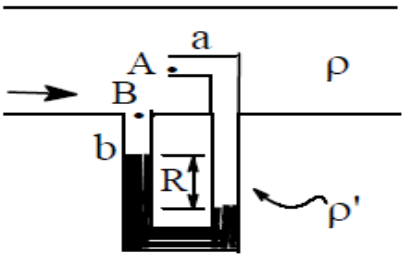


Fig 2

The pitot tube is a device to measure the local velocity along a streamline. The configurations of the device are shown in the figure. The manometer has two arms. One arm 'a' is placed at the center of the pipe and opposite to the direction of flow of fluid. The second arm 'b' is connected with the wall of the pipe. The difference of liquid in two arms of the manometer is the reading.

The tube in the 'a' hand measures the pressure head (X_A) and the velocity head $\left(\frac{U_A^2}{2g_C}\right)$. The 'b' hand measures only pressure head (X_B).

$$X_A + \frac{U_A^2}{2g_C} = X_B \qquad \text{or, } X_A - X_B = \frac{U_A^2}{2g_C} \qquad \text{or, } \Delta X = \frac{U_A^2}{2g_C} \quad \text{.....(i)}$$

Here ΔX_B is the pressure head of the fluid whose flow is to be measured that corresponds to R.

Since the manometer measures the pressure according to the following equation.

$$\Delta X = (\rho' - \rho)\, R\, g/g_C$$

or, $= (\rho' - \rho)\, R$ [Since $g/g_C \approx 1$]

where, ρ' = density of the liquid in the manometer

ρ = density of the fluid in the pipe.

Replacing ΔX in the equation (i) gives,

$$(\rho' - \rho)\, R = \frac{U_A^2}{2g_C} \qquad [U = U_A \text{ (let)}]$$

$$\therefore\ U = \sqrt{2\,(\rho' - \rho)\, g_C\, R} \quad \text{.....(ii)}$$

The velocity measured is the maximum velocity inside the pipe.

$$U_{max} = \sqrt{2\, g_C\, (\rho' - \rho)\, R}$$

By orifice meter or venturimeter average velocity of fluid is measured. With pitot tube velocity of only one point (i.e. at the center of the pipe) is measured. To convert U_{max} to average velocity ($\overline{U}$) the following relationship is taken into concern.

where, D = diameter of the pipe

U_{max} = maximum velocity of fluid

ρ = density of the fluid flowing

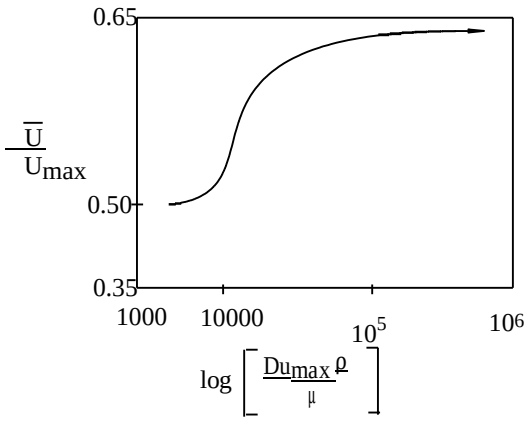
μ = viscosity of the fluid flowing

$\overline{U}$ = average velocity in the pipe

Disadvantage of pitot tube

1. It does not give the average velocity directly.

2. When velocity of gases are measured the reading are extremely small. In these cases some form of multiplying gauge like differential manometer and inclined manometers are used.



where D= diameter of the pipe
U_{max} = maximum velocity of fluid
ρ = density of the fluid flowing
μ = viscosity of the fluid flowing
U = average velocity in the pipe

SIZE REDUCTION

- It is operation of reducing large solid unit masses (vegetable drug, chemical substance) into small unit masses, coarse particle or fine powder
- It is unit operation (individual step)
- It is also k/a comminution (Latin world- minuere – less) or comminution or milling or grinding or pulverizing.
- Normally size reduction achieved by two method
  1- Chemical method – Precipitation method
  2- Mechanical method - Use mechanical force for grinding or size reduction

Objective /Advantage /importance of size reduction

- Properties of powder can be affected by the size of particle or surface area of powder

1. **Surface area-** surface area affects various properties of drugs.

If particle size decrease ⟶ surface area of particle increased.

a). Solubility –

If particle size decreases ⟶ surface area of particle increased

↓

Solubility increased

b). **Drying** – drying is faster after size reduction.

E.g. Medicinal plants ⟶ (Size reduction) smaller pieces/ particles (surface area of particle increased)

⇩

Drying fast

c). Adsorption -: adsorption is surface phenomena.

- Adsorption capacity of material will be increased when particle size decreased.

If particle size decreases ⟶ Adsorption capacity of material will be increased

d). **Absorption/Bioavailability** -: absorption of drugs affected by particle size.

E.g. Choremphenicol

- ✓ Particle size 50 µm ⟶ faster absorption
- ✓ Particle size 400 µm ⟶ slow absorption

e). **Extraction-**

If decreases size of crude drug ⟶ surface area of crude drug increased

↓

Extraction increased

2. **Size-** size affects various properties of dosage forms.

A – **Suspension -** drug particles suspended in liquid.

I. **If particle size small** – in sedimentation solid cake form & difficult to redisperse.
II. **If large particle**- in sedimentation porous cake form & easy to redisperse.
III. **Parenteral suspension**- Syringeability (i.e. ease of withdrawal from vial to syringe)

B- Mixing – if particle size uniform and small then effective mixing occurs.

C). Flow property- smaller particles and controlled size distribution have good flow property.

3. **Change of colour** **size reduction**
   - Red mercuric oxide ⟹ fine state (Yellow colour)
4. **It improves appearance** – milling of ointment ,cream and paste give smooth texture and elegant appearance
5. **Reduce irritation**- Ophthalmic preparation

FACTOR AFFECTING SIZE REDUCTION

- Size reduction operation used in many industries like flour, cements & coal etc.
- In pharmaceutical industries size reduction used for variety of material like chemical substances, animal tissue, and vegetable dugs.
- Properties of material affect size reduction.
- Following factor affect the size reduction like

1. **Hardness**
2. **Toughness**
3. **Abrasiveness**
4. **Stickiness**
5. **Softening temperature**
6. **Moisture content**
7. **Physiological effect**
8. **Bulk density**
9. **Purity required**
10. **Ratio of feed size to product size**

1). Hardness --: hardness is surface properties & it affects the process of Size reduction.

- Size reduction of harder material is difficult.
- Size reduction of harder and brittle (Glass) material is easy.

Moh's Scales -: An arbitrary scale of hardness

Range – (1 to 10) ⟶ graphite to diamond

Upto 3 = Soft material & can be marked with finger nail (wax)

3-7 = Intermediate (limestone)

Above 7 = Hard material & cannot be marked with knife blade (diamond)

2). Toughness-: (fibrous and flexible materials)

- The crude drug of fibrous in nature having higher moisture content and tough in nature.
- Size reduction of soft but tough materials is difficult than hard and brittle (Glass) substances.
- Toughness can be reduced by using Liquefied nitrogen gas.

Rubber ⟶ (Liquefied nitrogen; Temp. Decrease (-100 to -150^{0}C)) ⟶ rubber become brittle in nature ⟶ Size reduction easy

- This process is little used due to it cast.

3). Abrasiveness

- It is property of hard materials, particularly those of mineral in origin

During grinding of abrasive material ⟶ metal worn (detach) from instrument/mill. ⟶ Feed material contaminated with material of instruments

4). Stickiness -:

- Sticky materials cause a lot of difficulty in size reduction.
- Materials are gummy or resinous may adhere with part of instruments/mill and create problem in

size reduction.

- Before size reduction dry the sticky materials by two methods
    1. By using heat
    2. By using inert substance
    3. By intervention (use volatile substance)

5). **Softening temperature-**:

- Waxy substance like stearic acid, drug containing oil or fat
- During size reduction, heat is generated and waxy materials become sof.
- So some methods are used for cooling of mill like
    1. By applying cool water/ air stream
    2. By using liquid nitrogen gas.

6). Moisture content-:

- Moisture content of material affects size reduction.
- The presence of moisture in the materials influences a number of its properties like hardness, toughness or stickiness which affects the size reduction.
- Materials contain 5% moisture ⟶ suitable for Dry grinding
- Materials contains 50% moisture ⟶ suitable for Wet grinding

7). **Physiological effect-:**

- Some drug are very potent (having great power or effect like hormonal , cardiac and CNS drugs)
- During size, reduction of potent drug small amount of dust may have an effect on the operator.
- In such case, enclosed mills may be used to avoid dust

8). Bulk density-:

- It is defined as the mass of the material divided by the total volume it occupy
- Capacity of most batch mill depends on the volume of materials
- The output of size reduction of materials in machine depends upon bulk density of the substance.

9). Purity required-:

- In some mills, during size reduction surface of mill wear off (removal) and mix in product thus impurity come in powder.
- If high degree of purity is required, such mill must be avoided.

10). Ratio of feed size to product size -:

- Ratio of feed size to product size also affects the size reduction of materials.
- If small feed size ⟶ get fine powder
- Hence size reduction process carried in several stage

Preliminary crushing ⟶ (1) coarse powder ↓ (2) Fine powder

METHOD/MECHANISM OF SIZE REDUCTION

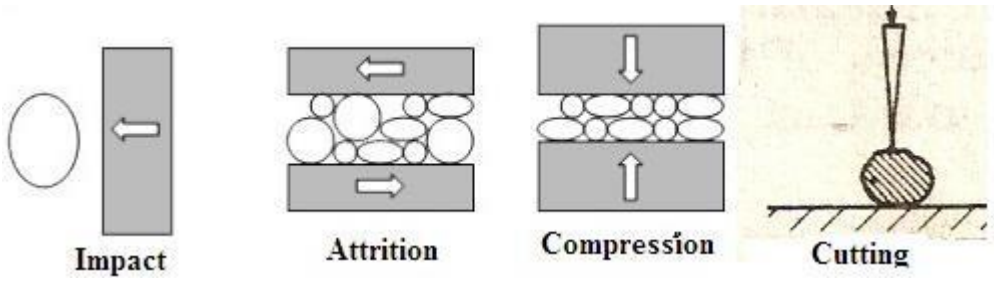
- Four methods are used for size reduction.
    - **a) Cutting**
    - **b) Compression**
    - **c) Impact**
    - **d) Attrition**

a) **Cutting** – material is cut by means of sharp blades.
e.g. - Cutter mill

b) **Compression-** material is crushed by application of pressure.
e.g. - Roller mill

c) **Impact**- materials are hit by moving objects like hammer.
- ❖ Impact occurs when moving objects (hammer) strike against stationary surface.
  e.g- Hammer mill
- ❖ Impact also occurs when moving materials strike against stationary surface
  e.g- Fluid Energy mill

d) **Attrition** – materials can be break by rubbing action between two surfaces.
- ❖ It is surface phenomena
- ❖ Material is subjected to pressure as in compression, but the surfaces are moving relative to each other.
  e.g- Fluid Energy mill



- ❖ Attrition and impact are the most commonly used methods.
- ❖ Compression and cutting are used rarely in size reduction.
- ❖

Particle size reduction on small scale (Pulverization)

1. **By Trituration**: rubbing action
   - ❖ The force acting on the pestle during trituration brings about crushing of ingredients.
   - ❖ Substances are reduced to a fine powder.
   - ❖ It is the continuous rubbing or grinding of the powder in a **mortar with a pestle**.
   - ❖ This method is used when working with hard, fracturable powders.
2. **By Intervention**:
   - ❖ Substance to be pulverized is first dissolved in a volatile solvent (**in which it is soluble**) which is then allowed to evaporate
     E.g.- camphor-(it is sticky and re-agglomerates or resists grinding).
     - ➢ Hence use alcohol or other volatile solvent for grinding.
     - ➢ Mix camphor and alcohol/volatile solvent
     - ➢ Solvent is removed by spraying the powdered camphor in a thin layer
3. **Levigation**:
   - ❖ Substance is ground in the presence of a liquid **in which it is insoluble**.
   - ❖ The material is introduced into the mill together with water
   - ❖ For cream and paste

Table 1: Mechanism of Size Reduction-Methodology

Methods	Examples	Approx. particle size (µm)
Cutting	Scissors Shears, Cutter Mill	100-80,000
Compression	Roller Mill, Pestle-Mortar	50-10,000
Impact	Hammer Mill, Disintegrator	50-8000
Attrition	Colloidal Mill, Roller Mill	1-50
Combined Impact and Attrition	Ball Mill, Fluid Energy Mill	1-2000

MILL USED FOR SIZE REDUCTION

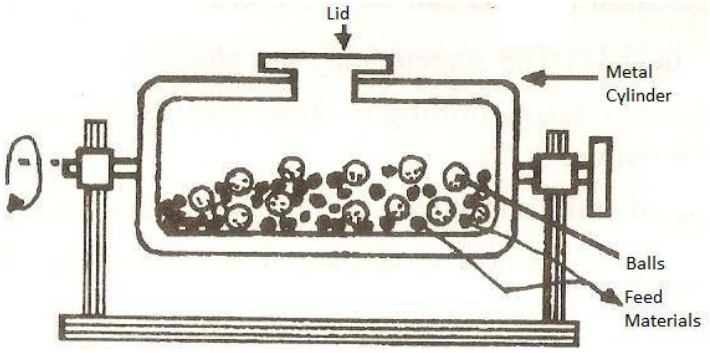
- following mill used for size reduction like ball mill, fluid energy mill, hammer mill, edge runner mill , end runner mill, disintegrator etc

BALL MILL (tumbling or pebble mill)

Principle- Impact **(**between balls and materials**)** and attrition **(**between the balls**)**

Construction

- **It** consist hollow metal cylindrical body.
- Cylindrical body is mounted on frame**.**
- Frame is rotates on its longitudinal axis**.**
- Length of cylinder is greater than its diameter.
- Cylinder is made up metal and coated with chrome or rubber**.**
- Cylinder contains balls.
- Ball occupies 30-50% space of mill.
- Ball is made up of iron, steel or stoneware.



Speed of mill-

a) **At low speed-** ball roll over each other and minimum size reduction occur.
b) **At high speed-** ball thrown out the wall by centrifugal force and **no size reduction**.
c) **At moderate speed (Cascading) -** ball are picked up by mill wall and fall to the bottom (Impact) and ball slide over each other (attrition)- **Maximum size reduction occur**
   - ✓ **Critical speed.**

$$n_C = \frac{76.63}{\sqrt{D}}$$

D= diameter of mill in feet
n_c = critical speed in rpm

- ✓ The point where the mill becomes a centrifuge is called the "**Critical Speed**",
- ✓ Ball mills usually operate at 65% to 75% of the critical speed.

Working

Materials and balls placed in chamber → Rotates the chamber/mill

↓

Size of materials reduces
(Due to Impact and attrition **b**etween balls and materials)

↓

Reduced particle separate through mesh/screen

Use- It is used for hard and abrasive materials
It is used for milling of dye, pigment and insecticide at low speed.

Advantages

1. It can produce very fine powder.
2. Wide variety of materials can use for size reduction.
3. It can be using both wet and dry grinding process.

4. They are economical and easy to operate.
5. Various shape and size of ball can be use.

Disadvantages

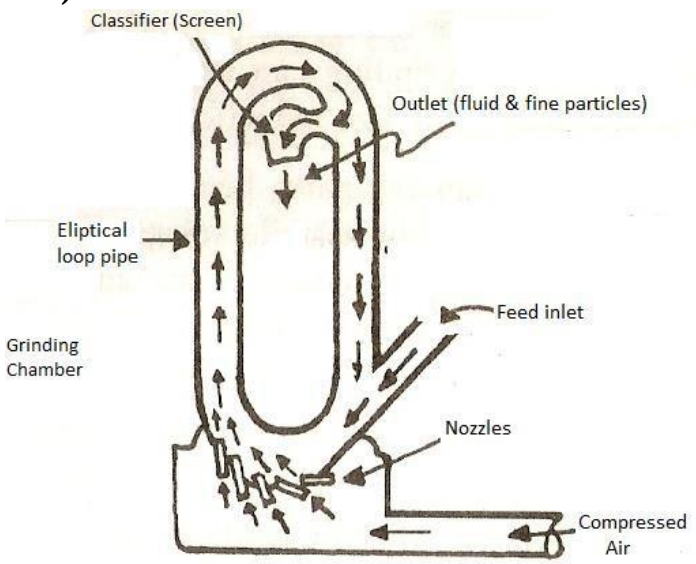
1. It is very noisy machine.
2. It is batch process.
3. It is slow process.
4. Thermolabile, Soft and fibrous materials cannot be milled by ball mill.
5. Wear occurs from the ball as well from casing, which cause contamination of product.

Variants- Hardinge mill, Tube mill, Rod mill (use rod instead ball) and continuous ball mill**.**

FLUID ENERGY MILL OR JET MILL OR MICRONIZERS OR ULTRAFINE GRINDERS

Principle- Impact and attrition **(**between materials and materials**)**

- ❖ Materials are suspended within a high velocity air stream.
- ❖ Milling takes place due to high velocity collisions between the suspended particles.



Construction:

- ❖ It consists of an elliptical or loop of pipe.
- ❖ Height of pipe about two meters.
- ❖ Diameter of pipe is 20 to 200 millimeters.
- ❖ It is made of stainless steel or tough ceramics
- ❖ Nozzles are placed bottom of pipe or opposed to the initial flow path of a powder.
- ❖ Compressed air is used at 600 kilopascals to 1.0 megapascals.
- ❖ Inert gases are used to minimize the oxidation of compounds.
- ❖ Feed inlet is fitted in the path of the airflow.
- ❖ An outlet is fitted upper side of pipe.
- ❖ An outlet attached with classifier (cyclone separator or bag filter) is to allow the escape of air.

Working

Materials placed through the feed inlet → Air/fluid is pass through nozzles at high velocity

↓

Materials thrown outward and circulate in grinding chamber

↓

Size of materials reduces
(Due to Impact and attrition **b**etween materials and materials)

↓

Reduced particle separate through outlet (cyclone separator)

U

se a) It is used to reduce the particle size of the drugs such as antibiotics and vitamins.

s: b) Ultrafine grinding can be achieved.

c) Moderately hard materials can be processed for size reduction.

Advantages:

a) It has no moving parts hence heat is not produced during milling.
b) Due to the expansion of gases, cooling effect is produced during milling.
c) Heat-labile substances can be milled like sulphonamides and vitamins.
d) substances can be milled like Griseofulvin (an antifungal drug) and antibiotics
e) It is a rapid and an efficient method.
f) Less chance of contamination.

Disadvantages:

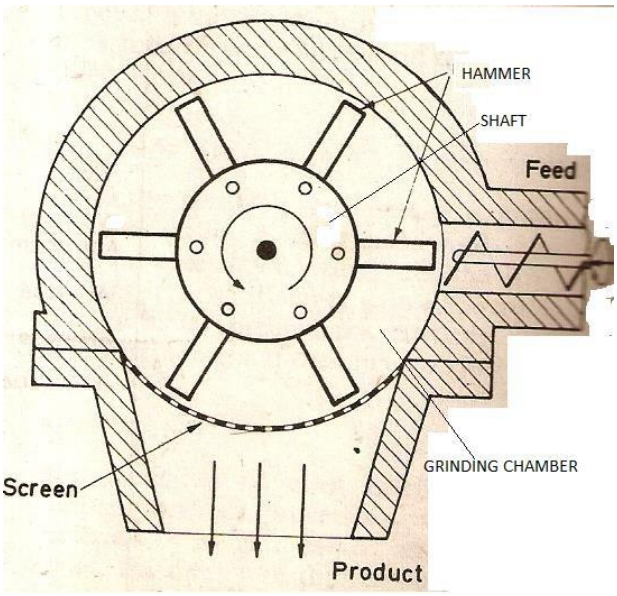
a) It is not suitable for soft, tacky and fibrous materials.
b) Expensive equipment (it needs additional accessories like fluid energy source and dust collector).

HAMMER MILL

Principle- Impact **(b**etween moving hammer and materials**)**

Construction

- ❖ It consist stout metal casing with central shaft.
- ❖ Shaft is rotated it own axis.
- ❖ Four or more hammers are attached with central shaft.
- ❖ Hammers are mounted with swivel joints.
- ❖ Hammers are also rotates.
- ❖ Hammers may be square faced, tapered or stepped form.
- ❖ Lower part of casing consist screen or mesh.
- ❖ Screen help in size separation of materials.
- ❖ Feed inlet placed side of casing.



Working

Materials load in chamber → Rotates the hammer → Rotating hammer beat the materials

↓

Size of materials reduces
(Due to impact between hammer and materials)

↓

Reduced particle pass through mesh/screen

Use- it is used for brittle materials and dry materials.

Advantages

- ❖ Easy installation.
- ❖ Various types of material can be used.
- ❖ Less space required.
- ❖ Continuous operation.

Disadvantages

- Screen may be clogged.
- It generates the heat due to high speed of operation.
- It cannot be used for sticky and fibrous materials.
- Heat-labile or thermolabile substances cannot be used.

Variants- Hammer crusher, vertical impact pulverizer

EDGE RUNNER MILL

Principle: - Impact & shearing

- It consist mortar made up of Steel or granite
- It consist two heavy rollers which rotate on the bed.
- Rollers made of iron or granite.
- Rollers are mounted vertically on horizontal shaft.
- Each rollers revolves it own axis, while both travel round shallow steel base bed.
- Outer part of roller travel greater distance than inner part of roller.
- Scrapers ensure that material is constantly returned to the grinding area.
- Generally rollers are rotate but some time bed base (mortar) is made to rotae.

Working: -

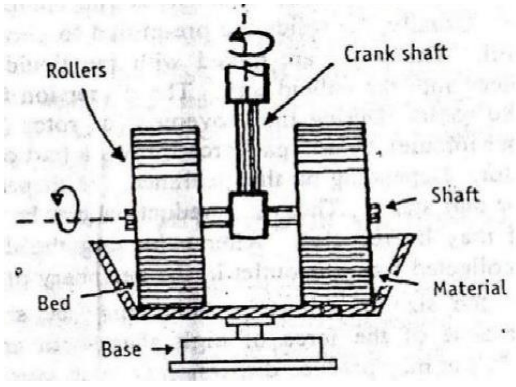
- Material is fed into the machine.
- Rollers revolve on it axis and create impact & shear.
- Size reduction is achieved.
- Material is continuously scraped from the sides of the mortar with knife.
- Resulting materials passed through sieve to get fine powder

Advantage

- Used for tough and fibrous materials.
- Wet and dry grinding.
- Very fine particles are obtained.

Disadvantages

- It cause noise pollution
- Chance of contamination.



END RUNNER MILL (Chilean mill, Putty chaser)

- It is mechanical mortar and pestle

Principle: Crushing due to weight of roller and shearing (friction).

Construction: It consists of mechanical mortar and pestle. which can rotate at high speed.

- ❖ It consists of mortar made up of steel which is fixed to a flanged plate.
- ❖ Pestle is dumb-bell shaped.
- ❖ Mortar is rotated by motor.
- ❖ Pestle rotates itself by friction
- ❖ Pestle can be raised from the mortar to facilitate emptying and cleaning.
- ❖ Scraper is also present which force the materials to grinding area.

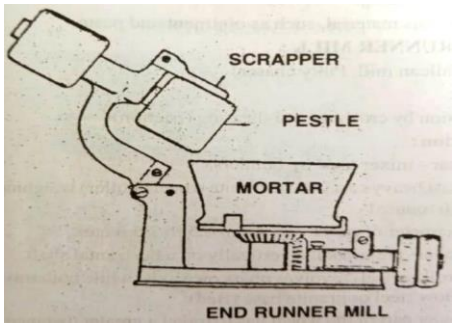
Working:

- ❖ The material placed in the mortar.
- ❖ The mortar revolves at a high speed.
- ❖ The revolving mortar causes the pestle to revolve and size reduction is achieved.
- ❖ Resulting materials passed through sieve to get fine powder.

Uses: suitable for fine grinding

Advantage

- ❖ To grind fibrous materials (bark, woods leaves, etc).
- ❖ Used for wet grinding
- ❖ Used for very viscous material (ointments and paste).
- ❖ It give moderately fine particles are obtained



Disadvantages: not suitable for drugs which are unbroken or slightly broken condition.

SIZE SEPARATION

- It is unit operation**.**
- It involves the separation of mixture into fraction of know particle size by means of screening surface.
- Size separation is also known as **sieving, sifting, classifying or screening**.
- It is based on physical differences between particles like size, shape and density.
- During size separation those particles remains on screen surface is known as **oversize**.
- During size separation those particles pass trough screen is known as **undersize.**

Application/objective

- Determination of particle size for production of tablet and capsule.
- It is a quality control tool for analysis of raw material.
- To improve mixing of powders
- To improve the solubility and stability of particles during production
- To optimize the process condition such as method of agitation, time of screening, feed rate etc.
- To measure the efficiency of size reduction equipments

OFFICIAL STANDARDS FOR POWDERS

- In general, powders are described as coarse and fine powders.
- Indian Pharmacopoeia has prescribed standards for powders for pharmaceutical purposes.
- Degree of coarseness or fineness is expressed by size of sieve/mesh through which powder is able to pass.
- The I.P-2007 specifies following grades of powder like

S. No.	Grade of powder	Sieve through which all particles must pass	Sieve through which not more than 40 per cent of particles pass
1.	**Coarse powder**	10	44
2.	**Moderately coarse powder**	22	66
3.	**Moderately fine powder**	44	85
4.	**Fine powder**	85	120
5.	**Very fine powder**	125	Not specified
6.	**Micro fine**	A powder of which not less than 90 per cent by weight of the particles pass through a sieve with mesh aperture of 45 μm	
7.	**Superfine powder**	A powder of which not less than 90 percent by number of the particles are less than 10 μm in size	

a) **Coarse powder** - 10/44 mm or 1700 μm (1.7mm)/355 μm
b) **Moderately coarse powder**- 22,/ 66 mm or 710/250 μm
c) **Moderately fine powder**- 44/85 mm or 355/180 μm
d) **Fine powder**- 85/120 mm or 180 μm
e) **Very fine powder**. 125μm/45 μm.
f) **Microfine powder**. A powder of which not less than 90 per cent by weight of the particles pass through a sieve with a nominal mesh aperture of 45 μm.
g) **Superfine powder**.

Coarse powder: A powder, all the particles must pass through a sieve No. 10 and not more than 40 % through sieve No. 44 is called coarse powder.

OR

A powder, all the particles of which pass through a sieve with a nominal mesh aperture of 1700 μm and not more than 40 per cent by weight through a sieve with a nominal mesh aperture of 355 μm.

SIEVES

- Sieves are used for pharmacopeial testing and size separation.
- Sieves are constructed like wire cloth and make square meshes.
- Sieves are woven from wire of brass, bronze, stainless steel or any suitable materials.
- Sieves should not be coated or plated.
- Material of the sieve cannot be reacting with the substance to be sieved.

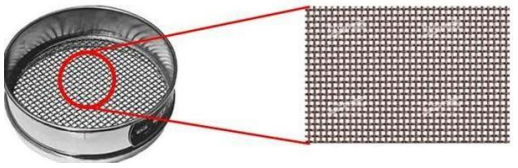
Sieve number- it is number of meshes in a length of **2.54 cm** in each transverse direction parallel to wires

Types of Sieves

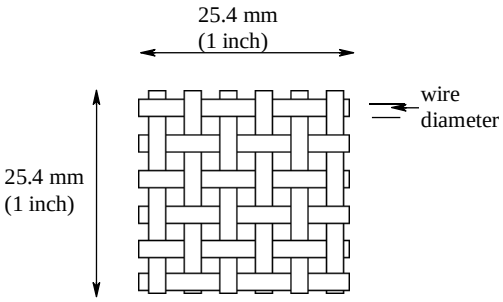
- The primary considerations for sieves are given to the size and shape of aperture opening.
- Square meshes are arranged as per the specifications.
- Sieves commonly used in pharmaceutical processing include
  - **Woven wire sieves**
  - **Bolting cloth sieves**
  - **Closely spaced bars (screens)**
  - **Punched plates**

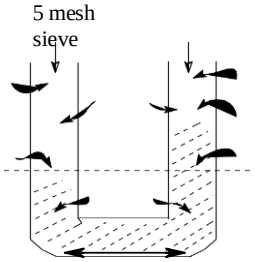
Standards of Sieves, Dimensions and Notations- Common standards used for sieves are:

(a) Tyler standard sieve series (in USA)
(b) British standard sieve series (in UK)
(c) IP standard sieve series (in India)
(d). International test sieve series (ISO) (World wide)



- Sieves used for pharmacopoeial testing must match with the following specifications:
  a) **Number of sieve**: number of meshes per linear length of 25.4 millimetres.
  b) **Nominal size of aperture**: it indicates the distance between the two adjacent wires.
  c) **Nominal diameter of the wire**: Wire having the specified diameter in order to give a suitable aperture size and sufficient strength to avoid distortion of the sieve.
  d) **Approximate percentage sieving area**: It expresses the area of the mesh as a percentage of the total area of the sieve. It depends on the size of the wire used for any particular sieve.
  e) **Aperture tolerance average size**: Some variation in the aperture size is unavoidable. This variation is expressed as a percentage and is known as the aperture tolerance average.





Tyler Standard Screen Scale

Mesh	Clear Opening, mm	Wire Diameter, mm
3	6.680	1.778
4	4.699	1.651
6	3.327	0.914
8	2.362	0.813
10	1.651	0.889
14	1.168	0.635
20	0.833	0.437
28	0.589	0.318
35	0.417	0.310
48	0.295	0.234
100	0.147	0.107
150	0.104	0.066

METHODS OF SIZE SEPARATION

- Screening is a method of separating particles according to size alone.
- The basic technique involved is passing the particles through a series of sieves of uniform size.
- The particles drop through the openings due to gravity.
- Coarse particles can drop easily through large openings, but it is difficult to screen fine powders.
- Size separation can be increased by inducing some type of motion (movement) to the particles.
- Size separation is basically assisted by **three methods**.
    1. **By sieving (Sifting)- Agitation, Brushing**
    2. **Centrifugal force-** Cyclone separator , air separator
    3. **Wet sieving (sedimentation/elutriation)**

1. Agitation Method- Sieves are agitated in a number of ways like

a) **Oscillation**: (shaking screen)
- The sieve is mounted in a frame that oscillates **back and forth (reciprocal motion)**.
- It is a simple method, but the material may roll on the surface of the sieve.
- The motion is parallel to the plane of the sieve.
- The reciprocating motion is induced by means of an ordinary eccentric on a rotating shaft.

b) **Vibration**:
- The sieve is vibrated at high speed by means of an eccentric device either electrically or mechanically.
- Rapid vibration is imparted to the particles that help the powder to pass through the sieve.
- A vibration also prevents blinding of meshes.

c). Gyration:
- It gives a rotary movement to the sieve.
- A system is made so that sieve is on to an eccentric flywheel
- Small amplitude to the sieve, which in the particles that helps to pass them
    (a) Gyrations in horizontal plane
    (b) Gyrations in vertical plane
- Gyratory screens are box like equipment, either round or square, with a series of screen cloths nested atop one another
- Normally, all the three modes of agitation are used simultaneously for an efficient size separation.

Advantages:
a) Agitation methods are inexpensive.
b) Simple and rapid.

Disadvantages:
a) If the powder is not dried, apertures become clogged with particles leading to improper sieving.
b) During agitation, attrition (particles colliding with each other) occurs causing size reduction.

2. Brushing Method
- In this case, a brush is used to move the particles on the surface of the sieve.
- This method keeps the meshes clear.
- The brush is rotated in the middle in the case of a circular sieve, but spiral brush is rotated on the longitudinal axis in case of horizontal cylindrical sieve.
- This is used for size separation of greasy or sticky powders such as waxes and soaps.

3. Centrifugal Method
- In this method, a high-speed rotor is fixed inside a vertical cylindrical sieve.
- On rotation the particles are thrown outwards by centrifugal force.
- The currents of air can be generated by means of a jet of air into the equipment.
- Air helps in separating the particles.

Examples - cyclone separator and air separator

Advantages:

- It used in cases of conventional sieving tends to block the sieves.
- Extremely useful for fine powder, because sieves have the limitation of mesh size.

4. Wet sieving- it is done by either sedimentation or elutriation methods.

4. Equipment used for size separation

Following equipments is use for size separation

i. **Sieve set with sieve shaker**-(Agitation Method)
ii. **Cyclone separator** (Centrifugal Method)
iii. **Air separator** (Centrifugal Method)
iv. **Sedimentation tank** (Wet sieving)

MECHANICAL SIEVE SHAKER

- Powder pass through number of sieve which oscillate back and forth (**Agitation)**

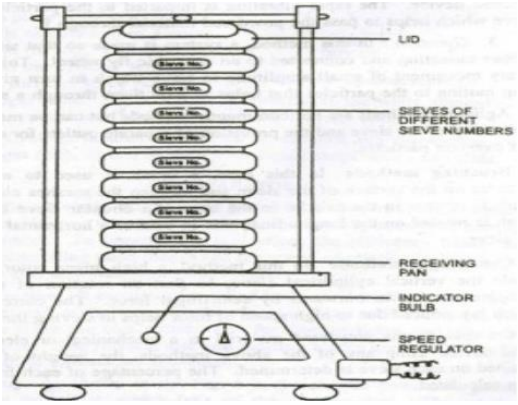
Sieve details

- It consist Set of sieves
- Arranged in ascending order
- Large size sieve at the top
- Small size at the bottom
- Receiver at the bottom of the small size sieve

Sieving procedure

- Powder kept in top sieve.
- Shake the sieve.
- Sieve gyrates and vibrates.
- Weight of powder on each sieve.
- Find the percentage of residue in each sieve

$$\text{Percentage} = \frac{\text{Residue in each sieve}}{\text{Total wt of powder}} \times 100$$



Advantages

- Size analysis data under controlled condition
- Inexpensive
- Simple & Rapid process
- Little variation between operation

Disadvantages

- Over loading sieve result error
- Insufficient time leads to wrong results
- Electrostatic attractions leads to aggregation
- Humidity, Hygroscopic powders leads to aggregation
- Pale like or long fibrous will not pass

CYCLONE SEPARATOR

Principle

- **Centrifugal force** is used to separate solid from fluids.
- Separation of particles depends on particle size and particle density.
- It is also possible to allow fine particles to be carried with the fluid.

Construction

- It consists of a short vertical, cylindrical vessel with a conical base.
- The upper part of the vessel is fitted with a tangential inlet.
- The solid outlet is at the base.
- Fluid outlet is provided at the center of the top portion, which extends inwardly into the separator.
- Such an arrangement prevents the air short-circuiting directly from the inlet to the outlet of the fluid.

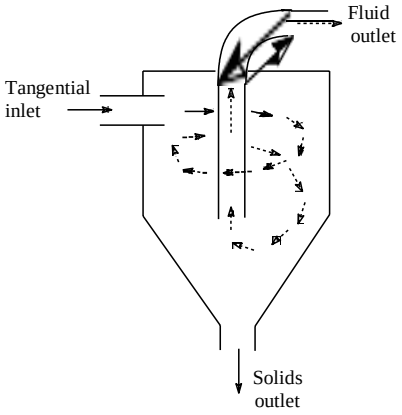


Fig. Cyclone separator

Working

- Suspension of solid and air introduce tangentially at very high velocity.
- Rotary moment of suspension is developed due to high speed/velocity.
- High speed producing the vertex and develop centrifugal force.
- The centrifugal force throws the particles to the wall of the vessel
- The fluid (air) can escape from the central outlet at the top.
- Coarser particles separates and fall through outlet
- Fine particles are allowed to be carried with fluid
- Separation of particles depends on the fluid velocity

Uses

- It is used to separate solid particles from gases/air.
- It is also used for size separation of solids in liquids.
- It is used to separate the heavy and coarse fraction from fine dust.

SEDIMENTATION METHOD (WET SIEVING)

- This method is used when particles are too small to be screened properly or when there is difference in settling rates of particles of different sizes.
- The methods of particle size separation are depending on sedimentation or elutriation.
- When particles are settled independently of one another, this condition is called free settling.
- A stage comes when acceleration of particles become zero and particles get settle at constant velocity.
- This velocity is called terminal settling velocity.
- When particles are so close and continuously collide with each other. This cause pushing of lighter particles by heavy particles. This is called Hindred settling.
- Stoke's law is not applicable in case of hindered settling.
- Size separation by sedimentation utilizes the differences in settling velocities of the particles with different diameter (d) and these can be related to Stoke's law.

Stoke's law

- When a solid particle is suspended in a liquid the particle settles downward at a velocity, V.
- This velocity is called sedimentation rate.
- It is found that this rate of sedimentation depends on the
  - Diameter of the particle
  - density of the liquid and particle
  - viscosity of the liquid
  - Acceleration due to gravity.
- All this parameters can be combined in the form of **Stoke's equation**:

$$V = \frac{d^2(\rho_1 - \rho_2)g}{18\eta}$$

Where d = diameter of the particle
ρ_1 = density of the particle
ρ_2 = density of the liquid
g = acceleration due to gravity
η = viscosity of the liquid.

Continuous Sedimentation Tank

- A shallow tank is arranged with inlet and outlet pipes.
- Particles entering the tank, two force act on particles

(i) **Horizontal force-** due to the flow of liquid carrying the particles forward.

(ii) **Vertical component-** due to gravity.

- ❖ This causes the particles to fall towards the bottom of the tank.
- ❖ It is governed by Stoke's law.

- **Coarsest** (largest) particles will settle near to the **inlet of liquid**.
- **Finest** (smaller) particles will settle near to the **outlet of the liquid**.

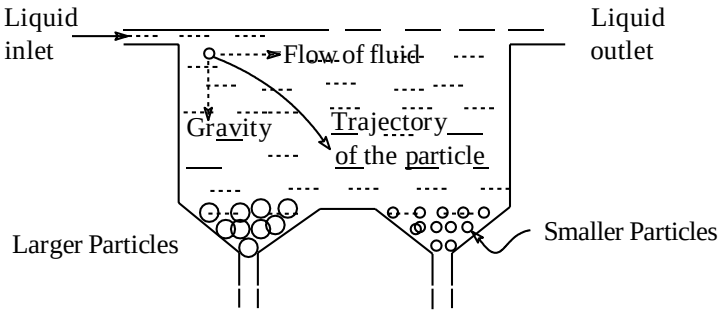


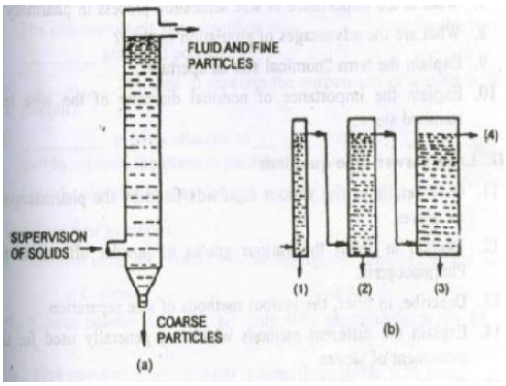
Fig. Sedimentation Tank

ELUTRIATION

- Elutriation is a process for separating particles based on their size, shape and density.
- Gas or liquid flowing in a direction usually opposite to the direction of sedimentation.
- This method is mainly used for particles smaller than 1 μm.
- In elutriation movement of fluid against the direction of sedimentation of the particle

Apparatus

- It consist of a vertical column
- An inlet near the bottom
- An outlet at the base for coarse particle
- An overflow near the top for fluid and fine particles.
- **One column will give single separation in two fractions**.
- For further fractions the number of tubes of increasing area of cross section is connected in series.
- Velocity gradient across the tube results in the separation of particles of different sizes
- The size separation of powers is based on the low density of fine particles and high density ofcoarse particles.
- Elutriation tank is used to separate the coarse and fine particles after levigation.
- The dry powder or paste made from levigation process is mixed with large quantity of water andmade suspend in the tank.
- Depending on density of particles they will settle down or suspended in water.
- The sample is drawn from different heights through outlets and dried.
- Thus the powder with various size fractions is obtained.
- The fractions are separated and dried.



Advantages:

- The process is continuous
- The separation is quick.
- The apparatus consists of vertical columns.

AIR SEPARATOR

Principle:

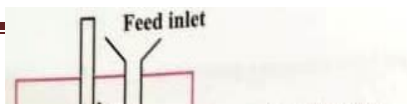
- Centrifugal force (air movement is obtained by means of rotating disc and blades)

Construction:

- It consists of vertical metal cylindrical vessel with conical base at the bottom,The feed inlet is fitted tangentially at the upper part of vessel. The outlet for collected solids is at the base of conical portion where as fluid outlet is at the centre of the top portion.
- The fluid outlet pipe extends down below inlet section to avoid air short-circuiting directly from the inlet into the outlet.
- The rotating disc and rotating blades are fitted on shaft is placed at the center of the vessel.
- It has two separate outlets at bottom for finer and coarser/heavy particles.

Working:

- The feed (powder) enters the centre of the vessel through the feed inlet.
- The feed falls on the rotating blade and the rotating disc.
- The rotating disc produces an air jet in the direction indicated in the diagram.
- The fine particles are collect by the air stream and brought into the space of the settling chamber, where the air velocity is sufficiently reduced so that the fine particles fall out



and are eliminated through the fine particle outlet.
- Particles too heavy to be collected by air stream are removed at the outlet of coarse particles.

Advantage
- Ease of installation
- Rotor speed is adjustable
- High product capacity
- Air flow can be adjust and easy to maintain.

Uses:
- It is attached to ball mill or hammer mill to separate and returnoversized particles for further size reduction.

Application
- It is used for separation of dry powders in micron-sized that couldnot be separated by traditional sieves.

BAG FILTER

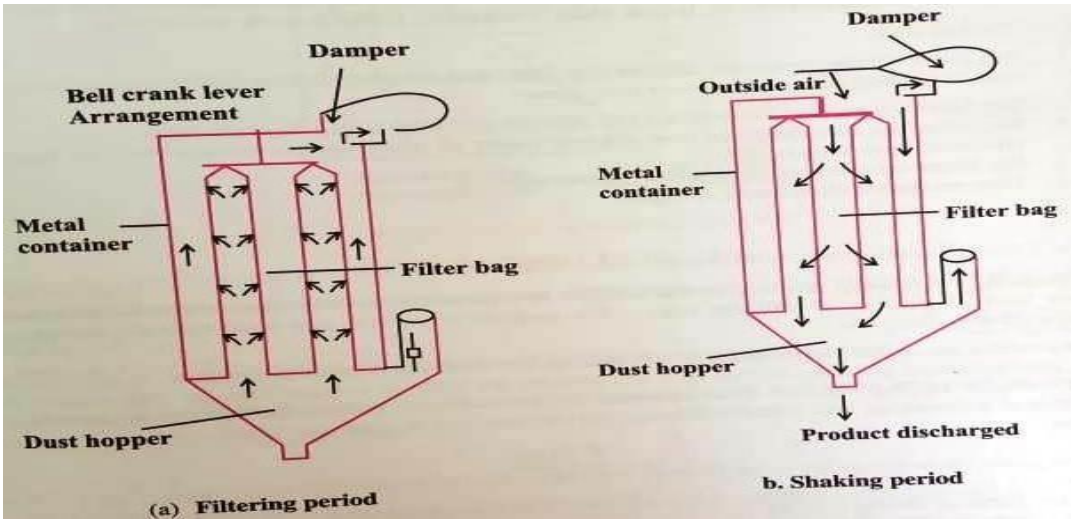
Principle
- The separation is performed in two stages.
- **First stage**- gas-containing dust is passed through a bag (cloth), by applying suction.
- **Second stage-** bags shaken by applying pressure so that the powder adhering to the bag falls andcollected from base.

Construction
- A series of bags (made of cotton or wool fabric) are suspended in a metal container.
- In the bottom portion, hopper is arranged to receive the feed.
- A bell-crank lever arrangement is provided at the top of the vessel to change the filtering action toshaking.

Working:
- During this period the gas containing dust enters through the hopper.
- Then it is passed inside the bags and at the top of the apparatus.
- The vacuum fan produces a pressure below atmospheric pressure inside the apparatus.
- As a result, the particles get trapped within the bags.

Shaking period:
- Bell-crank lever rotates and changes the position of damper.
- The outside air enters through the top in the metal casing and therefore the vacuum is broken.
- At the same time, it causes violent shaking or jerking action to the bags.
- Dust or fine particles are displaced from the bags.
- Maximum portion of dust falls into the hopper which is collected from the conical base.

Uses
- Bag filters are also commonly referred to as fabric dust collectors that are used in large industrialunits to separate dust particles from dusty gases.
- Bag filters are the most efficient and cost effective type of industrial dust collectors.
- Bag filters are considered the most efficient among all dust collectors because they can reach anefficiency level of up to 99%.
- Bag filters are also used along with a cyclone separator.
- They are used to clean the air of a room


Damper
Bell crank lever
Arrangement
Metal
container
Filter bag
Dust hopper
(a) Filtering period
Damper
Outside air
Metal
container
Filter bag
Dust hopper
Product discharged
b. Shaking period

EVAPORATION

Definition

Evaporation means simply vaporization from the surface of the liquid. Evaporation is an unit operation by which a solvent is evaporated from a solution by boilingthe liquor in a suitable vessel and withdrawing the vapor, leaving a concentrated liquid residue.

Objective of evaporation:

To make a solution more concentrated. Generally extracts are concentrated in this way.

Factors affecting evaporation:

(i) Temperature:

(ii) Temperature and time of evaporation

(iii) Temperature and moisture content

(iv)Type of product required

(v)Effect of concentration

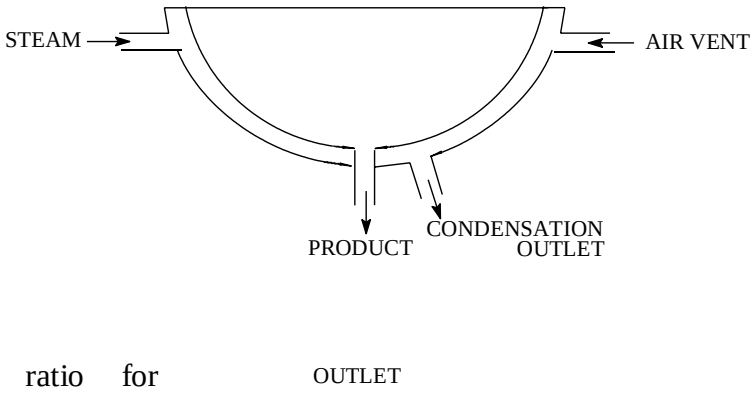
Natural Circulation Evaporator

EVAPORATING PAN

Construction

The apparatus consists of a hemispherical, or shallow pan, constructed from a suitable materialsuch as copper or stainless steel and surrounded by a steam jacket.

The hemispherical shape gives the best surface/ volume ratio for heating, and the largest area for separation of vapor.



The pan may have a mounting , permitting it to be tilted to remove the product, but the shallow form makes this arrangement somewhat unstable, and an outlet at the bottom, is common.

Working

- The dilute solution is taken in the pan.
- Steam is introduced through the steam inlet into the jacket to heat the pan.
- In these evaporators the movement of the liquid results from convection currents set up by the heating process.
- The concentrated liquid is collectedthrough the outlet placed at the bottom of the pan.

Advantages:

It is simple and cheap to construct.
It is easy to use, clean and maintain.

Disadvantages:

- Having only natural circulation, the overall coefficient of heat transfer will be poor and olids are likely to deposit on the surface, leading to decomposition of the product and a further deterioration in heat transfer.
- Also many products give rise to foaming.
- The total liquor is heated over all the time, which may be unsatisfactory with thermolabile materials.
- The heating surface is limited and decreases proportionally as the size of the panincreases.
- The pan is open, so the vapor passes to the atmosphere, which can lead to saturation of the atmosphere.
- Only aqueous liquids can be evaporated in these pans.
- Pan evaporation cannot be done under reduced pressure.
- Can only be used for thermolabile products.

Horizontal Tube Evaporator:

Horizontal tube evaporator frequently found to be the most adaptable choice for simple evaporation wherein liquids are not viscous and do not deposit scale or salt on surface.

Principle:

The principle mechanism involved in this type of evaporator is that steam is passed through tubes arranged horizontally. Heating causes evaporation of the feed outside the tubes discharging concentrate at the bottom and vapours passed out from the outlet at top.

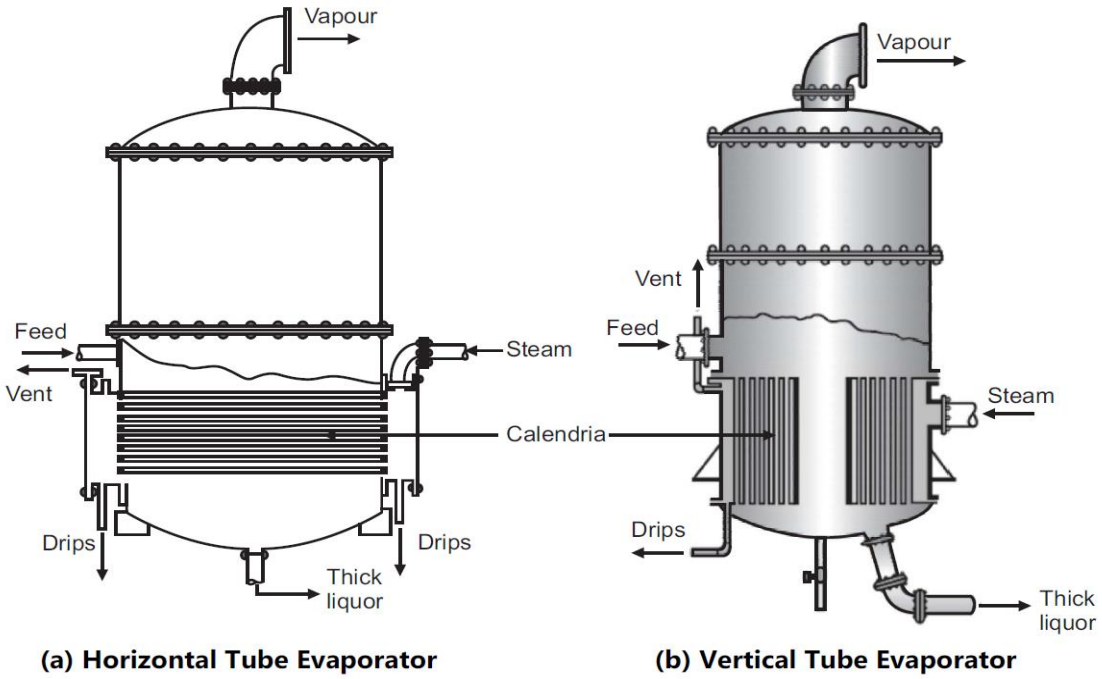
The vapour is removed from the top of the chamber and the product circulation take place by natural circulation over the heating coil.

Construction:

Horizontal-tube evaporators are designed with either rectangular or circular crosssections, with tubing of stainless steel, aluminium, nickel, carbon, spellerized iron pipe, leadcovered copper, or special bronze, Fig. 5.2 (a). The tubes are extended between two steam chests and arc is fastened to tube. Four-hole packing plate's force down conical gaskets around the tube ends into counter-sunk holes in the tube sheets. Secure sealing is obtained,

with facility for quick and easy renewal. Horizontal tubes of 2 to 3 cm diameter are extended across the bottom of a cylindrical chamber with 1 to 3 meters diameter and 2.5 to 4.5 meter height. In case of vertical tube evaporators the tubes are arranged vertically in calendria, Fig. 5.2 (b). A calendria is a heating part in

evaporator consisting of large number of smaller diameter tubes wherein liquid is concentrated while rising or falling.



(a) Horizontal Tube Evaporator (b) Vertical Tube Evaporator

Working:

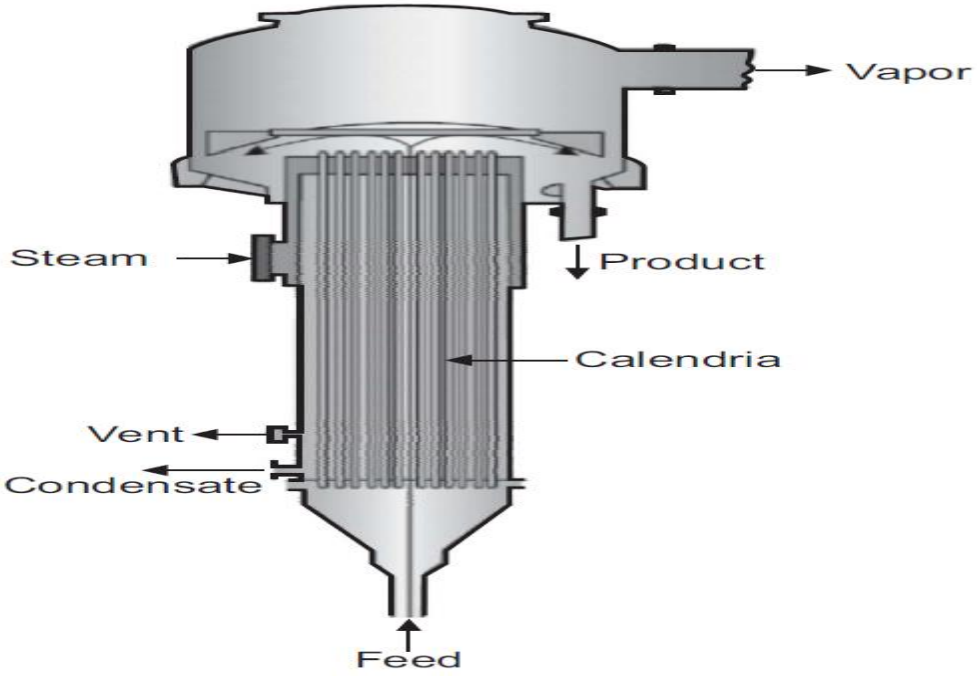
In the horizontal-tube evaporator, steam is fed into steam chest and is directed through the horizontal tubes to heat the liquid surrounding the tube in the bottom of the evaporator body. The definite path followed by the steam assures that all non-condensed gases and condensate are swept to the opposite steam chest, where they are withdrawn. The velocity and paths of circulation of the liquid depend upon the distribution, size, anti shape of the heating surface in the liquid compartment.

Advantages:

(i) A number of units can be joined to obtain more efficient effect.
(ii) It has low cost per unit of heating surfaces.
(iii) It has extreme simplicity.
(iv) Easy renewal of heating surfaces.
(v) Sectional construction with low maintenance cost.
(vi) Ease of operation.
(vii) Ability to carry large volume of liquor in the body.
(viii) It requires low headspace.
(ix) Small cargo space required for shipment.

Disadvantages:

(i) Cleaning and maintenance is difficult when compared with steam jacketed kettle.
(ii) During operation the pressure inside the evaporator increases that reduces the effective temperature gradients and may affects heat sensitive materials.
(iii) It may be used only when rigorous boiling can be obtained with natural circulation.
(iv) It is not suitable for viscous liquids.
(v) Since the boiling liquid is outside of the tubular heating surface, it is not easily cleaned by mechanical means.
(vi) It is not suitable when scaling or salting liquids are involved.



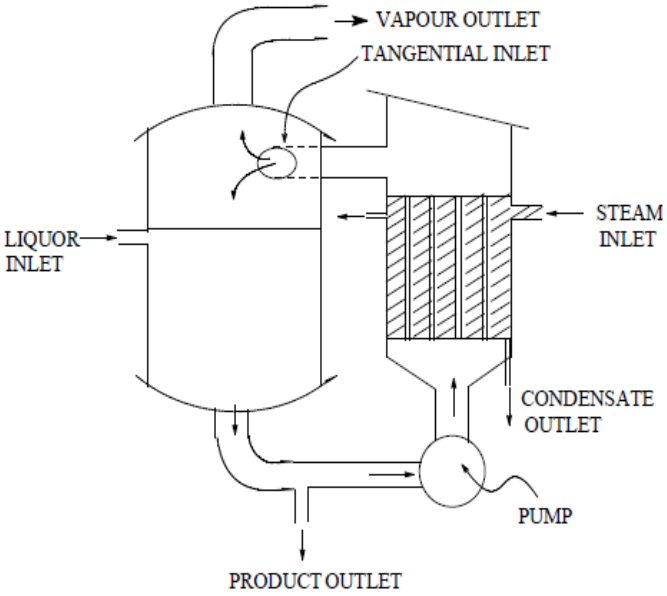
FORCED CIRCULATION EVAPORATORS

Forced circulation evaporators are natural circulation evaporators with some added form of mechanical agitation. Different forms of forced circulation evaporators can be designed.

- An evaporating pan, in which the contents are agitated by a stirring rod or pole could be described as a forced circulation evaporator.
- A mechanically operatedpropeller or paddle agitator can be introduced into an evaporating pan or still.
- Propeller or paddle agitatorcan be introduced into the downtake of a short-tube evaporator
- A typical forced circulation evaporator can be shown as follows:

Construction

The evaporator consists of a short tube calendria and a large cylindrical vessel (body of the evaporator) for separation of vapor and liquid takes place. The liquor inlet is provided at the side of the cylindrical vessel. A pump is fitted in between the calendria and the body of the evaporator. A tangential inlet for liquid under high pressure is placed at neck of the body of the evaporator. The vapor outlet is placed at the top of the body and it may be passed through a condenser to collect the condensed liquid.



Working Principle

Feed is introduced through the liquor inlet. Pump will force the liquid through the calendria. Steam heats the liquid inside the calendria. As it is under pressure in the tubes the boiling point is elevated and no boiling takes place. As the liquor leaves the tubes and enters the bodyof the evaporator through the tangential inlet there is a drop in pressure and vapor flashes off from the superheated liquor. The concentrated liquid is pumped out through the product outlet and the vapor is collected through the vapor outlet.

Advantages

- Rapid liquid movement improves heat transfer, especially with viscous liquids or materials that deposit solids or foam readily.
- The forced circulation overcomes the effect of greater viscosity of liquids when evaporated under reduced pressure.
- Rapid evaporation rate makes this method suitable for thermolabile materials, e.g. it is
- used in practice for the concentration of insulin and liver extracts.

FILM EVAPORATORS

Film evaporators spread the material as a film over the heated surface, and the vaporescapes the film.

Long tube evaporators (Climbing film evaporators)

Construction and working principle

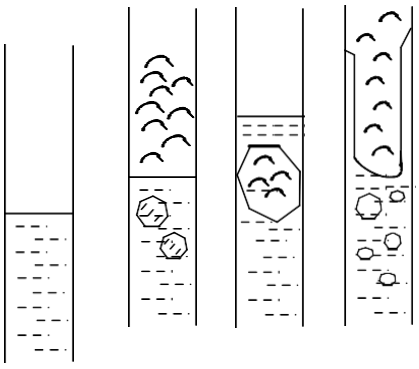
The heating unit consists of steam- jacketed tubes, having a length to diameterratio of about 140 to 1, so that a large evaporator may have tubes 50 mm in diameter and about.

The liquor to be evaporated is introduced into the bottom of the tube, a film of liquid forms on the walls and rises up the tubes, hence it is called climbing film evaporator.

At the upper end, the mixture of vapor and concentrated liquor enters a separator, the vapor passes to a condenser, and the concentrated liquid to a receiver.
Cold or pre heated liquor is introduced into the tube (fig.-i).

Heat is transferred to the liquor from the walls and boiling begins, increasing in vigor (fig.-ii).

Ultimately sufficient vapor has been formed for the smaller bubbles to unite to a large bubble, filling the width of the tube and trapping a 'slug' of liquid above the bubble (fig.-iii).

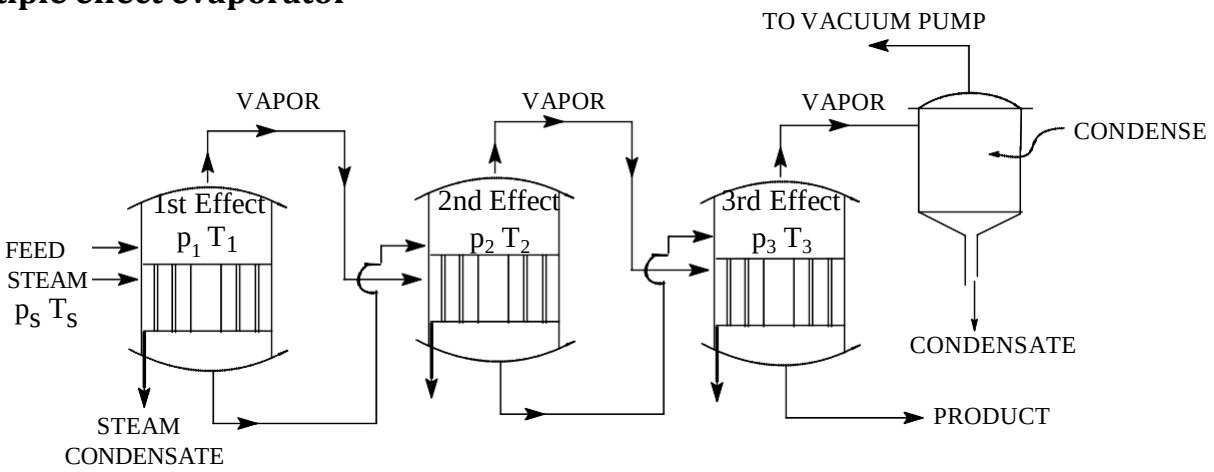


(i) (ii) (iii) (iv)

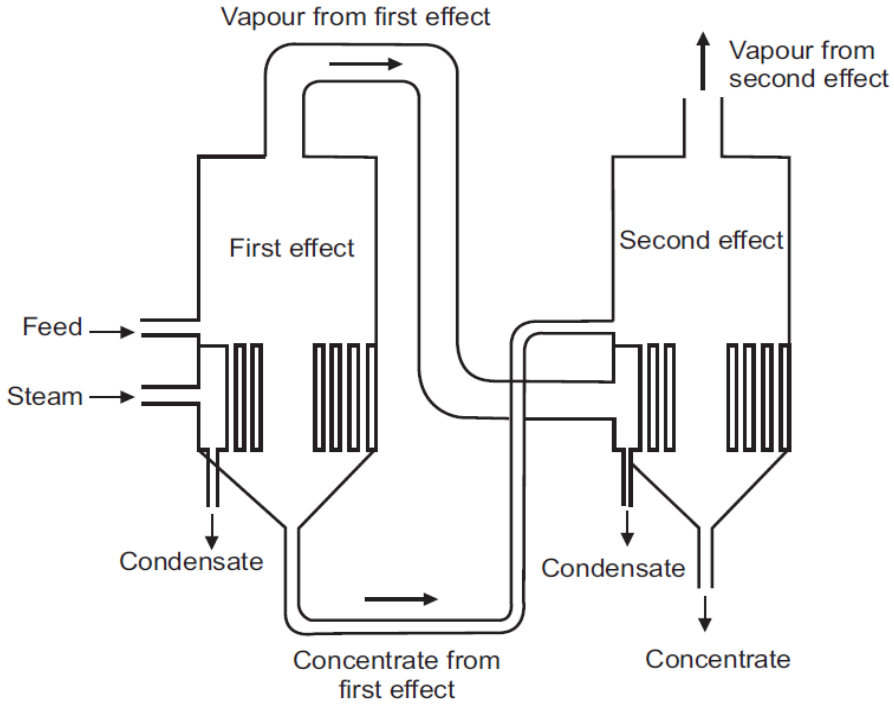
As more vapor is formed, the slug of liquid is blown up the tube (fig.-iv),

the tube is filled with vapor, while the liquid
continues to vaporize rapidly, the vapor escaping up the tube and, because of friction betweenthe vapor and liquid, the film also is dragged up the tube upto a distance of 5 to 6 metres.

Multiple effect evaporator



Triple-effect evaporator: p_s, p_1, p_2, p_3 vapor pressures, Ts, T1, T2, T3 temperatures where $p_s > p_1 > p_2 > p_3$.



In a single effect evaporator steam is supplied for heating the liquor. The total heat is not transferred form the steam. So the rest of the heat is wasted. To use that heat efficiently, connections are made so that the vapor from one effect serves as the heating medium for the next effect.

(i) The dilute feed (liquor) enters the first effect, where it is partly concentrated; it flows to the second effect for additional concentration and then to the third effect for final concentration. This liquor is pumped out of the third effect.

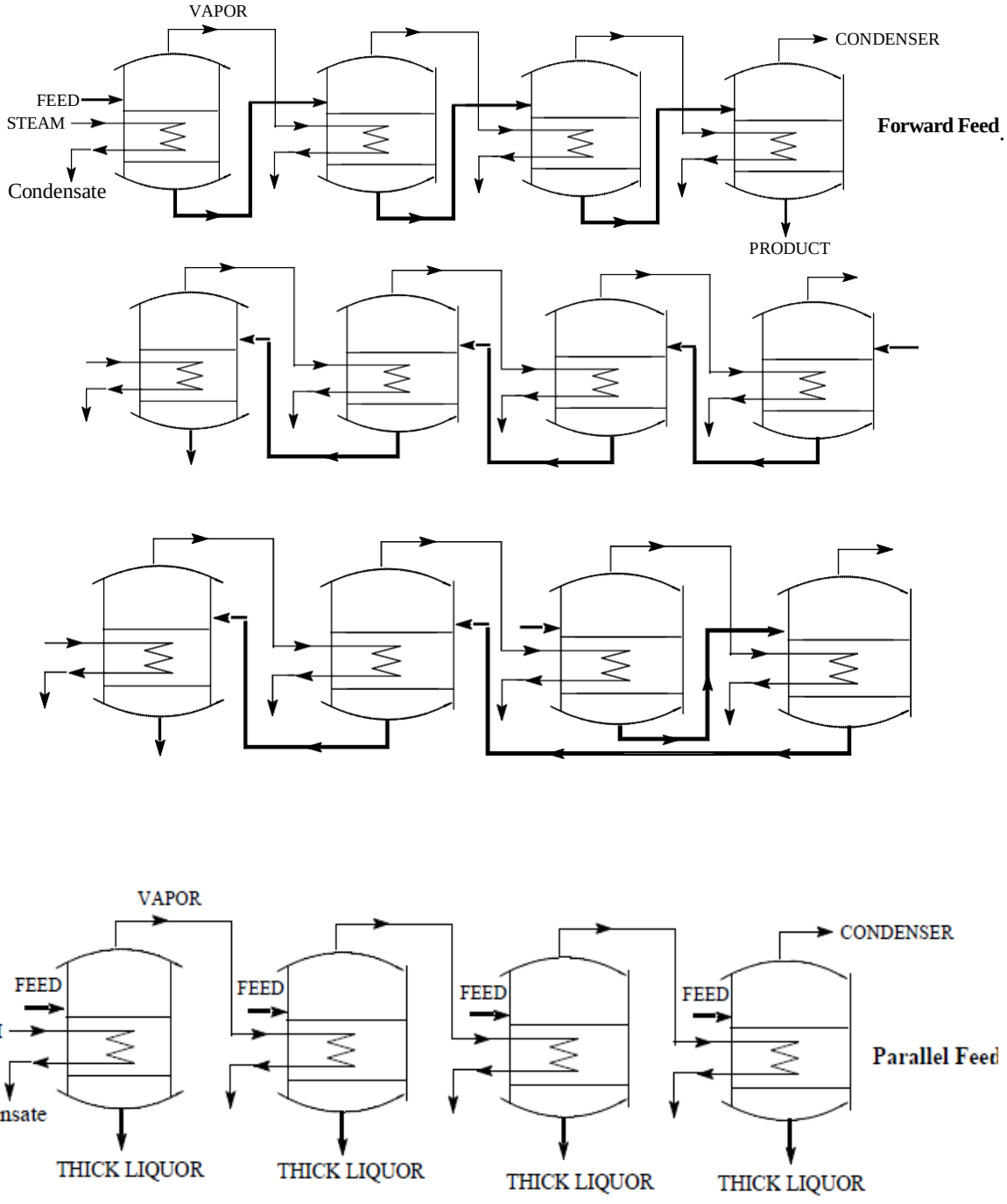
(ii) In the first effect raw steam is fed in which the vapor pressure in the evaporator is the highest, p_1. the second effect has the intermediate vapor pressure; i.e. $p_1 > p_2 > p_3$. This pressure gradient is maintained by drawing the vapor through a vacuum pump and condensing after the final effect.

(iii) Depending on the lowering of vapor pressure boiling point of the liquids of 2nd and 3rd effect will also be lowered; i.e. $T_1 > T_2 > T_3$.

(iv) In the 2nd effect vapor from the 1st effect (T_1) is heating the liquor (having temperature

T_2). So there is a temperature gradient ($T_1 - T_2$); consequently the liquor will be heated. Similar heating will be there in the 3rd effect also.

Methods of feeding



Advantages:

1. Feed moves from high pressure (in effect-2) to low pressure (in effect – 4) chambers, so pumping of liquor is not required.
2. Product is obtained at lowest temperature.
3. This method is suitable for scale-forming liquids because concentrated product is subjected to lowest temperature.

Disadvantages

It is not suitable for cold feed because, the steam input in effect-1 raises the temperature of the feed, and a small amount of heat is supplied as latent heat of vaporization. Therefore, amount of vapor produced will be less than the amount of steam supplied. Lower amount of vapor in effect-1 produces lower amount of vapor in the subsequent effects. Therefore, the overall economy is lower.

Backward feed

In backward-feed the feed enters in the last effect and moves towards first effect (i.e IV → III → II → I).

Advantages

It is suitable for cold feed, because the heat used for increasing the temperature in IV effect is already used for heating 3 times. This will give more economy.

The method is suitable for viscous products, because highly concentrated product is at highest temperature, hence lower viscosity (⟶ higher heat transfer ⟶ higher capacity)

Disadvantages

The liquid moves from low-pressure (IV) to high-pressure chambers (III ⟶ II ⟶ I) pumpingis requierd.

Mixed feed method

The feed enters in the intermediate effect, moves forward and then backward to effect-I (III ⟶ IV ⟶ II ⟶ I).

Advantages

- Liquid moves from high pressure (III) to low pressure (IV), hence no pump is required. Liquid moves from IV ⟶ II ⟶ I requires pump.
- Product is obtained from highest temperature (I) hence lowest viscosity.

Parallel feed

It is suitable where the feed has to be concentrated slightly.

ECONOMY OF MULTIPLE EFFECT EVAPORATORS

Assumptions: (a) Feed is at boiling point and
(b) Loss of heat is negligible

In effect-1

1 Kg of steam transfers its heat to feed. Since feed is at boiling point so the total amount of heat is used as latent heat of vaporization. Therefore, 1 kg steam will produce 1 kg vapor.

In effect – 2

1 Kg vapor from effect-1 will transfer heat to the liquor of effect -2. Here also 1 kg vapor produce 1 kg vapor from the liquor.

In effect – 3

1 Kg vapor from effect-II will produce 1 kg vapor in effect-3.

Therefore, 1 kg steam will produce 3 kg vapor.

Now, economy of a single effect evaporator = $\frac{vapor\ produced}{steam\ used} = \frac{1kg}{1kg} = 1$

And economy of a triple effect evaporator = $\frac{vapor\ \ produced}{steam\ used} = \frac{3kg}{1kg} = 3$

So for N number of effects economy will be N times that of a single effect evaporator

DISTILATION

Distillation may be defined as the separation of the constituents of a mixture includinga liquid by partial vaporization of the mixture and separate and collect the vapor.

Such separation may include

(i) one liquid from non-volatile impurities.

(ii) one liquid from one or more other liquids, with which it may be miscible, partially-miscible or immiscible

N.B.

In practice it is difficult to distinguish between evaporation, distillation and drying.Based on the intention:

(i) when condensation vapor is required the operation is called distillation

(ii) when the concentrated liquid residue is required the operation is called evaporation.

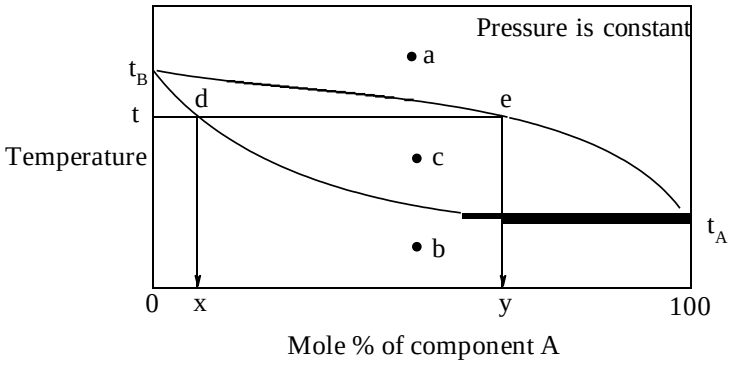
(iii)when the dried solid residue is required as product the process is called drying

BOILING POINT DIAGRAM OF A BINARY MIXTURE

The figure represents the boiling point and equilibrium-composition relationship, at constant pressure.

Two liquids A (b.p. t_A) and B (b.p. t_B) are taken in a chamber of constant pressure. Now at any temperature the vapor composition and liquid composition will give two lines when plotted vs. temperature.

In boiling point diagram, temperatures are plotted as ordinates and compositions as abcissas.



- The diagram consists of two curves, the ends of which coincide with the b.p. of two components (t_A and t_B).
- The upper-curve describes vapor composition and lower-curve liquid composition.
- At any temperature t the horizontal line cuts the vapor composition curve at ‘e’ which corresponds to vapor composition of y (mole%A) and cuts the liquid composition curve at ‘d’ which corresponds to liquid composition of x (mole% of A). So any two points on the same horizontal line (such as d and e) represent compositions of liquid and vapor in equilibrium at temperature ‘t’.
- For all points above the top line (such as point ‘a’) the mixture is entirely vapor.
- For all points below the bottom line (such as point ‘b’) the mixture is completely liquefied.
- For all points between the two curves (such as point ‘c’) the system consists partly of liquid and partly of vapor.

DISTILLATION METHODS

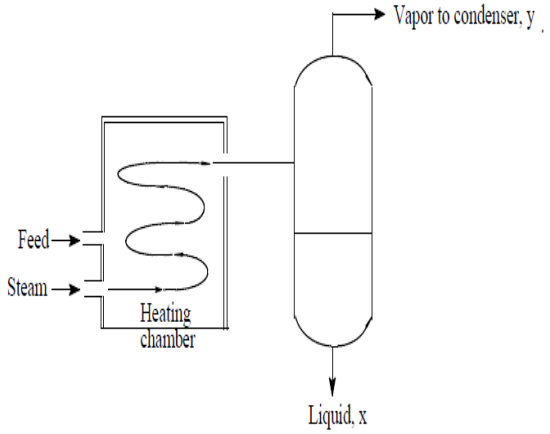
A. Distillation methods for miscible liquid systems

1. **Equilibrium or Flash Distillation**
2. **Simple or Differential Distillation**
3. **Fractional Distillation**
4. **Distillation under reduced pressure (e.g. Molecular Distillation)**
5. **Special Distillation Methods for non-ideal mixtures**
   (a) Distillation of Azeotropic Mixtures
   (b) Extractive Distillation

(B) Distillation of immiscible liquids (e.g. Steam Distillation)

1. EQUILIBRIUM DISTILLATION / FLASH DISTILLATION

There are two types of distillation that do not involve rectification



(a) Equilibrium distillation or flash distillation and

(b) Simple or differentialdistillation.

(a)Equilibrium distillation or flash distillation

This is a single stage operation where a liquid is partially vaporized, the vapors are allowed to come in equilibrium with the residual liquid and the resulting vapors and liquid are separated. *Use*: This method is used only when the difference between volatilities of two components is very large

Let us consider a binary system whose components are A and B. A is more volatile.

- Feed: W_F = number of moles of liquid fed
  x_F = mole fraction of component A in feed
- Suppose V moles are vaporized in an equilibrium-distillation process.

Now in

Liquid phase — Number of moles left in liquid phase = $(W_F - V)$ moles
Let the composition of the residual liquid = x mole fraction of A

Vapor phase — Composition of vapor phase = y mole fraction of A
Number of moles gained = V

Form material balance equation with respect to A

Moles of A at start = Moles of A in vapor phase +Moles of A in liquid phase

$$W_F x_F = V y + (W_F - V) x$$

In this case all the parameters are known except x and y.

2nd equation required for solving is obtained fromequilibrium curve of the A, B system.

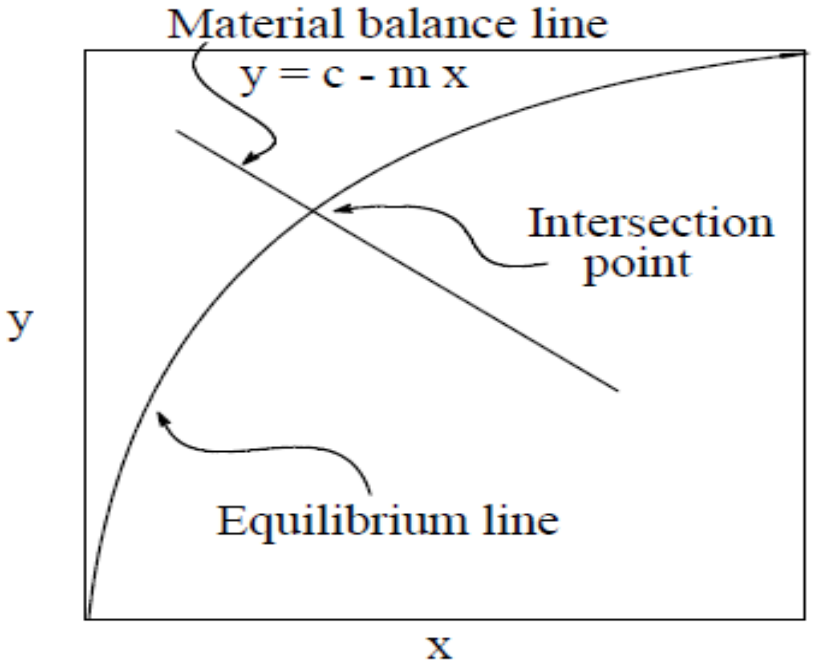
Eqn(i) is a straight line,

$$V\,y = W_F\,x_F - (W_F - V)\,x$$

$$\text{or,} \quad y = \left(\frac{W_F\,x_F}{V}\right) - \left(\frac{W_F - V}{V}\right) x$$

$$\text{or,} \quad y = c - m\,x$$

Plotting this equation in the equilibrium curve the point of intersection is obtained. The value of x and y can be obtained form the point of intersection.



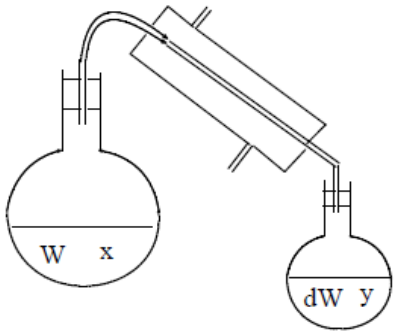
Equilibrium curve of A-B system

1. SIMPLE / DIFFERENTIAL DISTILLATION

In this process vapor is removed from the system assoon as it is formed and condensed.

***Use*:**

- This method is commonly used in laboratory
- In industries it is only used for systems having high relative volatilities.



Derivation of Raleigh's equation

Let us consider a batch of W_0 moles of liquid was taken at the beginning.
Suppose at any given time during distillation there are W moles of liquid left in the still.
At this time let the mole fraction of A in liquid is *x*.
Suppose a very small amount of liquid dW is vaporized. In the vapor phase the mole fraction of component A is *y*.

	At a given time	After a moment
Total moles of liquid present	W	W – dW
Moles of A present in liquid	W*x*	(W – dW)(*x* – d*x*)
Total moles of liquid removed		dW
Moles of A present in the vapor		*y*

Therefore a material balance equation with respect to A will be
xW = (W – dW) (x – dx) + ydW
xW = xW – xdW – Wdx + dWdx + ydW
dWdx is very small hence ignoring the term the equation will be
ydW – xdW = Wdx
or, (y – x)dW = W dx

or, $\frac{dW}{W}=\frac{dx}{y-x}$

Now, integrating between the limits

	Time = 0	Time = t_1.
Amount of liquid in the still (moles)	W_0.	W_1.
Moles of component A in liquid	x_0.	x_1.

$$\int_{W_0}^{W_1}\frac{dW}{W}=\int_{x_0}^{x_1}\frac{dx}{y-x} \qquad \text{or,} \qquad \boxed{\ln\frac{W_1}{W_0}=\int_{x_0}^{x_1}\frac{dx}{y-x}}$$

This equation is known as Raleigh's equation. It relates the amount of material distilled with instantaneous composition of the liquid at that moment
The function

$$\frac{dx}{y - x}$$

can be integrated graphically from the equilibrium curve, since the curves gives the relationship between x and y.

N.B.

x	y	y – x	1/(y – x)
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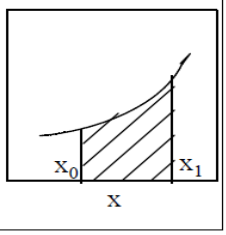
$$\frac{1}{y - x}$$



Application of Raleigh's Equation

1. By using the Raleigh's equation the effectiveness of simple distillation for a given system can be estimated.
2. It is used in determination of cut-off point when we can stop distillation as soon as the vapor composition falls below the required purity of the product.

SIMPLE DISTILLATION

Objective

Simple distillation is the process of converting a liquid into its vapors which, are passed through a cooling surface to condense the vapors. The condensed vapors are reformed into liquid which, is collected in a receiver.

Apparatus for laboratory scale

It consists of a distillation flask with a side arm sloping downward that is connected to a condenser. The condensed vapors are collected in a flask called '*receiver'*. The whole apparatus is made of glass.

A thermometer is fitted in the distillation flask to note down the temperature at which, the vapors are distilled.

Bumping is avoided by adding small pieces of porcelain or porous pot before distillation.

Apparatus for preparation of purified water

The boiler may be made of cast iron but the baffles and the condenser tubes that comes into contact with product are made of stainless steel or monel metal.

The cold water from the water tap enters the still through the inlet, which rises in the jacket fitted with a constant level device, the excess of water over flow through the outlet. A portion of hot water at 90 to 95^{0}C enters into the boiler through a narrow opening –

the level of water is maintained in the boilerup to overflow level.

The water is boiled in the boiler by means of heating coils. On heating, the dissolved gases in the condenser are allowed to escapethrough a small opening and only the steam

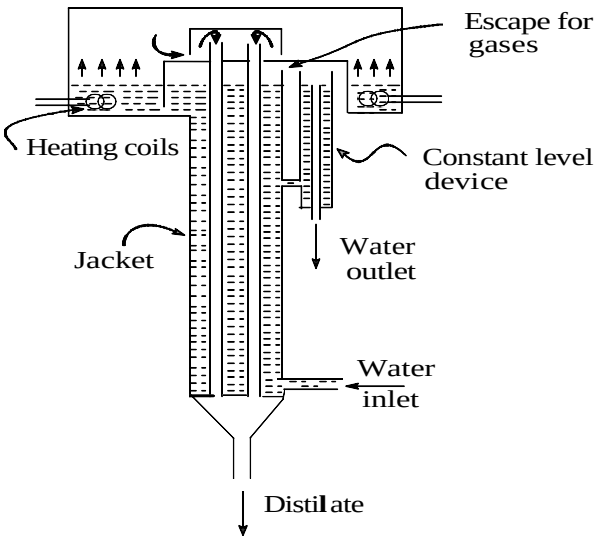


Fig. Distil ation unit for purified water

escapes into the condensing tubes.

Since the dissolved gases are more volatile than water they escape in the first portion of the distillate, therefore, must be rejected. Similarly, the last portion may contain volatile portion of the dissolved solid substances in tap water – hence, discarded.

Application of simple distillation in pharmacy

I. It is used for the preparation of distilled water and water for injection.

II. Many volatile oils and aromatic waters are prepared by simple distillation e.g. Spirit of nitrous ether and Aromatic Spirit of Ammonia

III. Concentration of liquid and to separate non-volatile solid from volatile liquids such as alcohol and ether.

3.FRACTIONAL DISTILLATION / RECTIFICATION

A rectifying unit consists primarily of

(a) a <u>still</u> or <u>reboiler</u>, in which vapor is generated,

(b) a <u>rectifying or fractionating column</u> through which this vapor rises in counter-current contact with a descending stream of liquid, and

(c) a <u>condenser</u>, which condenses all the vapor leaving the top of the column, sending part of this condensed liquid (the reflux) back to the column to *descend* counter to the rising vapors, and delivering the rest of the condensed liquid as product.

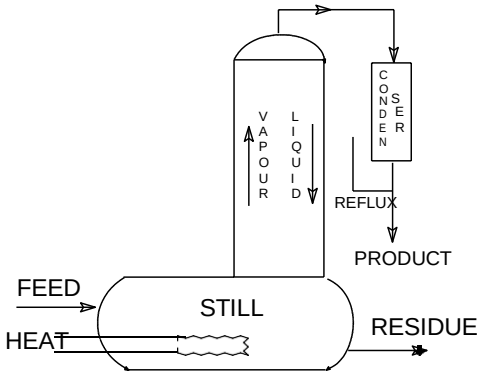


Diagram of still and fractionating column

As the liquid stream descends the column, it is progressively enriched with the less volatile constituent.

The top of the column is cooler than the bottom, so that the liquid stream becomes progressively hotter as it descends and the vapor stream becomes progressively cooler as it rises. This heat transfer is accomplished by actual contact of liquid and vapor, and for this purpose effective contact is desirable.

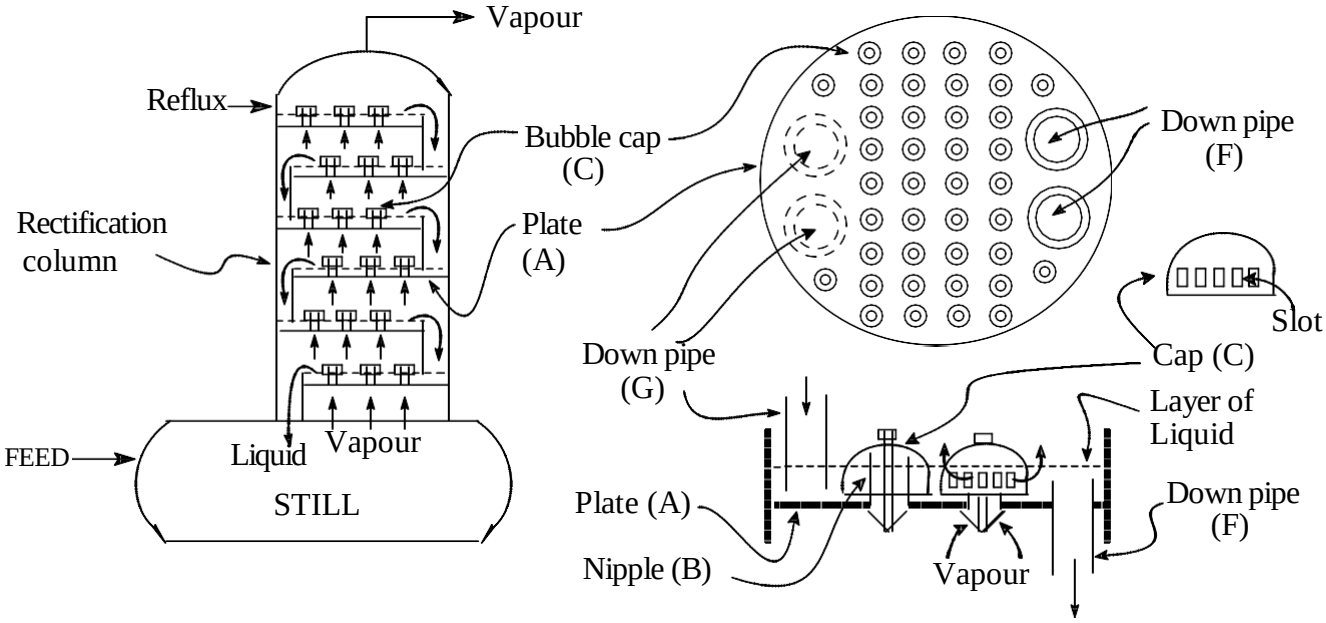
CONSTRUCTION OF RECTIFYING COLUMN

There are different varieties of equipments for rectification

(a) Plate column (i) Bubble cap column
(ii) Sieve-plate column

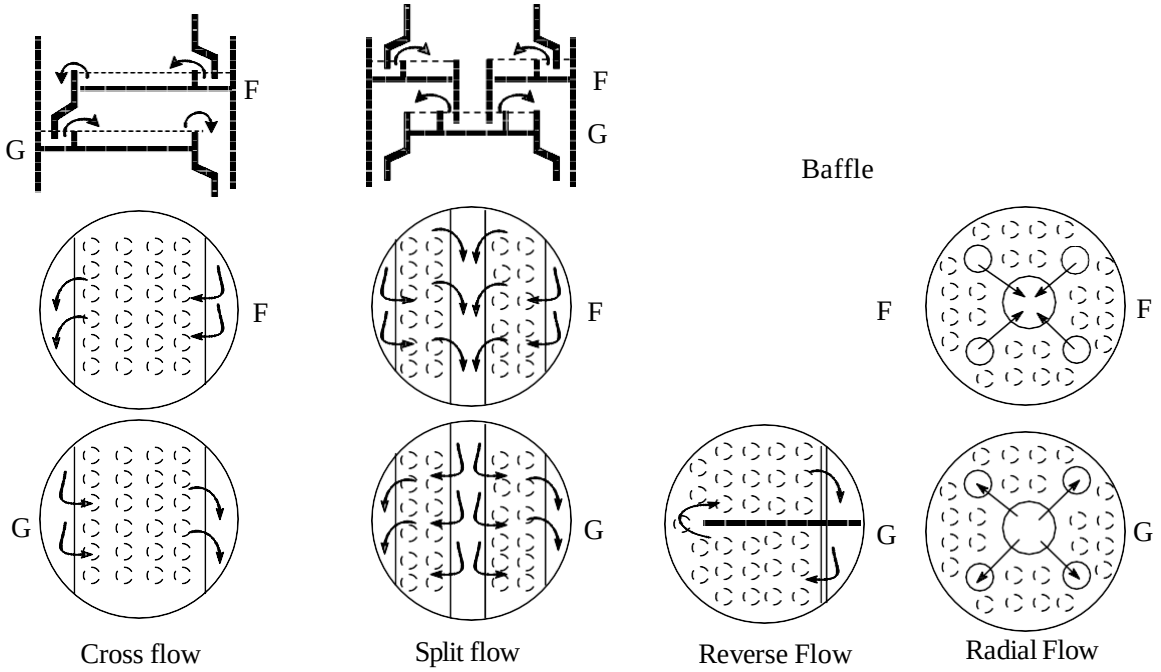
(b) Packed column

BUBBLE-CAP COLUMN



- The column is divided into sections by means of a series of *horizontal plates* A.
- Each plate carries a number of short *nipples* B (or *riser*). Each nipple is covered by a bell-shaped *cap* C that is secured by a spider and bolt with the plate. The edge of the cap is *serrated* or the sides may be *slotted*.
- Vapor rises from the plate below through the nipple, is diverted downward by the cap, and bubbles out under the serration or through the slots.
- A layer of liquid is maintained on the plate by means of an *overflow* or *down-pipe* (F) and the depth of the liquid is such that the slots are submerged.
- The *down-pipe*, (G) from the plate above, is sealed by the liquid on the plate below, so that the vapor cannot enter the down-pipe.
- Ordinarily, the liquid is delivered at one end of a diameter by the down-pipe from the plate above, flows the other end of the same diameter.

Types of down-comers



(a) Cross flow

The liquid flows across the plate from right to left on plate F and left to right on plate H and so on down the column.

(b) Split flow

On plate F the liquid flows form the two sides to the center. On plate H it flows from the center to the two sides and so on down the column. This arrangement is commonly known as split flow.

(c) Reverse flow

Liquid comes down the space on one side of the baffle and flows across the plate from right to left, around the end of the baffle, from left to right and down the space behind the weir. This arrangement is called reverse flow.

(d) Radial flow with circular down-take

One plate will have four or more down-comers around the circumference, and the next plate will have a down-comer at the center so that on the upper plate the flow is from the circumference towards center and on the next plate the flow is from the central down-take to the circumference.

Specification of bubble cap rectification column

Column diameter	2 to 15 ft	
Height	few feet to over 100 ft	
Bubble cap diameter	3 to 6 inches	
Slots in a 3 inches bubble cap may be	1/8 to 3/32	inch wide
	½ to 1	inch height

SIEVE PALTE COLUMNS

All the constructions are same as bubble cap columns. Instead of bubble cap plates, flat plates with a large number of relatively small perforations, drilled in them are used. These perforations are usually 3/16 to ¼ inch in diameter.

The velocity of the vapor through these holes is sufficient to produce the liquid running downthe holes.

PACKED COLUMNS

The column is entirely filled with some sorts of material that offers a large surface area supposedly wetted by the liquid.

A large variety of materials are used among which **Raschig rings** are popular. A Raschig ring is a hollow cylinder whose length is equal to its diameter. This may be made of metal (by sawing sections off a pipe), stone ware, ceramics, carbon, plastics, or other materials. Raschig rings are usually dumped at random in the column.

Advantages

(i) Have a low pressure drop per unit of height than bubble cap

(ii) For very small diameters of column, where it would be difficult to get in more than two or three bubble caps, a packed column can be used.

(iii) Since Raschig rings can be made of any material, hence packed columns can be used for corrosive materials.

(iv) The amount of liquid held up in the column is low so thermolabile liquid remains in contact with high temperature for a short time than bubble cap method.

Disadvantages

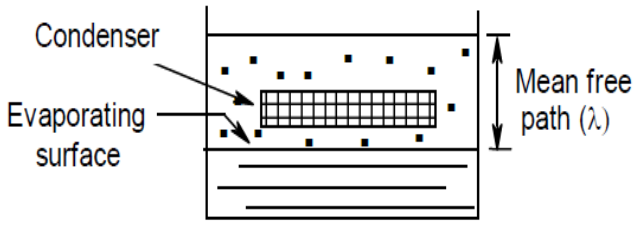
(i) They are relatively inflexible.

(ii) Distribution of liquid uniformly in such packed column is difficult. It is found that, as the liquid passes down the tower it tends to concentrate at the walls and leave the center dry.

3. DISTILLATION UNDER REDUCED PRESSURE / VACUUM DISTILLATION

Theroy

Liquid boils when its vapor pressure is equal to the atmospheric pressure. Liquids, which are decomposed at their boiling point under atmospheric pressure, can be distilled at a much lower temperature than its boiling point if the pressure is reduced on the surface of the liquid. Boiling under reduced pressure will also increase the rate of distillation.



Molecular Distillation

Theory / Principle of Molecular Distillation

In a high vacuum distillation operation, where the material distills from an evaporating

surface to a relatively cool condensing-surface. The conditions are such that, the mean free path of the distillating molecules is greater than the distance between the evaporating and condensing surface.

- The vacuum applied in these types of apparatus is about 1 □m Hg pressure or less.

Mean free path is defined as the average distance traveled by the molecules in a straight line without any collision. It can be calculated by Clausius law:

$$\lambda = \frac{1}{\sqrt{2\pi d^2 N}}$$

where, λ = mean free path (cm)

d = diameter of the molecules (cm)

N = number of molecules in 1 cm^3 volume.

N.B.

Temperature (^{0}C)	Volume (litre)	Pressure (mm Hg)	Number of molecules
0	22.4	760	6.023×10^{23}.
0	22.4	1×10^{-3}.	7.9×10^{17}.

It is clear from the above equation and chart that, mean free path can be increased, by reducing the number of molecules per cm^3 volume. The molecules evaporate from the surface and travel few cm without colliding with the molecules of the residual gas in the space above. If now the condensing surface is placed within distance, a major fraction of the molecules willcondense and will not return to the distilland. Thus each molecule distills itself and hence called "*Molecular Distillation*".

Characteristics of molecular distillation

1. Molecules having molecular weight within the range of 300 to 1100 dalton can be distilled by this method. [N.B. Low molecular weight (below 300 dalton) molecules will re-evaporate again from the condenser surface. High molecular weight molecules (greater than 1100 dalton) will not have sufficient volatility.]

2. The molecules to be distilled should reach the surface and evaporate. The molecules at the bottom of the distilland have to overcome the pressure of the layer above, to come to the surface. Hence, the layer should be thin and should be in a state of turbulent motion to facilitate the molecules to reach the surface.
3. The distilland should be degassed before entering in the still, because at very low pressure the dissolved gas will occupy all the space and rate of distillation will be reduced.

Falling Film Molecular Still

The vessel has a diameter of the order of 1 m and the walls are heated suitably by a heating jacket. Vacuum pumps are connected by a large diameter pipe. The feed flows down the walls and is spread to a film by the polytetrafluoroethylene (PTFE) wipers which move about 3 m/s giving a film velocity of about 1.5 m/s. The residue is collected at the bottom of the vessel and it is re-circulated (through the feed line).

The evaporated molecules are then condensed on the condenser surface. The condensate is taken out as product.

Centrifugal molecular still

The distilland (feed) is introduced on to the center of a bucket-shaped vessel (1 to 1.5 m in diameter) that rotates at high speed. The film of liquid that is formed moves outwards over the surface of the vessel to the residue- collection pipe. The vessel is heated by radiant heaters. Condensers and a collection device are located close to the inner surface of the rotor.

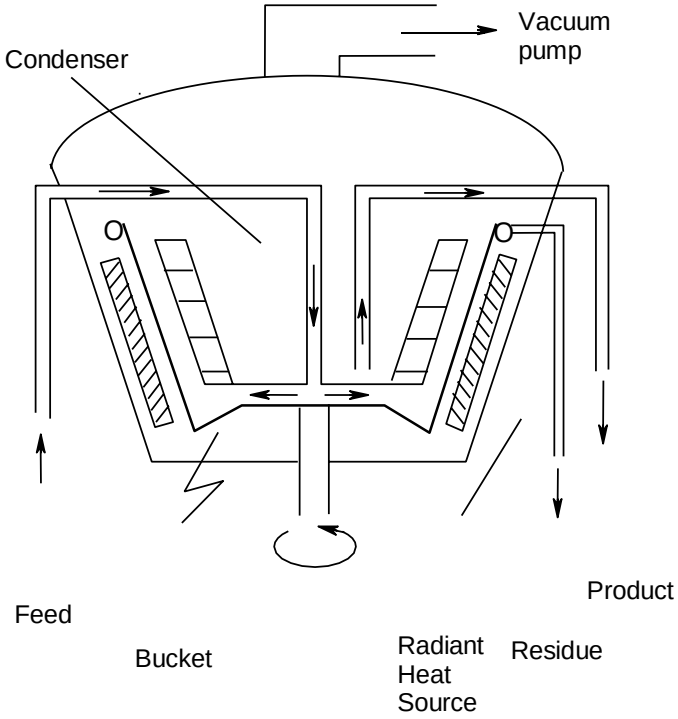


Fig. Centrifugal molecular stilll

Application of vacuum distillation in pharmacy

1. Vitamin concentrates

Vitamin A,D,E,K and tocopherols are obtained from vegetable and fish oils. The vitamin-A concentrate produced by molecular distillation is very pure and has good stability. As no chemical is used in this method which could split the ester linkage, the vitamins are retained in the natural ester form which is the most stable form of vitamin A. The stability of the concentrates is further enhanced by natural antioxidants distilling over from the original oil.

2. The fractionation of oil

The fractionation of oils into various components is carried out by molecular distillation.

Components	*Molecular Weight*	*Temperature Range*
(a) Fatty acids, unsaponifiable matter of low molecular weight.	150 – 300	50 – 140^{0}C
(b) Unsaponifiable matter like sterols, vitamins, dyes, waxy alcohols, monoglycerides	300 – 600	150 – 190^{0}C
(c) Triglycerides, sterol esters, vitamin esters,	600 – 900	Above 190^{0}C

Purification and fractionation of lanolin

It is used to get various fractions from Lanolin like, cetyl alcohol, cholesterol, ceryl alcohol, lanopalmitic acid, isocholesterol etc.

3. Separation of Poly Ethylene Glycol (PEG)

On laboratory scale it is used to separate PEG according to the degree of polymerization.

4. SPECIAL DISTILLATION METHODS FOR NON-IDEAL MIXTURES

Industrial scale distillation of Azeotropic Mixture

The liquor from fermentation process is a common source of ethanol and contains approximately 8–10% ethanol.

After simple distillation an azeotrope will form containing 95.6% (96E+4W) ethanol and boiling at 78.15^0C at atmospheric pressure.

In this type of system a reboiler is used instead of boiler. The feed liquor is introduced intothe system and must occur at a point where the equilibrium will not be disturbed. Hence, feedwill take place, at a place part of the way up the column, where the equilibrium composition on the plate is similar to the feed composition.

The plate below the *feed plate* form the stripping section where the rising vapor strips the more volatile component (ethanol) from the feed liquor while the upper section is known as the *rectifying section*.

The binary azeotrope produced at this stage is freed from water by making use of ternary azeotrope – ethanol, benzene, and water.

The ethanol/water azeotrope, with sufficient benzene (only required at start-up) is fed to column A and the pure ethanol is obtained as bottom product, since the ternary azeotrope takes off the water.

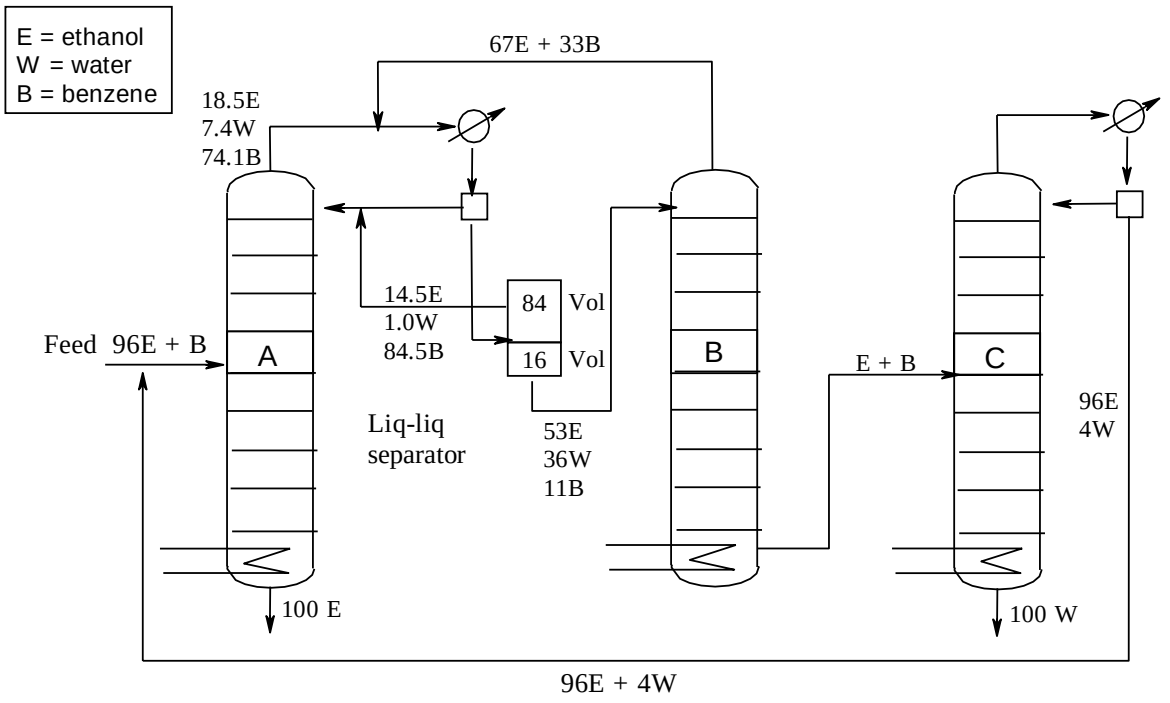


Fig. Plant for manufacture of Absolute ethanol (100% ethanol)

- The azeotrope (E+B+W) is taken from the top of the column A, condensed and separated (in liquid-liquid separator) into two layers, having the compositions given in the diagram.
- The upper layer predominates and, being rich in benzene (14.5E+1.0W+84.5B), is returned to column A. The lower layer (53E+36W+11B) is taken to column B, where the

benzene is recovered as the ethanol/benzene binary azeotrope (67E+33B) and is mixed with the vapor from ethanol.

- The ethanol / water residue passes to column C, where the ethanol is recovered as the ethanol/water binary azeotrope (96E+4W), which can be incorporated with the original feed.
- The final product from column A is 100% ethanol and from column C is 100% water.

5. DISTILLATION OF IMMISCIBLE LIQUIDS

Steam distillation

Steam distillation is used for the distillation of two immiscible liquids one of which is water.

Application:

(i) Separation of volatile oil e.g. eucalyptus oil, rose oil, clove oil etc. and

(ii) Preparation of some aromatic water e.g. concentrated rose water..

Theory

Volatile oils are mixtures of high molecular weight compounds having low vapour pressure (i.e. high b.p.). To separate these from the natural sources like petals of flowers, barks etc. it is not possible to take them to their boiling points around 200^0C. If these oils are distilled with water (low molecular weight but high vapour pressure i.e. low b.p.) then volatile oil will be distilled out at a temperature below 100^0C.

$$\frac{\text{Weight of volatile oil in distillate}}{\text{Weight of water in distillate}} = \frac{M_V P_V}{M_W P_W}$$

Where, M_W and M_V are molecular weights of water and volatile oil respectively.

P_W and P_V are vapor pressure of water and volatile oil respectively.

- The aqueous phase of distillate that is collected is water saturated with volatile oil i.e. called *aromatic water*.

N.B. When a mixture of two practically immiscible liquids are heated, while being agitated to expose the surfaces of both liquids to the vapor phase, each component independently exerts its own vapor pressure as a function of temperature as if the other constituent was not present.

Boiling begins and distillation may be effected when the sum of the partial pressures of the two immiscible liquids just exceeds the atmospheric pressure.

An immiscible liquid and water independently boils at high temperature but when steam is passed through a mixture of these liquids (agitation) it boils at a much lower temperature than the boiling point of water.

Example: Turpentine oil has a boiling point of about 160^0C, when mixed with water it can be distilled at about 95.6^0C if steam is passed through it.

Large scale apparatus

This consists of a still having a mesh near the bottom. The steam is generated by boilingwater below the mesh. The steam passes through the materials (to be extracted) packed over the mesh. The vapor containing volatile oil is then passed ot the condenser. The distillate is collected in *Florentine receivers*. Florentine receiver separates the oil and water depending on their densities. The aqueous phase may be re-circulated again to avoid loss of volatile oil in water.

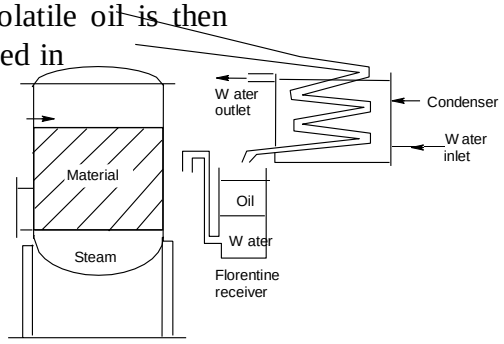


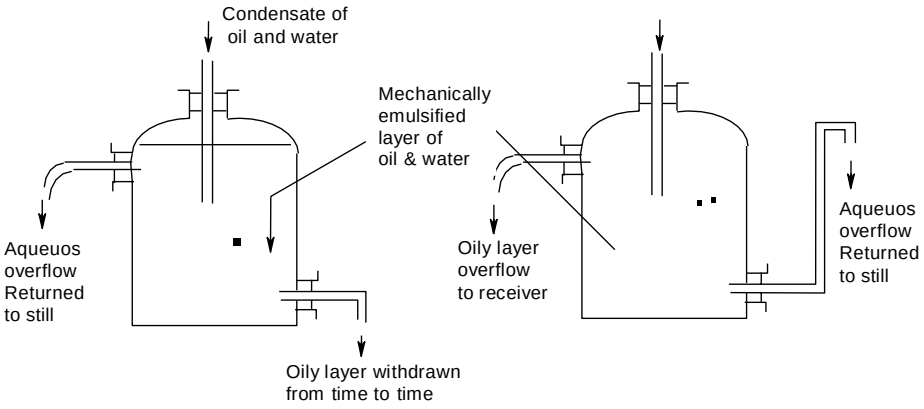
Fig . Steam distillaiton apparatus

Florentine Receiver

It is used for the separation of oil and water.Florentine receivers are of two types:

Type-I Used for separation of oil heavier than water.Type-II Used for separation of oil lighter than water.

Type-I receiver has tow taps. The tap fitted near the bottom of the vessel is used for collecting oil, whereas the tap fitted near the top of the vessel is used for water to overflow.



Type-II receiver is fitted with siphon at the bottom that works when it gets filled with water where as the tap

MIXING

The main objective of this mixing is to produce a bulk mixture which when divide intodifferent doses, every unit of divided doses must contain the correct proportion of each ingredient. It is critical process because the quality of the final product and its attributes are derived by the quality of the mixing.

APPLICATIONS OF MIXING

- Mixing is an intermediate step in production of tablet or capsule. Mixing of powders in different proportion prior to granulation or tableting.
- Dry mixing of materials for direct compression in to tablets.
- Dry mixing of powder or composites powders in capsule and insufflations respectively.
- Blending of powders are also important in preparation of cosmetic products such as facial powder or dental powder.
- In case of potent drugs where dose is low, mixing is critical factor. Otherwise it will affect content uniformity of tablet.

FACTORS AFFECTING MIXING

These factors include the following:

1. **Nature of product**: For effective mixing particle surface should be smooth.
2. **Particle size**: It is easier to mix powder of same particle size. Increasing the difference in particle size will lead to segregation.
3. **Particle shape**: Particle should be spherical in shape to get a uniform mixture.
4. **Particle charge**: some particle due to electrostatic charge exerts attractive force which leads to separation.
5. **Proportion of material**: It is easier to mix powders if available in same quantities.
6. **Relative density**: If the components have a different density, the denser material will sink through lighter material.
7. **Viscosity**: An increase in viscosity reduces the extent of mixing.
8. **Surface tension of liquids**: High surface tension reduces the extension of mixing.
9. **Temperature**: Temperature also affects the mixing because viscosity changes with increase in temperature.
10. **Mixture volume**: Mixing efficiency depends on mixture volume.
11. **Agitator type**: The shape, size, location and type of agitator also affect affects the degree of mixing.
12. **Speed/rpm of the impeller**: Mixing at less rpm is more homogenous than at higher rpm.
13. **Mixing time**: Mixing time is also very important for appropriate mixing.

DIFFERENCE BETWEEN SOLID AND LIQUID MIXING

The difference between solid and liquid mixing has been shown in Table 2.

Table . Difference Between Solid and Liquid Mixing

Solid Mixing	Liquids Mixing
In solid mixing two or more substances are intermingled by continuous movement of particles.	This is achieved by mixing elements of suitable shape to act as impeller to produce appropriate flow pattern in mixing vessel.
This is used for mixing of dry powders.	This is used in preparation of emulsion, suspension and mixtures.
Large sample size is required.	Small sample size is sufficient.
High power required for mixing.	Less power required for mixing.

DOUBLE CONE BLENDER

Principles of Double Cone Blender:

The Double Cone Blenders design is most often used for the intimate dry blending of freeflowing solids.

Construction of Double Cone Blender:

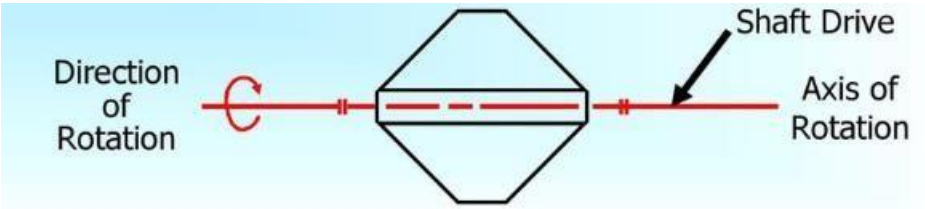


Fig.: Double Cone Blender

- The conical shape at both ends enables uniform mixing and easy discharge.
- The cone is statically balanced which protects the gear box and motor from any excessive load.
- Powder is loaded into the cone through a wide opening and discharged through a butterfly or a Slide valve.
- Depending upon the characteristic of the product, paddle type baffles can be provided on the shaft.
- Flame proof electricals can be provided as optional.
- 'Slant' design (off centre) CLIN CONE BLENDER are also used.
- Dust free bin charging system ensures minimum material handling.
- Mixing, uniform blending and de-agglomeration.

Working of Double Cone Blender:

- **Double Cone Blenders** are most often used for dry blending of free flowing solids. The solids being blended in these units can vary in bulk density and in percentage of the total mixture.
- Materials being blended are constantly being intermixed as the **Double Cone** rotates.

Merits of Double Cone Blender:

- If fragile granules are to be blended, double cone blender is suitable because of minimum attrition.
- They handle large capacities.
- Easy to clean, load, and unload.
- This equipment requires minimum maintenance.

Demerits of Double Cone Blender:

- Double cone blender needs high head space for installation.
- It is not suitable for fine particulate system or ingredients of large difference in particle size distribution, because not enough shear is applied.
- If powders are free flowing, serial dilution is required for the addition of low dose active ingredients.

TWIN SHELL BLENDER OR V CONE BLENDER

Principle of Twin Shell Blender:

The mixing occurs due to tumbling motion.

Construction of Twin Shell Blender:

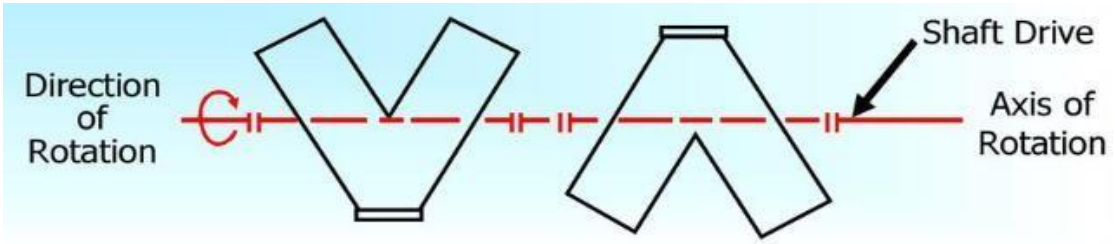


Fig.: Twin Shell Blender

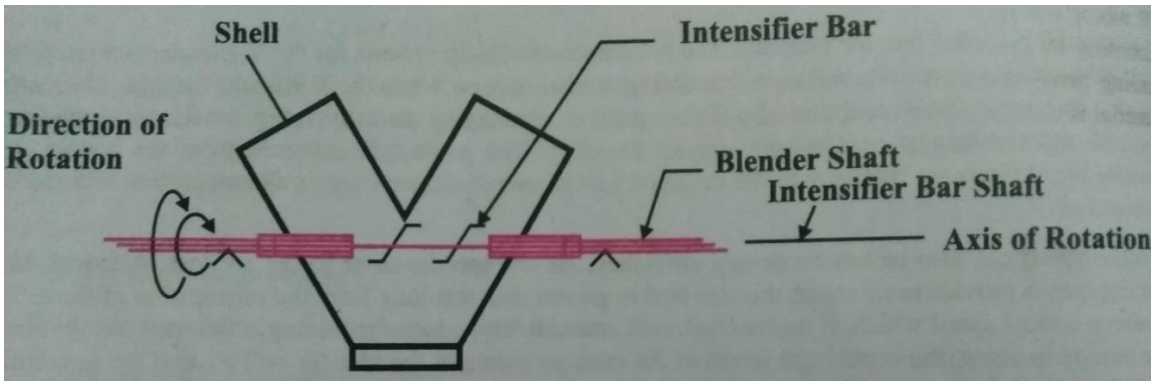


Fig.: V Blender with Intensifier Bar

- **Twin shell blenders** have two connected blending **shells** that are connected to form a V-shape. Intensifier bars are designed to break up clumps of solids while the product is separated in the two ends of the V (when the **twin shell blender** is upside down).
- It consists of horizontal shaft rotated about an axis causing the particles within the mixer to tumble over each other onto the mixture surface.
- The charging of materials into the V-Blender is through either of the two ends.

- Batches from 20 kg to 1 tonne can be loaded for mixing depending upon the size of the equipments.

Working of Twin Shell Blender:

- The V-**Blender** (also known as a **twin shell blender**) is one of the most commonly used tumbling **blenders**.
- The material is loaded into the blender.
- As the V-**blender** tumbles, the material continuously splits and recombines, with the mixing occurring as the material free-falls randomly inside the vessel.
- Tumble **blenders** rely upon the action of gravity to cause the powder to cascade within a rotating vessel.
- The recommended filled-up volume for the V-Blender is 50 to 60% of the total blender volume.
- The product is collected from the bottom of V.
- Normal blend times are typically in the range of 5 to 15 minutes depending on the properties of materials to be blended.

Marist of Twin Shell Blender or V Cone Blender:

V Cone Blender without Baffle-

- Have large capacities
- Easy handling
- Minimum maintenance

V Cone Blender with Baffle-

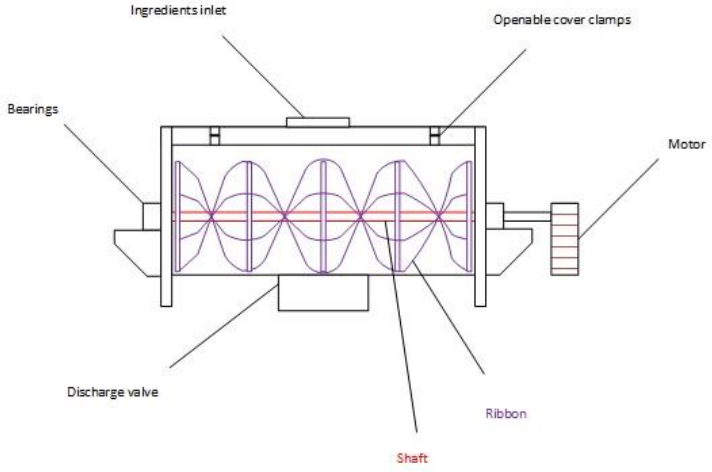
- Wet and dry mixing
- High shearing force
- Serial dilution is not required

RIBBON BLENDER

Principle of Ribbon Blender:

- The mechanism of mixing is shear which is transferred by moving blades (ribbon shaped) in a fixed (non-movable) shell.

Construction of Ribbon Blender:



Ribbon Blender

- A **ribbon blender** consists of a U-shaped horizontal trough (shell) containing a double helical **ribbon** agitator that rotates within.
- The agitator's shaft is positioned in the centre of the trough and has welded spokes on which the helical **ribbons** (also known as spirals) are welded.
- The blades have both right- and left-hand twists.
- The blades are connected to a fixed speed drive.
- The ribbon blender is top loading with a bottom discharge port.
- The trough can be closed with a lid.

Working of Ribbon Blender:

- Different powders are introduced from the top of the trough.
- The outer ribbon of agitator moves the material from the ends to centre while the inner ribbon moves the material from the centre to end.
- Through the fixed speed drive, ribbons are allowed to rotate.
- Radial movement is achieved because of the rotational motion of the ribbons.
- The difference in the peripheral speeds of the outer and inner ribbon results in axial movement, homogenous blending is achieved in short time.
- The powders are lifted by a centrally located vertical screw and allowed to cascade to the bottom of the container (tumbling action).
- The counter acting blades set up high shear and are effective in breaking up lumps or aggregates.
- Helical blades move the powders from one end to another.
- The blend is discharged from the bottom opening.

Merits of Ribbon Blender:

- The Ribbon mixer has price savings as a result of the thermal treatment is accomplished among a similar time and liner being utilized for combining step.
- Correct management of batch thermal treatment time. Low opportunity cost because the drying is accomplished within the same time and vessel getting used for transferring and combining.
- One of the most important benefits of using a ribbon mixer for any industrial project is that it blends nearly any material completely with nearly no flaws.
- This comes in handy for producing any material that must be mixed well like paint, concrete, and foods.
- These mixers are usually utilized in bakeries that require to blend large amounts of ingredients at only once. They will additionally mix materials fairly quickly, although the ribbons themselves inch.
- Finally, the common ribbon mixer features a very large trough, thus it’s ideal for big projects.
- High shear is also applied by exploitation perforated baffles that create a rubbing and breakdown aggregates. Headroom needs less aera.
- Protect the motor and ribbon mixer from overload.
- Once the load is just too massive to the drum and rotates, the operating liquid is ejected from the liquid plug to separate the operating machine and therefore the load, in order that the motor and instrumentality won’t be broken once beginning and overloading.
- The speed distinction caused by the impact is going to be mitigated by coupling.

Demerits of Ribbon Blender:

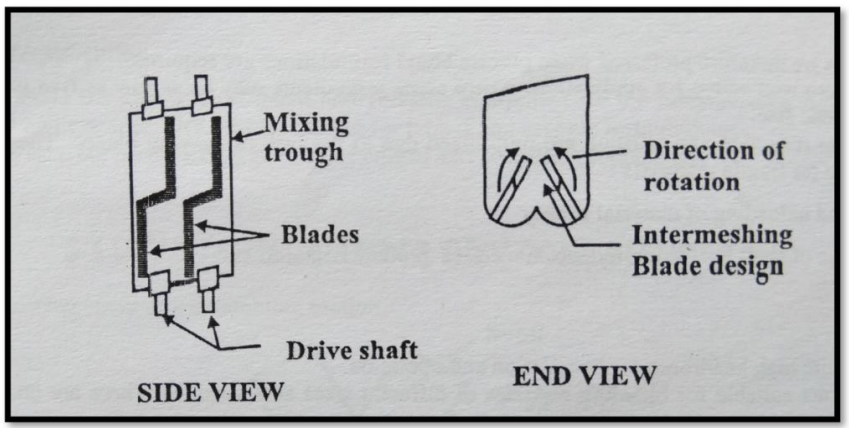
- The hydraulic mechanical device isn't loaded with the electrical converter generally, and may not modify the rotating speed of the ribbon mixer effectively, because the loading of hydraulic couplings is simple to make multiple transfer mechanical energy, leading to power consumption, thus it can't improve the start-up performance of the ribbon mixer.
- It is a poor mixer as a result of the movement of particles is two dimensional.
- Shearing action is a smaller amount than in planetary mixer.
- It has a set speed drive.
- Rate of mixing is greater at the surface, causing local differences in mixture composition.
- Attrition of particles may occur at the wall due to the higher forces present there.
- Prone to dead spots, especially near the discharge valve, and along the central axis.
- There aren't many downsides to using a ribbon mixer to combine ingredients. the sole major disadvantage that you simply may realize is that a ribbon mixer takes large amounts of power to work properly, thus you must positively detain mind your energy desires if you're about to use one in every of these machines.

SIGMA BLADE MIXER

Principle of Sigma Blade Mixer:

- The mechanism of mixing is shearing.

Construction of Sigma Blade Mixer:



Construction of Sigma Blade Mixer

- A ribbon blender consists of a u-shaped horizontal trough or shell containing a helical double ribbon agitator that rotates inside.
- The shaft of agitator is positioned in the centre of the trough on which the helical ribbons are welded.
- Since the ribbon stirrer consists of a set of internal and external helical ribbons, it is also called a double helical ribbon agitator.
- The ribbon blenders are powered by a drive system consisting of a motor, a gear box and couplings.
- These are powered by 10 HP to 15HP motor for 100Kg of product mass to be blended.
- The specific power ranges from 3 to 12 KW/M^3 according to the products to be mixed.

Working of Sigma Blade Mixer:

- Different powders are introduced from the top of the trough.

- The body is covered because considerable dust may be involved during dry blending and granulating solution may evaporate during wet granulation.
- Through the fixed speed drive, the sigma blades are allowed to rotate.
- The blade moves at different speeds.

Merits of Sigma Blade Mixer:

- Sigma blade mixture creates a minimum a dead space during mixing.
- Ideal for mixing, kneading of high viscous mass, sticky and dough like products.
- Extruding.
- These mixers and their variants are capable of handling viscosities as high as 10 centipoises.

Demerits of Sigma Blade Mixer:

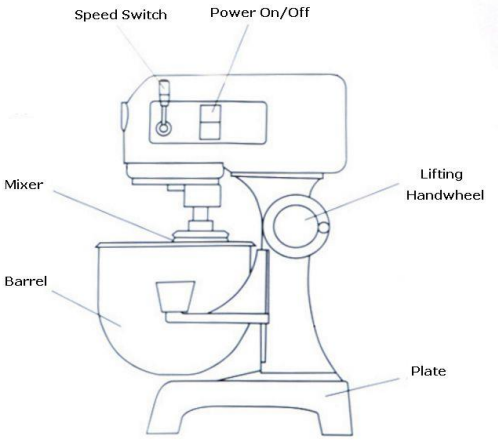
- Sigma blade mixer works at a fixed speed.
- Power consumption in double arm kneader is very high compared to other type of mixer.

PLANETARY MIXER

Principle of Planetary Mixer:

The principle of planetary mixers is very simple, which usually have two or three multi-hinged blades, when the paddles are revolution and rotation running at the same time, so that the material will flows up and down as well as around the inner cylinder, which can reach the mixing effect in a very short time. It is also known as change can mixer.

Construction of Planetary Mixer:



Planetary Mixer

Construction of Planetary Mixer

- It consists of a vertical cylindrical shell, which can be removed either by lowering it beneath the blade or raising the blade above the bowl.
- The mixing blade is mounted from the top of the bowl.
- The mixing shaft if driven by a planetary gear train.
- It rotates around the ring gear, which further rotates round the mixer blade.
- It is normally built with a variable speed drive.

Working of Planetary Mixer:

- The material to be mixed is loaded in mixing bowl.
- The blade rotates on their own axis when they orbit the mixing bowl on a common

axis. Therefore, there is no dead space in the mixing and high shear is applied for mixing.

- After mixing, the material is discharged through a bottom valve, or by manual scooping of the material from the bowl.

Merits of Planetary Mixer:

- Simple construction, operation, and relatively low cost.
- No dead spot in the mixing.
- Rotation speed of the blade can be varied.
- Used for wet granulation process.
- High mixing efficiency.

Demerits of Planetary Mixer:

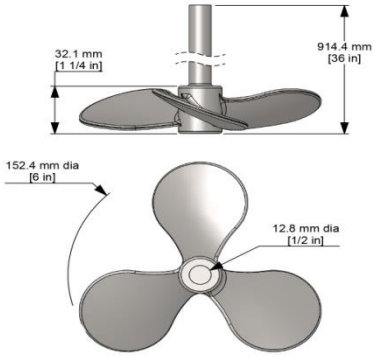
- Requires high power.
- Heat build-up within powder mix.

PROPELLERS

Principle of Propeller Mixer:

- The propeller mixer mainly works on the principle of shearing force.

Construction of Propeller Mixer:



Three Bladed Design Propeller

It consists of vessel and propeller.
A propeller has angled blades, which cause the fluid to circulate in both an axial and radialdirection.
Size of the propeller is small and many increases up to 0.5meters depending on size of tank.Small size propeller can rotate up to 8000 rpm.

Working of Propeller Mixer:

- A vortex forms when a centrifugal force is imparted to the liquid by the propeller blades cause it to backup ground the sides of the vessel and creates a depression at the shaft.
- As the speed of the rotation is increased air may be sucked in to the fluid by the formation of a vortex this causes frothing and possible oxidation.
- Another method supressing vortex is to fit vertical baffles in to the vessel.
- Installation of vertical propeller reduces the vortex to considerable extent.
- Vertical propeller mixer consists of three blades (4 ft long).

Horizontal or Inclined Propeller or Marine Propeller are also used on side entry mixers.They

are mounted with the impeller shaft inclined at an angle to the vessel axis to improve the process results.They provide good blending capability in small batches of low to medium viscosity.

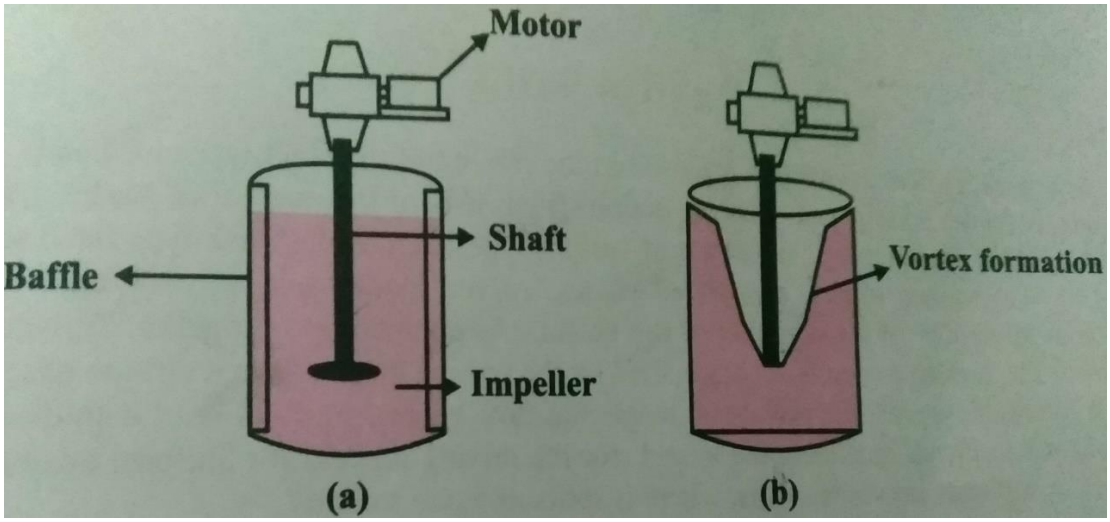


Fig: (a) Baffled tank with no vortex formation; (b) Unbaffled tank having vortex formation

Merits of Propeller Mixer:

- Propeller is effective when high mixing capacity is required.

Demerits of Propeller Mixer:

- Propellers are not effective for liquids having viscosity greater than 5.0 Pascal second.
- Equipment cost is high.

TURBINES

Principle of Turbine Mixer:

A turbine mixer is a mechanical device that is used in mixing different type of liquids. The turbine mixer works mainly on the principle of shearing action.

Construction of Turbine Mixer:

- Turbine consists of number of blades attached to the circular disk.
- The blades used in the mixture are of various types: flat blades, disk-type flat blades, inclined blades, curved blades, arrow headed blades, and so on.
- The diameter of turbine varies from 30 to 50 percentage of the diameter of vessel.
- As compared to propeller turbines rotates at lower speed.

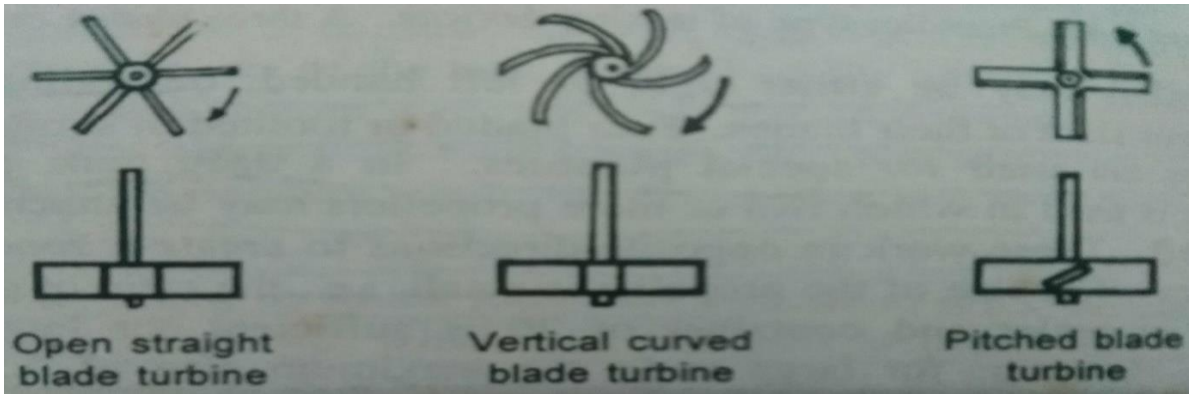


Fig.: Different types Turbines

Working of Turbine Mixer:

When turbine mixer operates at sufficiently at high rotational speeds, the radial tangentialflow becomes pronounced with the formation of vortex.

It is necessary to install baffles in the vessel for the mixing process for uniform mixing. The radial flow of the impeller impinges on vessel walls, where it slits in to two streams.

Merits of Turbine Mixer:

- Turbines give greater shearing force than propeller.
- Therefore, turbines are suitable for emulsification.

Demerits of Turbine Mixer:

- Turbines have less pumping rate.

PADDLES

Principle of Paddles:

- Mixer works mainly on the principle of shearing action

Construction of Paddles:

- Paddles consist of two long flat blades attached vertically to a shaft.
- IT rotates at low speed.
- Paddle mixer is suitable to mix viscous liquids or semisolids
- Blades used in this mixer are dished or hemispherical in shape.
- The diameter of paddle is 50-80 percentage of inside diameter of vessel.

Working of Paddles:

- Paddles push liquid radially and tangentially.
- There is no axial movement of flow during mixing.

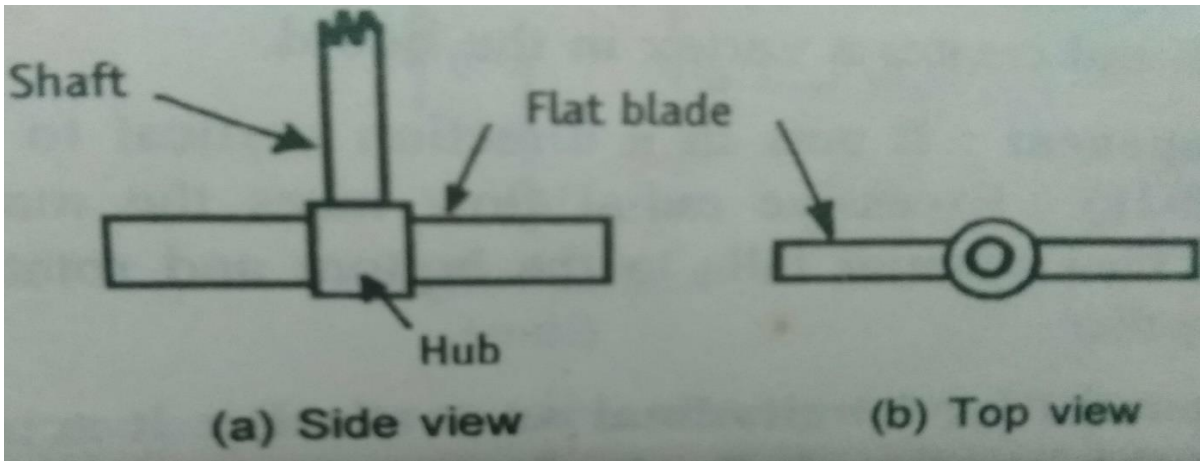


Fig: Paddle type of Agitator or Impeller

Merits of Paddles:

- Vortex formation is not possible.
- It has low speed.
- Mixing efficiency is better.

- No dead spots and deposited solids.

Demerits of Paddles:

- Here suspension mixing is poor.
- Baffled tanks are required.

SILVERSON EMULSIFIER

Principle of Silverson Emulsifier:

The silverson homogenizer works on the principle that the large globules in a course emulsion are broken in to smaller globules by intensive shearing forces and turbulence by high speed rotors.

Construction of Silverson Emulsifier:

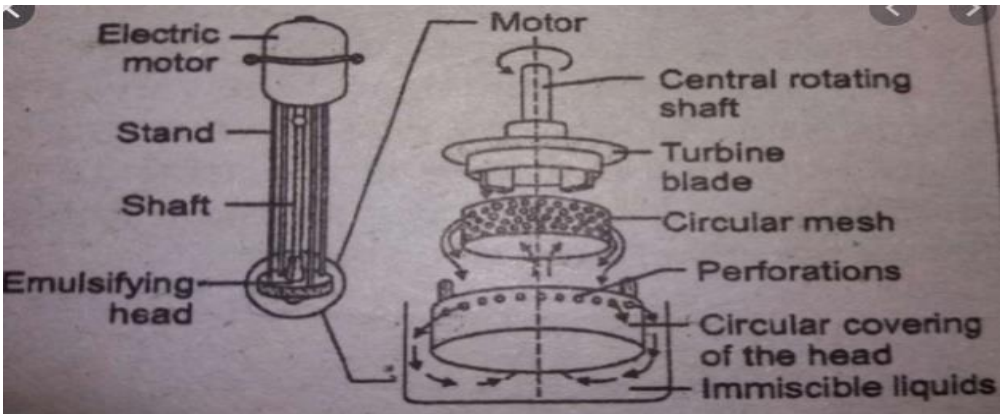


Fig: Silverson Emulsifier

- It consists of emulsifier head.
- The emulsifier head consist of a number of turbine blades.
- The blades are surrounded by mesh which is enclosed by cover having perforations.
- The blades are rotated by using electric motor fitted at the top.
- There is also one shaft whose one end is connected to motor and other end is connected to head.

Working of Silverson Emulsifier:

- The emulsifier head is dipped in to the vessel containing immiscible liquids.
- When the motor is started, shaft rotates the head.
- Therefore, turbines blades also rotate at very high speed.
- The liquids are sucked trough the fine holes.
- The complex flow pattern can cause droplet break up under either laminar or turbulent conditions
- Centrifugal force expels the content through mesh and then to cover and subjects them to mechanical shear.
- This is followed by intense hydraulic shear.
- The oil is reduced in to globules quickly resulting in a homogenous uniform product.
- Then the fine emulsion emerge trough opening of cover.
- As a result, bigger globules rapidly break in to smaller globules.

Merits of Silverson Emulsifier:

- Fast and efficient.
- They are used to get a fine droplet or particle size (2-5 microns).
- Process efficiency is good.
- Low operating cost.

Demerits of Silverson Emulsifier:

- Chance of chocking of pores of mesh.

DRYING

Drying involves removal of water or another solvent by evaporation from a solid, semi-solid or liquid by application of heat and finally a liquid free solid product is obtained.

Difference between Drying and Evaporation

Drying	Evaporation
Done to get a stable dry product.	Final product is either concentrated suspension or wet slurry.
Removal of less amount of moisture.	Removal of large amount of liquid.
Drying occurs below boiling point.	Evaporation occurs more at boiling point.
Emphasize on solid product.	Emphasize on reducing the volume.

OBJECTIVES OF DRYING

1. The main **objectives of drying** include to preserve foods and increase their shelf life by reducing the water content
2. Reduce space requirements for storage and transport.
3. 3.To avoid or eliminate moisture which may lead to corrosion and decrease the product or drug stability.
4. To improve or keep the good properties of a material like granules, e.g. Flowability, compressibility.

APPLICATIONS OF DRYING

In pharmaceutical technology, drying is carried out for one or more of the following reasons:

1. Preparation of bulk drugs
2. Preservation of drug products
3. Improved characteristics
4. Improved handling
5. Drying reduces moisture content.

MECHANISM OF DRYING PROCESS

Drying does not mean only removal of the moisture but during the process, physical structure as well as the appearance has to be preserved. Drying is basically governed by the principles of transport of heat and mass. Different types of solids may have to be handled for drying crystalline, granular, beads, powders, sheets, slabs, filter-cakes etc. The mechanism of moisture transport in different solids may be broadly classified into

(i) transport by liquid or vapour diffusion
(ii) capillary section, and
(iii) pressure induced transport.

The mechanism that dominates depends on the nature of the solid, its pore structure and the rate of drying. Different mechanisms may come into play and dominate at different stages of drying of the same material.

The following terms are commonly used in Designing of Drying Systems:

Bound Water: Moisture content of a substance which exerts as equilibrium vapour pressure less than of the pure liquid at the same temperature is referred to as bound moisture or bound wated.

Unbound Water: Moisture content of the solid which exerts an equilibrium vapour pressure equal to that of pure liquid at the given temperature is the unbound moisture or unbound water.

Free Moisture Content (FMC): The moisture content of solid in excess of the equilibrium moisture content is referred as free moisture. During drying, only free moisture can be evaporated. The free moisture content of a solid depends upon the vapour concentration in the gas.

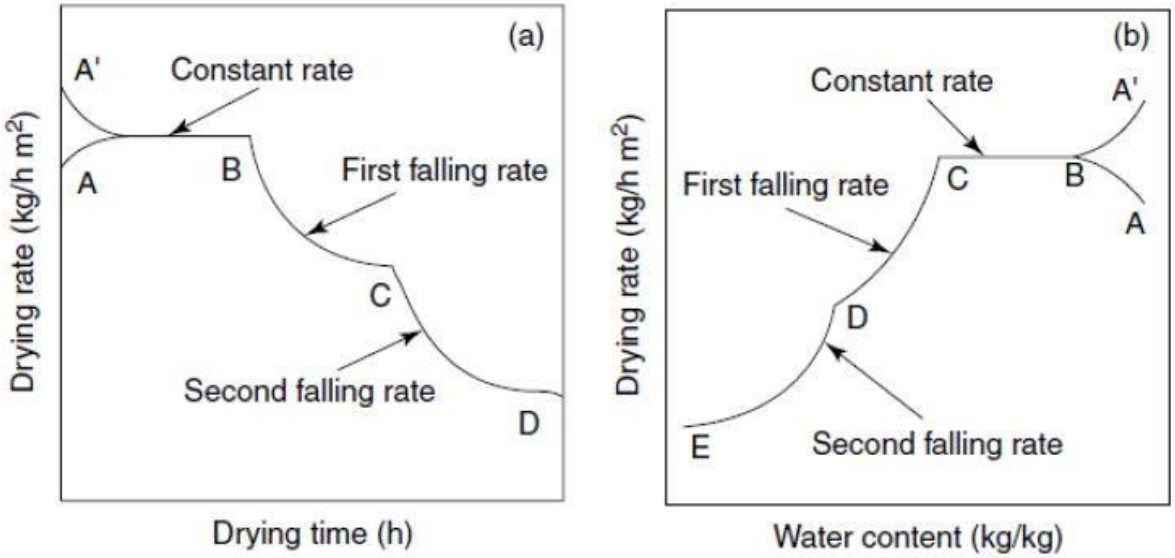
Equilibrium Moisture Content (EMC): The moisture contents of solid when it is in equilibrium with given partial pressure of vapour in gas phase is called as equilibrium moisture content. The EMC of a hygroscopic material surrounded at least partially by air is the **moisture content** at which the material is neither gaining nor losing **moisture**. The value of the EMC depends on the material and the relative **humidity** and temperature of the air with which it is in contact.

Critical Moisture Content (CMC): Similarly, the moisture content at which the constant rate drying period ends and the falling rate drying period starts is called critical moisture content.

Constant Rate Drying Period: During the constant rate drying period, the moisture evaporated per unit time per unit area of drying surface remains constant.

Falling Rate Drying Period: In falling rate drying period the amount of moisture evaporated per unit time per unit area of drying surface continuously decreases.

RATE OF DRYING CURVE



Typical drying rate curves: (a) drying rate versus drying time and (b) dryingrate versus water content

- Drying curve usually plots the drying rate versus drying time or moisture contents.
- Three major stages of drying can be observed in the drying curve.

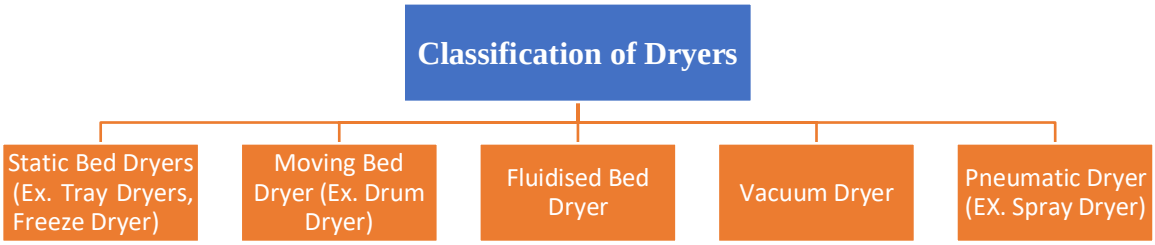
1. **Transient early stage**, during which the product is heating up (transient period)
2. **Constant rate period**, in which moisture is comparatively easy to remove
3. **Falling rate period**, in which moisture is bound or held within the solid matrix

Critical moisture content: The moisture content at the point when the drying period changes from a constant to a falling rate.

The drying behaviours of food materials depend on the porosity, homogeneity, andhygroscopic properties.

Hygroscopic food materials enter into the falling rate faster compared to non-hygroscopic food materials.

CLASSIFICATION OF DRYERS



TRAY DRYER

Principle of Tray Dryer:

The basic working **principle** of this incredible machine is the continuous circulation of hot air. It works on the principle of convection drying.

Construction of Tray Dryer:

Tray Dryer is used for the best drying results in conventional process. It is a double walled cabinet with Single or Two doors. The gap between two walls is filled with high density fibre glass wool insulation material to avoid heat transfer. Doors are provided with gaskets. Stainless steel trays are placed on the movable trolleys. Tray Dryer is provided with control panel board, process timer, Digital temperature controller cum indicator etc. Tray Dryer is available in capacities ranging from 6, 12, 24, 48, 96, 192 trays.

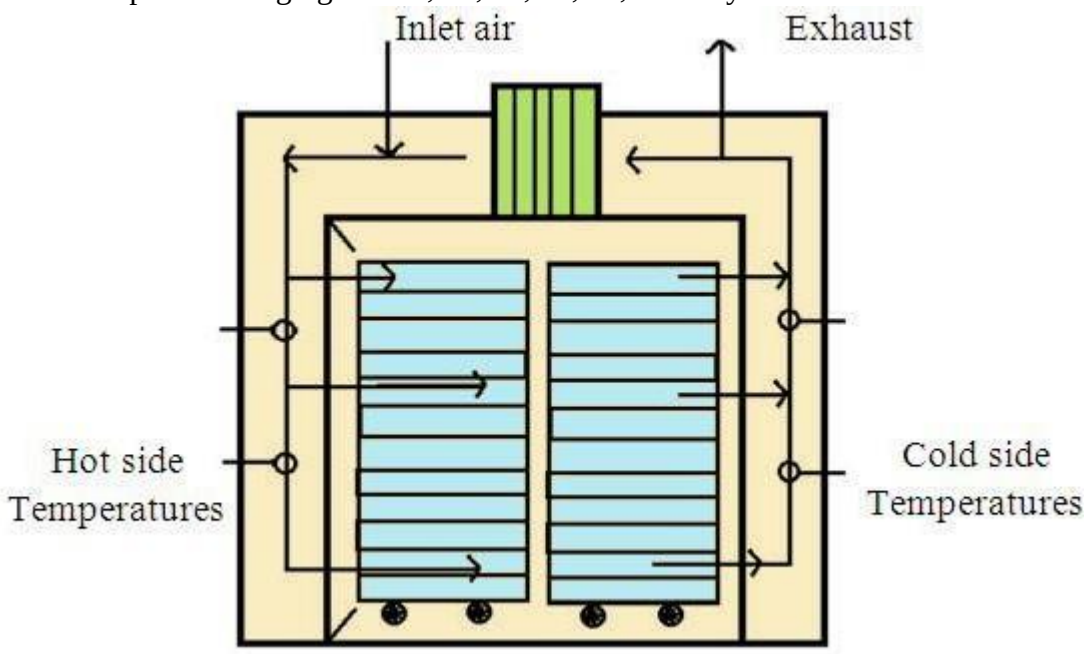


Fig: Tray Dryer

Working of Tray Dryer:

- In tray dryer hot air is continuously circulated. Forced convection heating takes place to remove moister from the solids placed in trays.
- Simultaneously the moist air is removed partially.
- Wet solid is loaded in to the trays. Trays are placed in the chamber.
- Fresh air is introduced through in let, which passes through the heaters and gets heated up.
- The water is picked up by the air. As the water evaporates from the surface, the water diffuses from the interior of the solids by the capillary action.
- These events occur in a single pass of air. The time of contact is short and amount of water picked up in a single pass is small.
- Therefore, the discharged air to the tune of 80 to 90 % is circulated back through the fans. Only 10 to 20% of fresh air is introduced.
- Moist air is discharged through outlet. Thus, constant temperature and uniform air flow over the materials can be maintained for achieving uniform drying.
- In case of the wet granules as in tablets and capsules drying is continued until the desired moister content is obtained.

- At the end of the drying trays or trucks are pulled out of the chamber and taken to a tray dumping station.

Advantages of Tray Dryer:

- Each batch is handled as a separate entity.
- It is more efficient in fuel consumption.
- It is operated batch-wise.
- It is simple to use.
- It provides tendency to over-dry the lower trays.
- It requires little labour costs – merely load and then unload.

Disadvantages of Tray Dryer:

- The process is time-consuming.
- It requires extra cost.
- Not suitable for oxidizable and thermolabile substances.

DRUM DRYER OR ROLL DRYER

Principle of Drum Dryer:

In **drum drying**, the heated surface is the envelope of a rotating horizontal metal cylinder. The cylinder is heated by steam condensing inside, at a pressure in the range of 200 to 500 kPa bringing the temperature of the cylinder wall to 120–155°C.

Construction of Drum Dryer or Roll Dryer:

A **drum dryer** consists of one or two horizontally mounted hollow cylinder(s) or drums of about 0.75-1.5 m in diameter and 2-4 m in length, made of high-grade cast iron or stainless steel, a supporting frame, a product feeding system, a scraper, and auxiliaries. The drum is heated internally by steam, and rotated on its longitudinal axis. The external surface of the drum is polished. Liquid or slurry is placed as feed in a pan. The drum is partially dipped in pan. The spreader is used to spread liquid film evenly on roller. The rotation of the drum adjusted so that all of the liquid is fully vaporized. The drum is rotated continuously. The dried deposits can be scrapped off with the help of doctor knife.

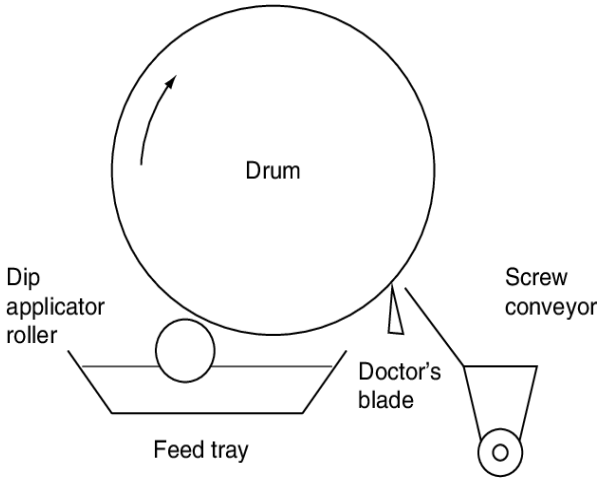


Fig.: Drum Dryer or Roll Dryer

Working of Drum Dryer or Roll Dryer:

- As the drum rotates, the liquid material gets adhere to external surface of drum.
- The liquid is spread as film on to the surface.
- The drying of the material is done by process of steam when passed in to the drum.
- By the mechanism of conduction, the heat gets transferred in to drum and drying process takes place.
- The material is completely dried during whole process duringits revolution.
- The dried material is scrapped by the knife and that fall in to the bin.

Advantages of Drum Dryer or Roll Dryer:

- Drying takes place in less time.
- It is suitable for thermosensitive drugs.
- It occupies less space.
- In order to reduce the temperature of drying the drum can be enclosed in a vacuum chamber.
- Rapid drying takes place due to rapid heat and mass transfer.

Disadvantages of Drum Dryer or Roll Dryer:

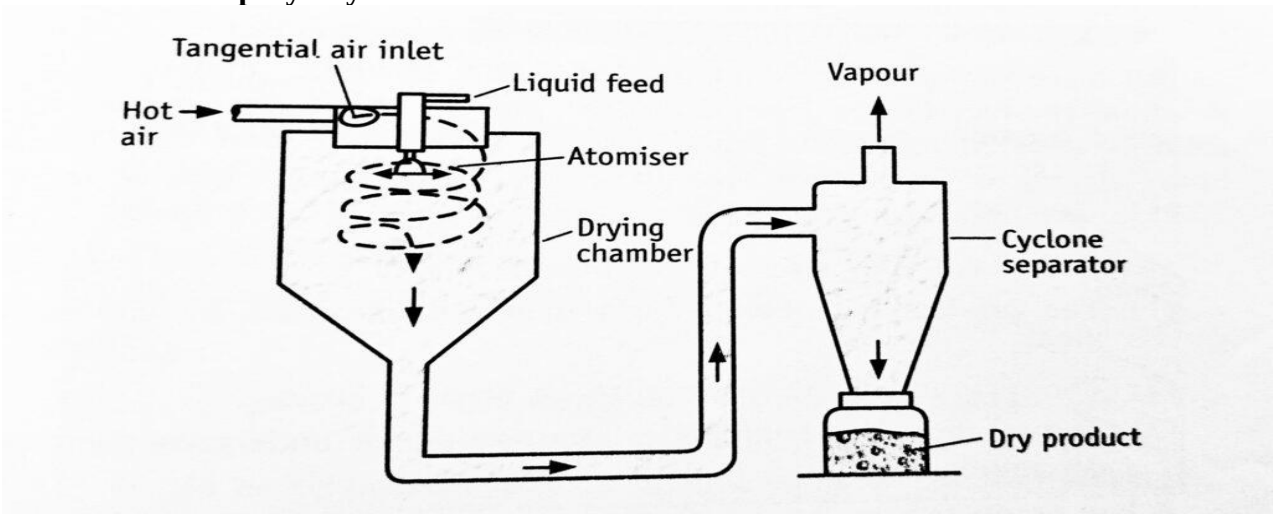
- Maintenance cost is high.
- Skilled operations are essential to control thickness of film.
- It is not suitable for less solubility products.
- The operating conditions are critical. It is necessary to introduce careful control on feed rate, film thickness, speed of drum rotation and drum temperature.

SPRAY DRYER

Principle of Spray Dryer:

Spray drying is an industrial process for dehydration of a liquid feed containing dissolved and/or dispersed solids, by transforming that liquid into a **spray** of small droplets and exposing these droplets to a flow of hot air.

Construction of Spray Dryer:



Spray Dryer

A spray dryer is composed of a feed pump, atomizer, air heating unit, air dispenser, drying chamber (diameter of the drying chamber ranges between 2.5 to 9.0 m and height is 25 m or

more) and also systems for exhaust air cleansing and also powder recovery/separator. The spray disk atomizer is about 300 millimetres in diameter and rotates at a speed of 3,000 to 50,000 revolutions per minutes. In the spray dryer the liquid to be dried is atomised into the good droplets, that are tossed radially into a relocating stream of warm gas.

Working of Spray Dryer:

- A spray dryer takes a liquid stream and separates the solute or suspension as a solid and the solvent into a vapor.
- The solid is usually collected in a drum or cyclone.
- The liquid input stream is sprayed through a nozzle into a hot vapor stream and vaporized. Solids form as moisture quickly leaves the droplets.

The three stages that occur in a spray dryer before drying is accomplished include:

- Atomization
- Spray-air mixing and moisture evaporation.
- Dry product separation from the exit air.

The nature of the final product obtained after drying in a spray dryer depends on;

- The design and operation of the spray dryer.
- The physicochemical properties of the feed.

Advantages of Spray Drying:

- Product quality and properties can be effectively controlled and maintained through the entire drying operation.
- Thermolabile products/ pharmaceuticals can be dried at atmospheric pressure and low temperature.
- Feedstock in solution, slurry, emulsion, paste, and melt form can be dried if pumpable.
- Corrosion problem is minimal and the selection of materials of construction of spray dryer is simplified since the dried material comes in contact with the equipment surfaces in an anhydrous condition.
- Spray dryer produces dry powder particles of controllable particle size, shape, form, moisture content, and other specific properties irrespective of dryer capacity and heat sensitivity.
- Spray dryer handles a wide range of production rates and provides extensive flexibility in its design that is product specification are readily met through the selection of appropriate spray dryer design and its operation from a wide range of available design.
- It is energy-intensive equipment because specific heat of evaporation can be supplied in a short time. The temperature difference across the drying chamber is relatively small and an appreciable amount of heat is lost with exhaust air.

Disadvantages of Spray Drying:

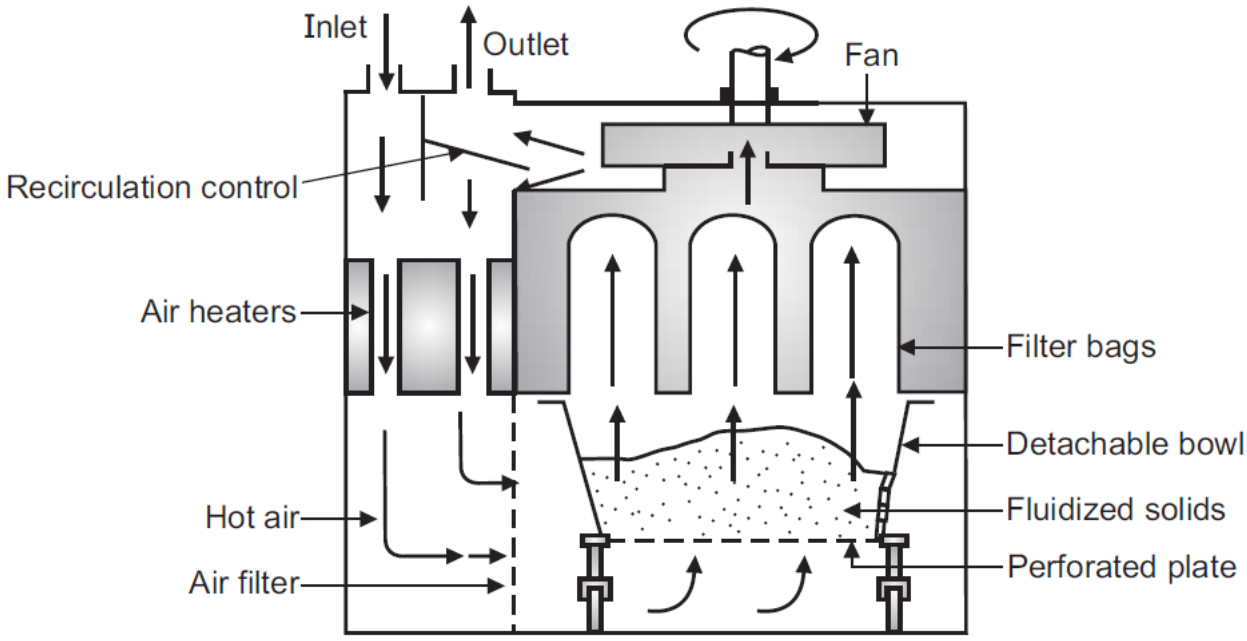
- Spray dryer is bulky and also expensive to install.
- It is difficult to clean after use.
- It has a low thermal efficiency that is a lot of heat is wasted during operation.
- Solid materials cannot be dried using spray dryers.
- Product degradation or fire hazard may result from product deposit on the drying chamber.

FLUIDIZED BED DRYER (FBD)

Principle of Fluidized Bed Dryer (FBD): The equipment works on a principle of fluidization of the feed materials. In fluidization process, hot air is introduced at high pressure through a perforated bed of moist solid particulate. The wet solids are lifted from the bottom and suspended in a stream of air (fluidized state).

Construction of Fluidized Bed Dryer (FBD):

- The dryer is made up of stainless steel or plastic.
- A detachable bowel is placed at the bottom of the dryer, which is used for charging and discharging.
- The bowel has a perforated bottom with a wire mesh support for placing materials to be dried.
- A fan is mounted in the upper part for circulating hot air.
- Fresh air inlet, prefilter and heat exchanger are connected serially to heat the air to the required temperature.
- The temperature of hot air and exit air are monitored.
- Bag filters are placed above the drying bowl for the recovery of fines.



Fluidized Bed Dryer

Working of Fluidized Bed Dryer:

- The wet granules to be dried are placed in a detachable bowl. The bowl is inserted in the drier.
- Fresh air can pass trough a prefilter, which is then heated when passing trough a heat exchanger.
- Hot air flows through the bottom of the bowl.
- At the same time, fan start to rotate.
- The air speed increases gradually.
- When the velocity of air is greater than the sedimentation rate of the granules, the granules remain suspended in the gas stream.

- After specific time, a pressure point is reached in which the friction drag on a particle is equal to the force of gravity.
- The granules rise in the container due to high gas velocity of 1.5 to 7.5 meter per minute and then fall back. This state is known as fluidized state.
- The gas surround to each granule do dry them completely.
- The air comes out of the dryer passing through the filters in the bag.
- The entrained particles remain adhered to the interior surface of bags.
- Periodically bags are shaken to remove entrained particles.

Advantages of Fluidized Bed Dryer:

- It takes less time to complete drying as compared to other dryer.
- Drying is achieved at constant rate.
- Handling time is also short.
- It is available at different sizes with different drying capacity.
- The equipment is simple and less labour cost required.
- More thermal efficiency.
- Drying capacity is more than other dryer.
- It facilitates the drying of thermolabile substances since the contact time of drying is short.
- It is batch type or continuous type process.

Disadvantages of Fluidized Bed Dryer:

- Many organic powders develop electrostatic charge during drying. To avoid this efficient electrical grounding of the dryer is essential.
- Chances of attrition of some materials resulting in production of fines.

VACUUM DRYER

Principle of Vacuum Dryer:

Vacuum drying is generally used for the drying of substances which are hygroscopic and heat sensitive, and is based on the principle of creating a vacuum to decrease the chamber pressure below the vapour pressure of the water, causing it to boil. Hence, water evaporates faster. The heat transfer becomes, i.e., rate of drying enhances substantially.

Construction of Vacuum Dryer:

The oven is divided into hollow trays which increases the surface area for heat conduction. The oven door is locked air tight and is connected to **vacuum** pump to reduce the pressure. The materials to be dried are kept on the trays inside the **vacuum dryer** and pressure is reduced by means of **vacuum** pump.

The enclosed space (aproximately 1.5 meter cube) is devided in to a number of portions by means of 20 hollow shelves, which are part of the jacket. These shelves provide larger surface area (about 45 to 50 meter square) for conduction of heat. Over the shelves, metal trays are placed for keeping the material. The oven door can be locked tightly to give an air tight seal. The oven is connected to a vacuum pump by placing condenser in between.

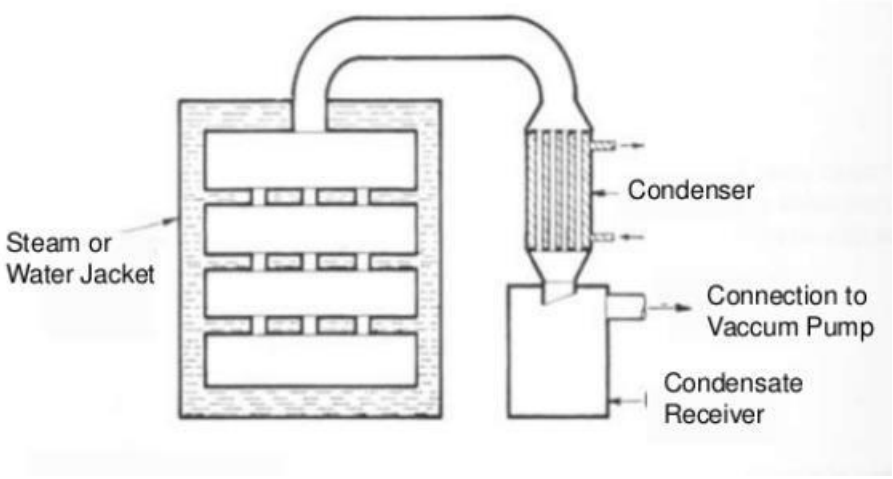


Fig.: Vacuum Dryer

Working of Vacuum Dryer:

- The tray that are present in the dryer are used to dry the material that are placed in theshelves and the pressure is reduced to 30 to 60 Kps by vacuum pump.
- The door closes firmly and steam passes through the jacket space and the shelves.
- So the heat transfer is carried out by the conduction mechanism.
- When evaporating under vacuum, the water is evaporated from the material at 25 -30℃.
- The vapour goes to the condenser.
- After drying vacuum line is disconnected.
- Then the materials are collected from the tray.

Advantages of Vacuum Dryer:

- Material handling is easy.
- Hollow shelves which are electrically heated can be used.
- It provides large surface area. So the heat can be easily transfer through the body ofthe dryer and last drying action takes place.
- Hot water can be supplied through the dryer, which help in drying process at thedesired temperature.

Disadvantages of Vacuum Dryer:

- Dryer is a batch type process.
- It has low efficiency.
- It is more expensive.
- Labour cost is too high.
- Needs high maintenance.
- There is a danger of overheating due to vacuum.

FREEZE DRYER

Principle of Freeze Dryer:

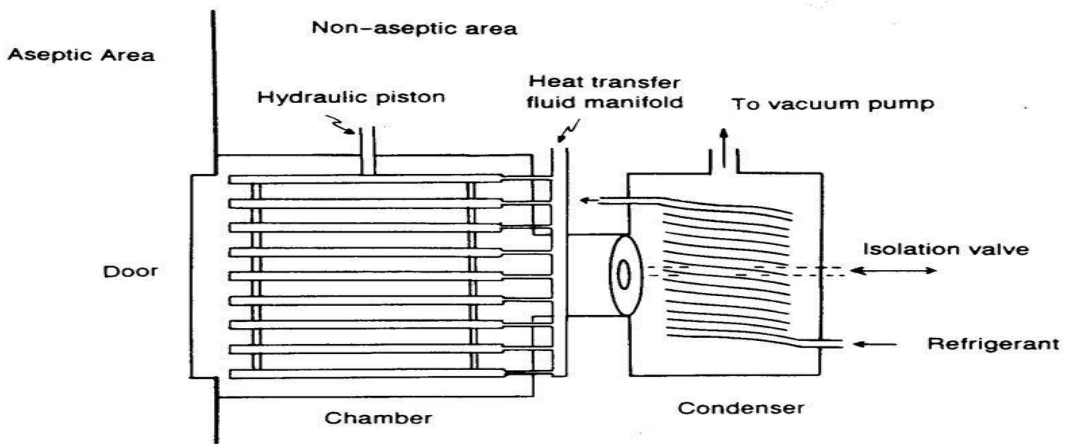
Freeze drying or lyophilisation is a drying process used to convert solutions or suspensions of labile materials into solids of sufficient stability for distribution and storage. The fundamentalprinciple in freeze-drying is sublimation, the shift from a solid directly into a gas. Just like evaporation, sublimation occurs when a molecule gains enough energy to break free from the molecules around it. Drying is achieved by subjecting the materials to temperature and pressures below the triple point.

Construction of Freeze Dryer:

The construction of freeze dryer is shown in figure. It consists of

- Drying chamber in which trays are loaded.
- Heat supply in radiation source, heating coils.
- Vapour condensing or adsorption system.
- Vacuum pump or stream ejector or both.

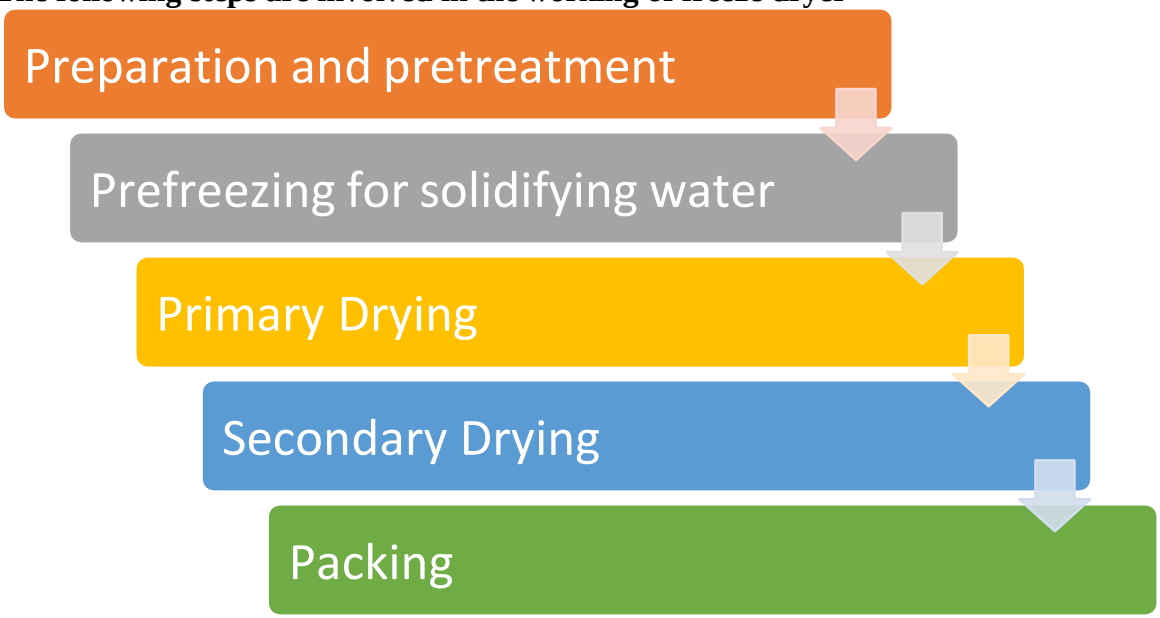
The chamber for vacuum drying is generally designed for batch operation. It consists of shelves for keeping the material. The distance between subliming and condenser must be less than the mean path of molecules. This increases the rate of drying. The condenser consists of relatively large surface cooled by solid carbon dioxide slurred with acetone or ethanol. The temperature of condenser must be much lower than evaporated surface of frozen substance. In order to maintain this condition, the condenser surface is cleaned repeatedly.



Freeze Dryer

Working of Freeze Dryer:

The following steps are involved in the working of freeze dryer



Pretreatment includes any method of treating the product prior to **freezing**. This may include concentrating the product, formulation revision (i.e., addition of components to increase stability, preserve appearance, and/or improve processing), decreasing a high-vapor- pressure solvent, or increasing the surface area. This reduces the actual drying by 8 to 10 times. The final product becomes more porous.

During **pre-freezing**, the **freeze dryer** works as a **freezer** in that no vacuum is applied. Vials, ampoules or bottle in which the aqueous solutions are packed or frozen in cold shelves (about -50℃). During this stage, cabinet is maintained at low temperature and atmospheric pressure. The normal cooling rate is about 1 to 3 kelvin per minute so that large ice crystals with relatively large holes are formed on sublimation of ice. This is also responsible for giving a porous product.

Primary drying (sublimation of ice under vacuum): In this step, the material to be dried is spread as much large surface as possible for sublimation. The temperature and pressure should be below the triple point of water, i.e., 0.0098℃ and 0.533 Kilopascal, (4.58 mmHg) for the sublimation, when water alone is present.

When a solution of solid is dried, the depression of freezing point of water occurs. Hence it is essential that the temperature be brought bellow the eutectic point. The pressure and temperature at which the frozen solid vaporises without conversion to a liquid is referred to as the eutectic point. Depending on the drug substance dissolved in water, the eutectic point is determined. The usual range is from -10℃ to 30℃. The condition of 1 to 8 K bellow eutectic point is sufficient.

Vacuum is applied to the tune of about 3 mmHg (0.4 Kilopascals) on the frozen sample. The temperature is linearly increased to about 30℃ in a span of 2 hours.

Heat (about 2900 Kilojoules per kg) is supplied which transfers as latent heat and icesublimes directly into vapour state. The heat controls the movement of ice layer inwards. It has to be controlled in such a manner so as to get highest possible water vapour at ice surface without melting the material. As soon as the vapour molecules are formed, these are removed. The overall driving force is the temperature difference (also vapour pressure difference) between evaporating surface and condenser.

As the drying proceeds, the thickness of frozen layer decreases and thickness of partially dried solids increases. Primary drying stage removes easily removable moisture. During this stage, about 98% to 99% water is removed. Till traces of moisture is present in the sample.

Secondary drying (removal of residual moisture under high vacuum): During this stage traces of moisture is removed. The temperature of solid is raised to as high as 50 to 60℃, but vacuum is lowered bellow that is used in primary drying (50 mmHg). The rate of drying is very low and it takes about 10 to 20 hours.

Packing is done by replacing vacuum with inert gas, bottles and vials are closed.

Advantages of Freeze Dryer:

- It is suitable for drying heat sensitive products
- Freeze dried products is porous and easy to dehydrated and instantly dissolved.
- Drying takes place at very low temperature, so that enzyme action is inhibited andchemical decomposition, particularly hydrolysis, is minimized.
- Denaturation of protein does not occur.
- Loss of volatile material is less.
- Sterility can be maintained.

Disadvantages of Freeze Dryer:

- The process is very slow**.**

- Expensive process.
- It is not a general method of drying, but it is limited to certain type of valuableproducts that cannot be dried by any other means.
- The period of drying is high.
- The product is prone to oxidation, due to the high porosity and large surface area.Therefore, the product must be vacuum packed or with an inert gas or in container.

FILTRATION

.

When solid are present in very low concentration, i.e., not exceeding 1.0%w/v, the process of its separation from liquid is called clarification.

Terms used in Filtration

- Slurry - Suspension to be filtered
- Filter medium - Porous medium used to retain solid
- Filter cake - Accumulated solids on the filter
- Filtrate - Clear liquid passing through the filter

Process of filtration

- Pores of filter medium are smaller than size of particles to be separate.
- Filter medium (filter paper) is placed on a support (mesh)
- Slurry is placed over the filter medium
- Due to pressure difference across the filter, fluid flows through the filter medium
- Gravity is acting over the liquid medium
- So solids are trapped on the surface of the filter medium

Applications of filtration

- Production of sterile products
- Production of bulk drugs
- Production of liquid dosage formulation
    - Dewaxing of oils
    - Removing suspended oils from aqueous solutions
    - Removing of undesirable solids
    - Clarifying the potable water
- Effluents and waste water treatment

Mechanism of filtration

The mechanism whereby particles are retained by a filter is significant only in initial stages of filtration.

1. Straining - Similar to sieving, i.e., particles of larger size can't pass through smaller pore size of filter medium.

2. Impingement - Solids having the momentum move along the path of streaming flow and strike (impinge) the filter medium. Thus the solids are retained on the filter medium.

3. Entanglement - Particles become entwined (entangled) in the masses of fibres (of cloths with fine hairy surface or porous felt) due to smaller size of particles than the pore size. Thus solids are retained within filter medium.

4. Attractive forces - Solids are retained on the filter medium as a result of attractive force between particles and filter medium, as in case of electrostatic filtration.

Types of filtration

Surface/ screen filtration –

- It is a screening action by which pores or holes of medium prevent the passage of solids.
- Mechanism involved: straining and impingement
- For this, plates with holes or woven sieves are used.

Depth filtration -

- In this slurry penetrates to a point where the diameter of solid particles is greater than that of the tortuous void or channel.
- Mechanism: Entanglement
- The solids are retained with a gradient density structure by physical restriction or by adsorption properties of medium.

THEORY OF FILTRATION:

1. Poiseullie's Equation

- Poiseullie considered that filtration is similar to the streamline flow of liquid under pressure through capillaries.
- Poiseullie's Equation is-

$$V = \pi \Delta P r^4 / 8 L \eta$$

Where, V = rate of flow, m3 /s (l/s)

ΔP= Pressure difference across the filter, Pa

r = radius of capillary in the filter bed, m

L = thickness of filter cake (capillary length), m

η = viscosity of filtrate, Pa.s

- If the cake is composed of bulky mass of particles and the liquid flows through the interstice, then flow of liquids through these may be expressed by this equation.

2. Darcy's Equation

- Poiseullie's law assumes that the capillaries found in the filter are highly irregular and non-uniform.
- Therefore, if the length of capillary is taken as the thickness of bed, a correction factor for radius is applied so that the rate is closely approximated and simplified.
- The factors influencing the rate of filtration has been incorporated into an equation by Darcy, which is:

$$V = KA\Delta P / \eta L$$

Where, K = permeability coefficient of cake, m2

A = surface area of porous bed (filter medium), m2

K depends on characteristics of cake, such as porosity, specificv surface area and compressibility.

- Permeability may be defined quantitatively as the flow rate of a liquid of unit viscosity across a unit area of cake having unit thickness under a pressure gradient of unity.
- This equation is valid for liquids flowing through sand, glassv beds and various porous media.
- This model is applied to filter beds or cakes and other types of depth filter.
- This equation is further modified by including characteristics of K by Kozeny-Carman.

3. Kozeny-Carman (K-C) equation

- Kozeny-Carman equation is widely used for filtration.

$$V = A/\eta S^2 * \Delta P/KL * €3/(1-€)^2$$

Where,

ε = porosity of cake (bed)

S = specific surface area of particles comprising the cake m2 / m3

K = Kozeny constant (usually taken as 5)

Limitations:

- It does not consider the fact that depth of granular bed is lesser than the actual path traversed by the fluid.
- The actual path is not same throughout the bed, but it is sinuous or tortuous.

FACTORS INFLUENCING FILTRATION

1. Surface area of filter medium –

2. Pressure drop across the filter medium

3. Viscosity of Slurry

4. Filter Media

Material used as filter media

- **Woven material**
- **Perforated sheet metal**
- **Bed of granular solid built up on supporting medium**
- **Membrane filter media**

5. CARTRIDGES

- **Surface cartridges**
- **Depth type cartridges**

6. Filter Aids

PLATE AND FRAME FILTER PRESS

Principle:

- Mechanism is surface filtration.
- The slurry enters the frame by pressure and flows through filter medium.

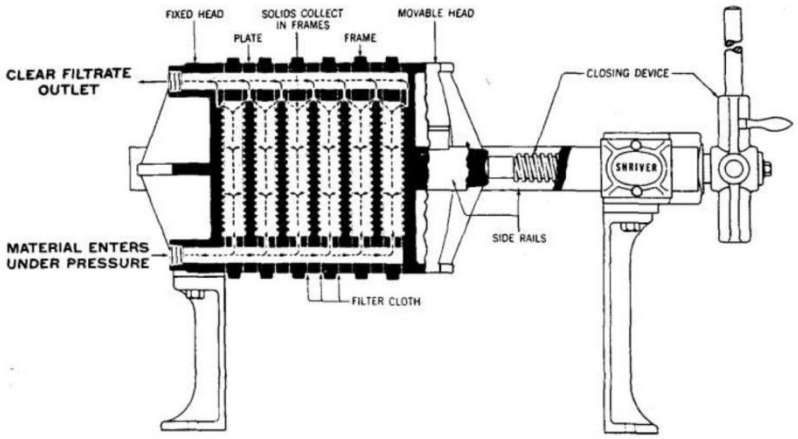


Fig - Filter press

Construction

- The Filter press is made of two types of units, plate and frames.
- Usually made of aluminium alloy.
- Sometimes, these are also lacquered for protection against corrosive chemicals and made suitable for steam sterilization.

Frame-

- It contains a open space inside wherein the slurry reservoir is maintained for filtration and an inlet to receive the slurry.
- It is indicated by two dots in description.
- Frames of different thickness are available.
- It is selected based on the thickness of cake formed during filtration.
- Optimum thickness of frame should be chosen.

Plate

- The plate has a studded or grooved surface to support the filter cloth and an outlet.
- It is indicated by one dot in description.
- Plate supports the filter medium, receiving the filtrate and outlet.
- The filter medium usually cloth is interposed between plate and frame.
- Plate, filter medium, frame, filter medium and plate are arranged in sequence and

clamed to a supporting structure.

- It is normally described by dots as 1.2.1.2.1 so on.
- A number of plates and frames are employed so that the filtration area is as large as necessary.
- Number of filtration units are operated in parallel.
- Channels for slurry inlet and filtrate outlet can be arranged by fitting eyes to the plates and frames, these join together to form a channel.
- In some types only one inlet channel is formed, while each plateν is having individual outlets controlled by valves.

Working

Working can be divided into two steps

1. Filtration operation

- Frame- marked by 2 dots
- Plate – marked by 1 dot
- Slurry enters the frame from the feed channel and passes through the filter medium on the surface of the plate
- The solid forms a filter cake and remain in the frame
- The thickness of the cake is half of the frame thickness, because on each side of frame filtration occurs
- Thus two filter cakes are formed , which meet eventually in the centre of the frame
- The filtrate drains between the projections of the surface of the plate and escape from the outlet
- As filtration proceeds, the resistance of the cake increases and filtration rate decrease
- At a certain point process is stopped and press is emptied and cycle is restarted.

2. Washing of cake (if desirable)

- When washing of cake is also required modified plate and frame filter is used.
- For this purpose an additional channel is included called as washing plate and are identified by 3 dots.
- In the half of the washing plate, there is a connection from washν water cannel to the surface of plate.
- The sequence of arrangement of plates and frames can be represented by dots as 1.2.3.2.1.2.3.2.1 so on (between 1 and 1, 2.3.2 must be arranged.

Procedure for washing the press

- Filtration proceeds in the ordinary way until the frames are filled with cake.
- To wash the filter cake, the outlets of washing plates are closed.
- Wash water is pumped in the washing channel. The water enters through the inlets on the surface of washing plate.
- Water passes through the filter cloth and enters frame which contains the cake. Then water washes the cake, passes through the filter cloth and enters the plate down the

surface

- Finally washed water escapes through the outlet of that plate.

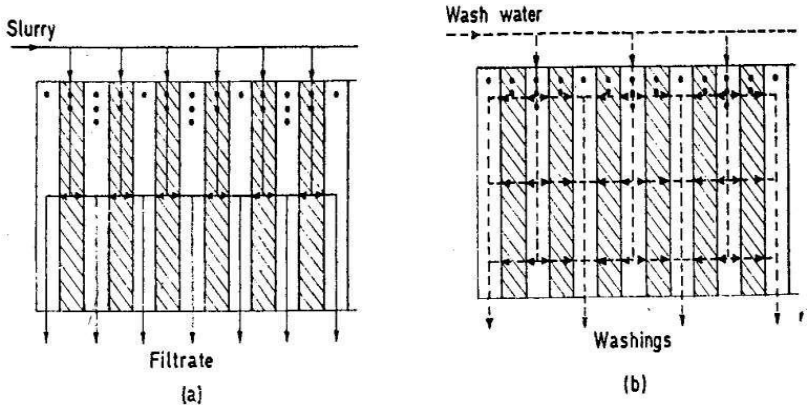


Fig – Diagrammatic working procedure

Advantages

- Construction of filter press is very simple and a variety of materials can be used.
- Provide large filtration area in relatively small floor space.
- The capacity being variable according to thickness of frames and number used.
- Sturdy construction permits the use of considerable pressureν difference. (2000 Kilopascals normally used)
- Efficient washing of cake is possible.
- Operation and maintenance is easy.
- It produces dry cake in form of slab.

Disadvantages

- It is a batch filter, so it is a time consuming.
- The filter press is an expensive filter, the emptying time, the labour involved, and the wear and tear on the cloths resulting in high costs.
- Operation is critical, as the frames should be full, otherwiseν washing is inefficient and the cake is difficult to remove.
- The filter press is used for slurries containing less about 5 %ν solids In view of the high labour costs , it is most suitable for expensive materials e.g. the removal of precipitated proteins from insulin liquors.

FILTER LEAF

Principle:

- It is an apparatus consisting of a longitudinal drainage screen covered with a filter cloth.
- The mechanism is surface filtration and acts as sieve or strainer.
- Vacuum or pressure can be applied to increase the rate of filtration.

Construction:

- The leaf filter is consisting of a frame enclosing a drainage screen or grooved plate.
- The frame may be any shape circular, square or rectangular.
- The whole unite being covered with filter cloth.
- The outlet for the filtrate connects to the inside of the frame through suction.

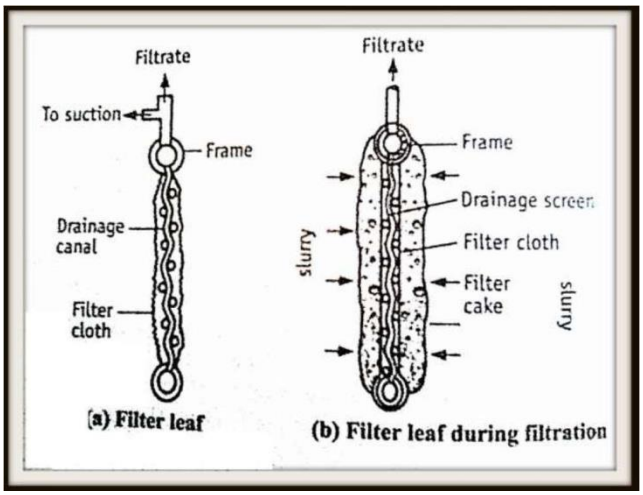


Fig – Filter leaf

Working

- The filter leaf is immersed in the slurry
- Vacuum system is connected to the outlet
- The slurry passes through the filter cloth
- Finally filtrate enters the drainage canal and goes through the outlet into receiver
- Air is passed to flow in reverse direction which facilitates removal of cake

Advantages

- Simplest form of filter used for batch process.
- A number of units can be connected in parallel to increase the surface area of filtration.
- Pressure difference can be obtained either with vacuum or using pressure up to the order of 800 kilopascals.
- Labour costs for operating the filter leaf are fairly moderate.
- The efficiency of washing is high.
- The slurry can be filtered from any vessel.
- The cake can be washed simply by immersing the filter in a vessel of Water.

METAFILTER

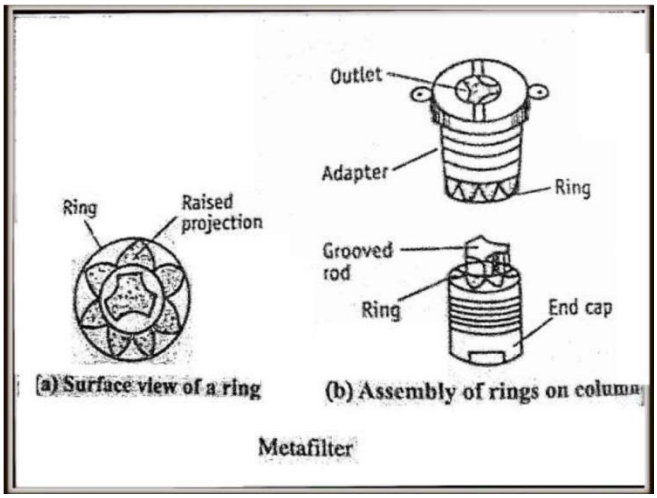
Principle:

- Mechanism is surface filtration.
- In this, metal rings contain semicircular projections, which are arranged as a nest to form channels on the edges.

Construction

- Metafilter consists of a series of metal rings.
- These are threaded so that a channel is formed on the edges.
- It contains a grooved drainage column on which a series of metal rings are packed.
- These rings are usually made up of stainless steel and have dimensions of about 15.0 mm internal diameter and 22.0 mm external diameter.
- Each metal ring has a number of semicircular projections (0.8 mm in thickness) on one side of surface.
- The projections are arranged as a nest to form channels on the edges.
- These rings are tightened on the drainage column with a nut.

- Metafilters are also known as edge filters.



Working

- Filters are placed in a vessel
- Slurry is pumped under pressure or occasionally by applying reduced pressure to the outlet side
- Slurry passes through the channels formed on the edges between the rings
- The clear liquid rises up and collected from the outlet into receiver
- For separation of fine particles, a bed of suitable materials such kieselguhr is first built up.
- The pack of rings serves essentially as a base on which the true filter medium is supported.

Advantages

- Can be used under high pressures, without any danger of bursting the filter medium.
- Running cost is low, as separate filter medium is not used.
- Can be constructed from a material that can provide excellent resistance to corrosion and avoid contamination of sensitive products.
- It is extremely versatile filter because fine as well as large both type of particles can be separated.
- Removal of cake can be carried out by simply back- flushing with water.
- Change over from one batch to another or one product to another is easy.
- Sterile products can be handled.

CARTRIDGE FILTER

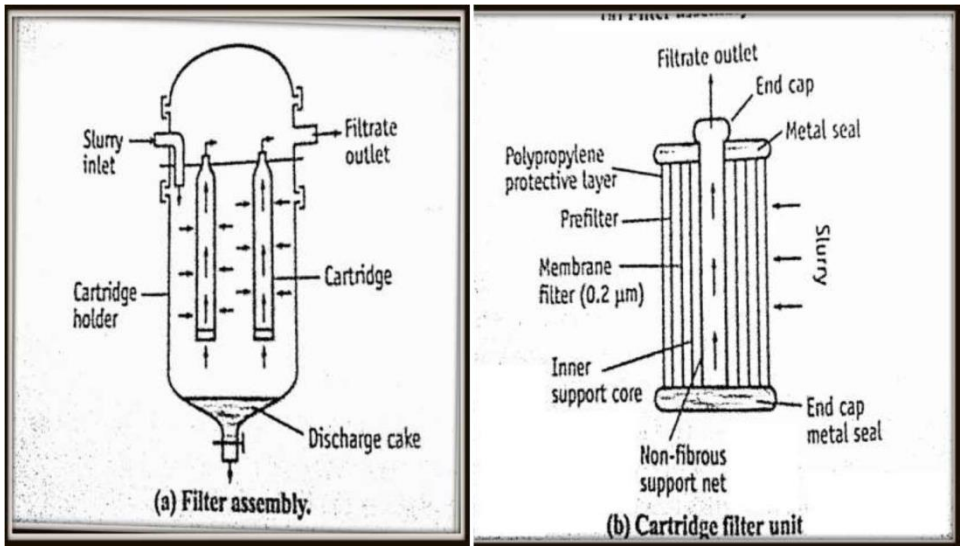
Principle

- It is a thin porous membrane in which pre filter and membrane filter are combined in a single unit.
- The filtration action is mainly sieve like and particles are retained on the surface.

Construction:

- It has cylindrical configuration made with disposable or changeable filter media.
- Made up of either plastic or metal.
- Consist of two membrane filters (sieve like) made of polypropylene: prefilter and actual filter for filtration.
- A protective layer surrounds them.

- The cartridge is housed in a holder and a number of cartridges can be placed in a same housing.
- The housing is closed with the lid.
- Housing has provisions for slurry inlet and outlets.



filterWorking:

- Slurry is pumped into cartridge holder
- It passes through cartridge filter unit by straining
- The clear liquid passes through the centre
- Moves up to collect through outlet

Advantages:

- Autoclaving can be done for sterile operations due to stainless steel construction.
- Cartridges with self cleaning devices are advantageous.
- Rapid disassembling as well as reusing of filter medium is possible.
- Cartridges are not brittle, when they are dry.
- Used as in-line continuous filtration, this reduces handling of solution. It minimizes chances of contaminations.

Disadvantages:

- A number of manufactures provide the components, which are generally not interchangeable between suppliers.
- Cost of disposable elements offsets the labour saving in terms of assembly and cleaning of cartridge clarifiers.

ROTARY DRUM FILTER

Principle:

- Slurry filtered through sieve like mechanism on the rotation drum surface, under the condition of vacuum.
- In addition compression, drying (using hot air), and removing the filter cake (using knife) are possible.

Construction:

- It consists of a metal cylinder mounted horizontally.
- The drum may be up to 3 meters in diameter and 3.5 meters in length and gives surface area of 20 meter square.

- The curved surface being a perforated plate, supporting a filter cloth.
- Internally, it is divided into several sectors and a separate connection is made between each sector and a special rotary valve.

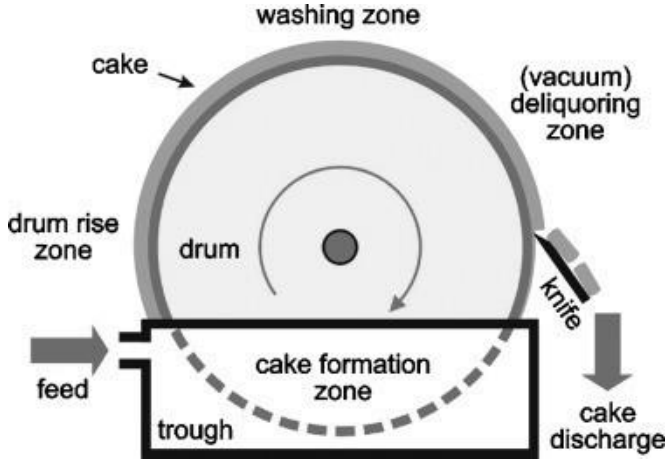


Fig: Drum Filter

FilterWorking

- The drum is dipped into the slurry and vacuum applied to the outlet, which is connected to the filtrate receiver.
- When the cake has formed, the cake drained or partially dried by vacuum.
- The drum is sprayed with water to wash the cake.
- Retaining the vacuum connection drains the cake and produces partial dryness then, removed by a doctor knife.
- When the solids of the slurry are too much that the filter cloth becomes blocked with the particles, a pre-coat filter may be used.
- A pre-coat of filter aid is deposited on the drum prior to the filtration process.

Advantages

- The rotary filter is automatic and is continuous in operation, so that the labour costs are very low.
- The filter has a large capacity, so it is suitable for the filtration of highly concentrated solutions.
- Variation of the speed of rotation enables the cake thickness to be controlled.
- Pre-coat of filter aid could used to accelerate the filtration rate.
- Filter has large surface area.

Disadvantages

- The rotary filter is a complex piece of equipment, with many moving parts and is very expensive.
- In addition to the filter itself, some accessories are connected, e.g., a vacuum pump, vacuum receivers, slurry pumps and agitators are required.
- The cake tends to crack due to the air drawn through by the vacuum system, so that washing and drying are not efficient.
- Being a vacuum filter, the pressure difference is limited to 1 bar and hot filtrates may boil.
- It is suitable only for straight- forward slurries

MEMBRANE FILTERS

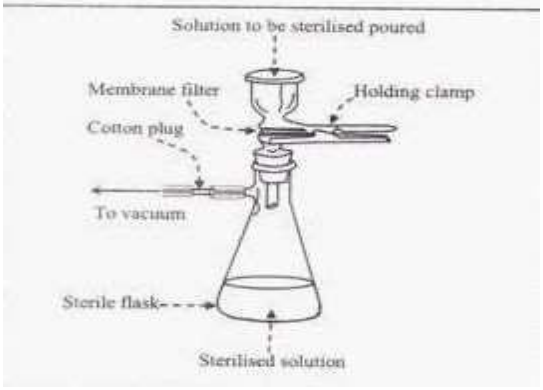
Principle:

Membrane filters act as a barrier to separate contaminants from water, or they remove the particles contaminating the water.

Reverse osmosis, ultrafiltration, and nanofiltration all use a membrane in their different filtration processes.

Construction

- Membrane filters are made of thin and flat membranes of cellulose derivatives, such as, cellulose acetate and cellulose nitrate.
- These filters are brittle when in dry condition and can be stored for an indefinite period.
- The filters are between 50 and 150 µ thick and are available in sizes upto 60 cm^2 .



filterWorking

- A membrane filter has 400 to 500 million pores per square centimetre of filter surface.
- The pores are absolutely uniform in size and occupy about 80% of filter volume.
- To avoid rapid clogging of a membrane, pre-filtration is often required.
- The selection of a membrane filter for a particular application depends on the particles to be removed.

Advantages:

- These filters are mainly used for sterilization of both aqueous and oily liquids.
- The membrane filters cannot be used for filtration of organic solvents, such as alcohols, ketones, esters and chloroform.

SEITZ FILTER

Principle

- It is based on filtration of asbestos pad filter disc

Construction

- It consists of two parts. Lower part fitted with a perforated plate over which

compressed asbestos pad is placed.

- Upper part has a value through which pressure can be applied.
- Both parts joined together by winged nuts.
- A valve is present on the upper part through which vacuum is applied
- The asbestos pads may yield alkali and cause precipitation of alkaloids
- It may shed fibres into the filtrate and absorb drug from solution.

Advantages:

- No risk of contaminating the filtrate.
- Apparatus is very simple to use.
- For viscous solution they are more suitable.

Disadvantages:

- Asbestos may shed loose fibers.
- Pad may absorb sufficient amount of medicament.

CENTRIFUGATION

- Centrifugation is a process which involves the use of the centrifugal force for the sedimentation of heterogeneous mixtures with a centrifuge, used in industry and in laboratory settings.

Applications of centrifugation

- Production of bulk drugs
- Production of biological products
- Biopharmaceutical analysis of drugs
- Evaluation of suspensions and emulsions
- Determination of molecular weight of colloids

CLASSIFICATION OF CENTRIFUGES

Sedimentation centrifuge

It is a centrifuge that produces sedimentation of solids based on the difference in the densities of two or more phases of the mixture.

Filtration centrifuge

It is a centrifuge in which solids pass through the porous medium based on the difference in the densities of the solid and liquid phases

PERFORATED BASKET CENTRIFUGE

Principle:

- Perforated basket (bowl) centrifuge is a filtration centrifuge.
- The separation is through a perforated wall based on the difference in the densities of solid and liquid phases. The bowl contains a perforated side-wall.
- During centrifugation, the liquid phase passes through the perforated wall, while solid phase is retained in the bowl.
- The solid is removed after cutting the sediment by a blade after stopping the centrifuge.

Construction:

- It consists of a basket, made of steel (sometimes covered with vulcanite or lead) or copper or monel or any other suitable metal.
- The basket may have a diameter of 0.90 metres and a capacity of 0.085 metre cube.
- The basket is suspended on vertical shaft and is driven by a motor using suitable power systems such as belt pulleys, water turbines and electric motors.
- Surrounding the basket, a casing stationary is provided which collects the filtrate and discharges it at the outlet.

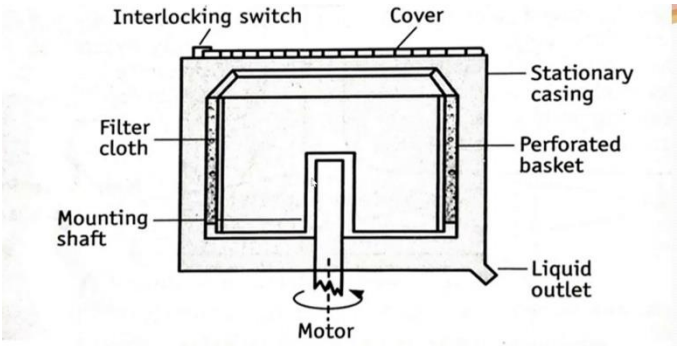


Fig - PERFORATED BASKET CENTRIFUGE

Working:

- The material is kept in the basket when the basket is stationary.
- Power is applied to rotate the basket and maximum speed must be attained quickly. The basket runs at 1000 revolutions per minute.
- During centrifugation, the liquid passes through the perforated wall, while the solid phase retains in the basket. The liquid leaves the basket and is collected at the outlet.
- The cake is then spun to dry as much as possible.
- After a definite period of time, the power is turned off. By applying a brake the centrifuge is stopped.
- The basket is brought to rest. The solid cake is cut using a blade and then unloaded manually.

Advantages:

- The centrifuge is very compact and it occupies very little floor space.
- It can handle slurries with a high proportion of solids and even those having paste like consistency.
- The final product has very low moisture content.
- In this method, the dissolved solids are separated from the cake.
- The process is rapid.

Disadvantages:

- The entire cycle is complicated resulting in considerable labour costs.
- It is a batch process.
- If the machine is adapted for prolonged operation, there is considerable wear and tear of the equipment.

NON-PERFORATED BASKET CENTRIFUGE

Principle:

- This is a sedimentation centrifuge.
- The separation is based on the difference in the densities of solid and liquid phases without a porous barrier.
- The bowl contains a non-perforated side-wall.
- During centrifugation, solid phase is retained on the sides of the basket, while the liquid remains at the top, which is removed by a skimming tube.

Construction:

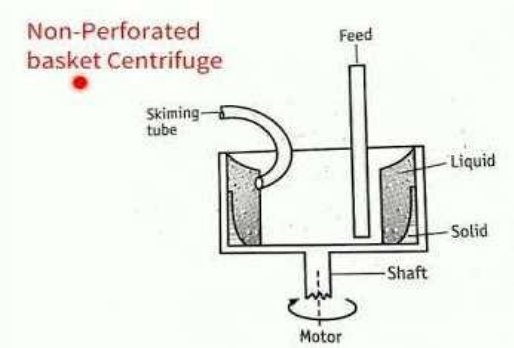


FIG -Non-perforated basket centrifuge

- It consists of a basket, which may be made of steel or any other suitable metal.
- The basket is suspended on vertical shaft and is driven by a motor using a suitable power system.

Working:

- The suspension is fed continuously into the basket.
- During centrifugation, solid phase is retained on the sides of the basket, while liquid remains on the top.
- The liquid is removed over a weir or through a skimming tube.
- When a suitable depth of solids has been deposited on the walls of the basket, the operation is stooped.
- The solids are then scraped off by hand or using a scraper blade.

Advantages:

- Non-perforated basket centrifuge is useful when the deposited solids offer high resistance to the flow of liquid.

SEMI-CONTINUOUS CENTRIFUGE OR SHORT CYCLE AUTOMATIC BATCH CENTRIFUGE

Principle:

- It is a filtration centrifuge.
- The separation is through a perforated wall based on the difference in the densities of solid and liquid phases.
- The bowl contains a perforated side-wall. During centrifugation, the liquid phase passes through the perforated wall, while solid phase retains in the bowl, the solid is washed and removed by cutting the sediment using a blade.
- It is a short cycle automatic batch centrifuge.

Construction:

- It consists of a vertical perforated basket, which is supported from a horizontal shaft driven by a motor.
- From the open side of the basket, provisions are made at the centre to introduce feed and wash pipe through horizontal tubes.
- A feeler rides over the feed, which is connected to diaphragm valve through air supply.
- The feeler controls the thickness of the feed. Hydraulic cylinder attachment is made in such a manner that the discharge chute enters from the sides of basket, when discharge of crystals is desirable.

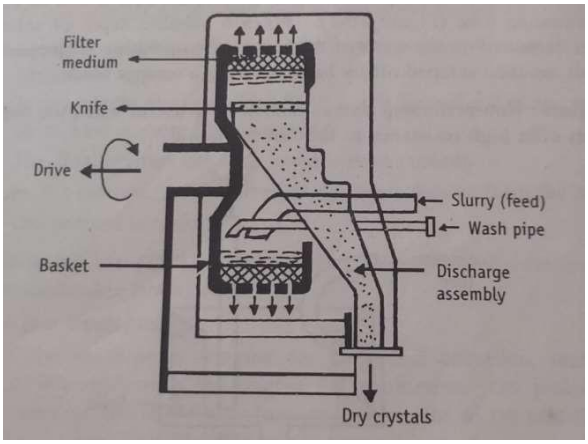


Fig - semi-continuous centrifuge

Working:

- The perforated basket is allowed to rotate and slurry is introduced from the side pipe.
- During centrifugation, the slurry passes through the perforated wall. The solids are retained in the basket, while filtrate leaves the basket, which is collected at outlet.
- Further, the cake is washed with water.
- The wash escapes from the basket through the filtrate outlet
- After achieving the desired thickness (50 to 70 millimetres), the feeler cuts off the air supply to a diaphragm valve that automatically shuts off the entry of slurry.
- The hydraulic cylinder is actuated, which lifts the knife along with the discharge chute.
- The knife does not cut the cake completely down to the screen, but leaves a layer of crystals that acts as a filter medium for further separation in the next cycle.
- The residual crystals may be given a brief wash before starting the next cycle.
- Therefore, the entire cycle is semiautomatic.

Advantages:

- Short-cycle automatic batch centrifuge is used when solids can be drained fast from the bowl.

Disadvantage:

- During discharge, considerable breakage of crystals is possible.
- Construction and functioning is complicated.

SUPERCENTRIFUGE

Supercentrifuge is a continuous centrifuge used for separating two immiscible liquid phases.

Principle:

- It is a sedimentation centrifuge.
- Supercentrifuge is a continuous centrifuge used for separating two immiscible liquid phases.
- The separation is based on the difference in the densities between two immiscible liquids. Centrifugation is done in the bowl of small centrifuge.

Construction:

- It consists of a long hollow cylindrical bowl of small diameter.
- It is suspended from a flexible spindle at the top and guided at the bottom by loose-fit bushing.
- Two liquid outlets are provided at different heights at the top of the bowl, for simultaneous recovery of the separated liquids using modified weirs.

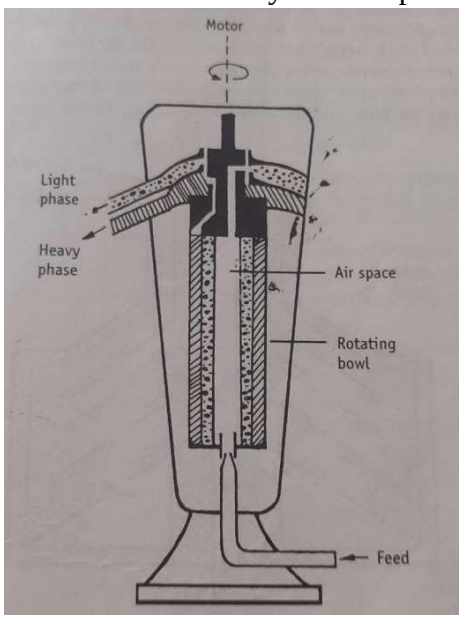


Fig 4 - Supercentrifuge

Working:

- The centrifuge is allowed to rotate on its longitudinal axis at a high frequency usually about 2000 revolutions per minute with the help of drive-assembly.
- The feed is introduced from the bottom of the centrifuge using a pressure system.
- During centrifugation, two liquid phases separate based on the difference in their densities.
- The heavier liquid is thrown against the wall, while the lighter liquid forms an

inner layer. Both liquids rise to the top of the vertical bowl.

- The liquid-liquid interface (the so-called neutral zone) is maintained by an hydraulic balance.
- These two layers are simultaneously separately removed from different heights through modified weirs.
- Thus the supercentrifuge can work for continuous separation of immiscible liquid phases.

Advantages

- It is used for separating liquid phases of emulsions in food and pharmaceuticals.

MATERIALS OF PHARMACEUTICAL PLANT CONSTRUCTION

For manufacturing of pharmaceuticals, bulk drugs, antibiotics, biological products etc., number of equipments are used. The equipments are generally used for processing and packing of products. Some products such as storage of biological products need to be handled carefully. Therefore design of equipment, material selection and fabrication technique need to be considered carefully. These factors affect the success or failure of new chemical plant. The choice based on expert advice, previous experience and laboratory tests.

FACTORS AFFECTING DURING MATERIALS SELECTED FOR PHARMACEUTICALS PLANT CONSTRUCTION

The selection of a material for the construction of the equipment depends on the following properties:

1. Chemical factors

a. Contamination of the products
b. Corrosion of materials of construction

2. Physical factors

a. Strength
b. Mass
c. Wear properties
d. Thermal conductivity
e. Thermal expansion
f. Ease of fabrication
g. Cleaning
h. Sterilization
i. Transparency

3. Economic factors

1. Chemical Factors

Each time a chemical is placed in a container or equipment, the chemical is exposed to the construction material of the container or equipment. Therefore, the material of construction may contaminate the product (*contamination*) or the product may destroy the material of construction (*corrosion*).

a. **Product contamination**:

Iron contamination can change the color of products (such as gelatin capsules), catalyze some reaction that can increase the decomposition rate of the products. The leaching of glass can make the aqueous product alkaline. This alkaline medium may catalyze the decomposition of the product. Heavy metals, such as lead, inactivate penicillin.

b. Corrosion of construction materials

The products can be corrosive in nature. They can react with the material and can destroy it. This can decrease the life of the equipment. Extreme pH, strong acids, strong alkalis, powerful oxidizing agents, tannins etc., reacts with the materials, therefore, some alloys that having special chemical resistance are used.

2. Physicals Factors

a. **Strength**:

The material must have sufficient physical strength to withstand the pressure and stress required. Iron and steel can satisfy these properties. The tablet punching machine, the die and the upperand lower punches are made of stainless steel to withstand very high pressure. Glass, though has strength but fragile in nature. The aerosol container must withstand very high pressure, so tin containers covered with some polymers (lacquered) are used. The plastic materials are weak, so they are used in some packaging materials, such as blister packs.

b. **Mass**:

For transportation, lightweight packaging materials are used. Plastic, aluminum and paper packaging materials are used to package pharmaceutical products.

c. **Wear properties**:

When there is a possibility of friction between two surfaces, the softer surface disappears and these materials contaminate the products. For example, during milling and grinding, grinding surfaces can wear out and contaminate the powder. When pharmaceutical products of very high purity are required, grinding surfaces of ceramic and iron are not used.

d. **Thermal conductivity**:

In evaporators, dryers, stills and heat exchangers, the materials used should have very good thermal conductivity. In this case, iron, copper or graphite tubes are used for effective heat transfer.

e. **Thermal expansion**:

If the material has a very high coefficient of thermal expansion then as the temperature increases, the shape of the equipment changes. This produces unequal stresses and can cause fractures. Therefore, materials that are capable of maintaining the shape and dimension of the equipmentsat the working temperature should be used.

f. **Ease of fabrication**:

During the manufacturing of equipment, the materials undergo various processes, such as casting, welding and forging. For example, glass and plastic can be easily moulded into containers of different shapes and sizes. The glass can be used as coating material for reaction vessels.

g. **Cleaning**:

Smooth and polished surfaces facilitate ease in cleaning. After completing the operation, the equipment is thoroughly cleaned so that the previous product cannot contaminate the next product. The surfaces of glass and stainless steel can be smooth and polished.

h. Sterilization:

In the production of parenterals, ophthalmics and bulk drugs, all equipment must be sterilized properly to avoid microbial contamination of the product. This is usually done by introducing high pressure stream. The material must withstand at high temperature (121^0C) and pressure (15 pounds per square inch). If there are rubber materials, it must be vulcanized to withstand the light temperature.

i. **Transparency**:

In the reactors and fermentors a visual port is provided to observe the progress of the process that takes place inside the chamber. In this case, borosilicate glass is often used. In the parenteral and ophthalmic containers, the particles, if any, are observed with polarized light. The walls of the containers must be transparent to see through it. The glass is used as perfect material.

3. Economic Factors

The initial cost of the equipment depends on the material used. Several materials may be suitable for construction from the physical and chemical point of view, but of all the materials only the cheapest material for the construction of the equipment is chosen. Materials that require a lower maintenance cost are used because in the long term it is economical. The material used for construction of plant is classified as metals (ferrous and non ferrous) and non metals (organicand inorganic).

CORROSION

It is defined as the reaction of a metallic material with its environment, which causes a measurable change to the material and can result in a functional failure of the metallic component or of a complete system. Exposure of surface to air, water and caustic chemicals are the measure causes of corrosion. The surface changes due to corrosion are carried through

the equipment and destroy the performance and fabrication in due course. According to the environmental conditions corrosion can be of dry or wet type as follows:

1. Dry Corrosion: It involves the direct attack of gases and vapor on the metals through chemical reactions. As a result an oxide layer is formed over the surface. This type of corrosion is not common.

2. Wet Corrosion: This corrosion involves purely electrochemical reaction that occurs when the metal is exposed to an aqueous solution of acid and alkali. The moisture and oxygen are also responsible. This type of corrosion is quite common.

e.g. $Zn + 2HCl \rightarrow ZnCl_2 + H_2\uparrow$

THEORY OF CORROSION

1. Corrosion Reaction on Single Metal

A single piece of metal (e.g. Fe) when comes in contact with acid (e.g. HCl) small galvanic cells may be set up on the surface. Each galvanic cell consists of (i) anode regions and (ii) cathode regions.

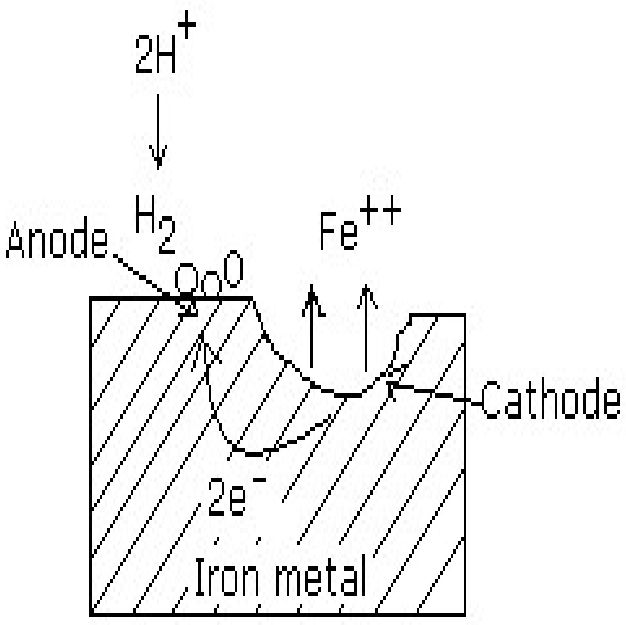


Fig. : Electrochemical mechanism of corrosion

Reaction at anode: Fe on the iron leaves two electrons to the metal and itself becomes Fe^{++} ion. Fe^{++} ion is soluble in water, so it is released in the medium. Thus the iron surface is corroded.

Reaction at cathode: The released electron is conducted through the metal piece into cathode region. Two electrons are supplied to two protons (H^+) to form two atoms of H. Hydrogen atoms are unstable, hence two H atoms will combine to form a molecule of stable H_2. In the

absence of acid, water itself dissociates to generate H^+ ion.

$2H^+ + 2e^- \rightarrow H_2\uparrow$, Hydrogen ($H_2$) forms bubbles on the metal surface. If the rate of hydrogen formation is very slow then a film of H_2 bubbles will be formed that will slow down the cathode reaction, hence the rate of corrosion will slow down. If the rate of hydrogen production is very high then hydrogen molecules cannot form the film on the surface. So the corrosion proceeds rapidly.

2. Corrosion Reactions between Metals

If two metals come in contact with a common aqueous medium then one metal will form anode and the other will form cathode. Now if both the metals are connected with a wire the reaction will proceed. Anode metal will be corroded and hydrogen will form at the cathode.

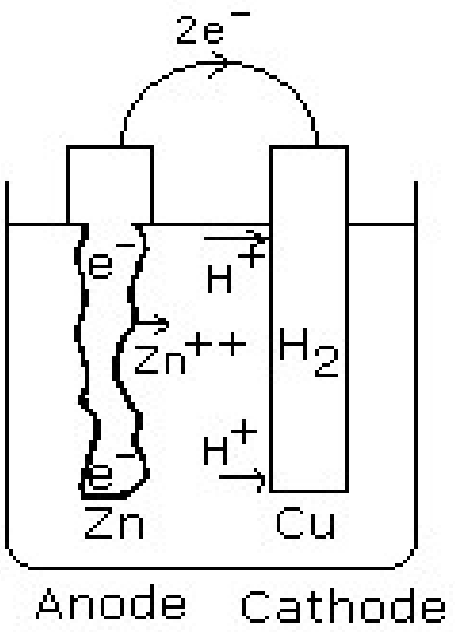


Fig. : Galvanic mechanism of corrosion

For example if a zinc and a copper plate is immersed in an acidic medium then zinc will formanode and will be corroded while hydrogen will be formed at copper plate.

Anode reaction: $Zn \rightarrow Zn^{++} + 2e^-$.

Cathode reaction: $2H^+ + 2e^- \rightarrow H^2\uparrow$

So anode will be corroded and hydrogen will be evolved at cathode.

3. Corrosion Involving Oxygen

The oxygen dissolved in the electrolyte can react with accumulated hydrogen to form water.Depletion (reduction) of hydrogen layer allows corrosion to proceed.

At cathode: $O_2 + 2H_2 \rightarrow 2H_2O$

The above reaction takes place in acid medium. When the medium is alkaline or neutral oxygenis absorbed. The presence of moisture promotes corrosion.

FACTORS INFLUENCING CORROSION

1. pH of the Solution

Iron dissolves rapidly in acidic pH. Aluminium and zinc dissolves both in acidic and alkaline pH.Noble metals are not affected by pH e.g. gold and platinum.

2. Oxidizing Agents

Oxidizing agents may accelerate the corrosion of one class of materials whereas retard anotherclass.

- e.g. O_2 reacts with H_2 to form water. H_2 is removed, corrosion is accelerated. Cu in NaCl solution follows this mechanism also.
- e.g. Oxidizing agents forms a surface oxide (like Aluminium oxide) and makes the surface more resistant to chemical attack.

3. Velocity

When corrosive medium moves at a high velocity along the metallic surface, the rate of corrosion increases because of:

- Corrosion products are formed rapidly and washed away rapidly to expose new surface for corrosion reaction.
- Accumulation of insoluble films on the surface is prevented.
- The corrosion is rapid in the bends of the pipes, propellers, agitators and pumps.

4. Surface Films

- Thin oxide films are formed on the surface of stainless (rusting). These films absorb moisture and increase the rate of corrosion.
- Zinc oxide forms porous films. Fluid medium can enter inside and thus corrosion continues. Nonporous films of chromium oxide or iron oxide prevent corrosion.
- Grease films protect the surface from direct contact with corrosive substances.

TYPES OF CORROSION

1. Fluid Corrosion: General

When corrosion is generally confined to a metal surface as a whole, it is known as general corrosion. This corrosion occurs uniformly over the entire exposed surface area. e.g. swelling, cracking, softening etc. of plastic materials.

2. Fluid Corrosion: Localised

a. **Inter-granular corrosion**:

During heat treatment or welding, some components get precipitated at the grain boundary of themetal.

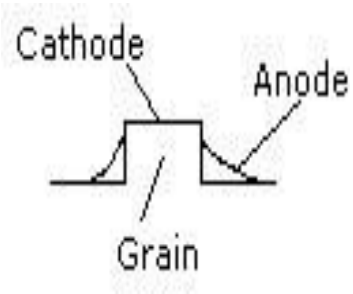


Fig. : Inter-granular corrosion

These boundaries act as anodes and grains as cathodes. So corrosion of anode region occurs.

b. **Pitting corrosion**:

On metal surface small holes or pits are created due to local corrosion and these pits increase insize rapidly. In the pits the metals dissolve rapidly especially by chlorine and chloride ions.

c. **Stress corrosion**: Certain area of metal may be subjected to thermal, mechanical or chemical stresses. The surface area becomes anode and acts as corrosion area.

d. **Fretting corrosion**: Equipment showing high vibrations destroys the surface of metal (e.g. steels balls in ball-bearing) by mechanical hitting.

e. **Corrosion fatigue**: Cyclic stress breaks the protective film, so corrosion increases.

3. Fluid Corrosion: Biological

Metabolic action of micro-organisms can either directly or indirectly cause deterioration of a metal by:

- Creating electrolyte concentration cells on the metal surface.
- Influencing the rate of anodic / cathodic reactions.
- Sulphates are converted in to hydrogen peroxide (H_2S) because of action of reducing bacteria on them. This reacts with iron to produce ferrous sulphide (FeS). Thus the iron gets corroded.

PREVENTION OF CORROSION

Following methods may be adopted for preventing or reducing corrosion:

1. Material Selection

- Pure materials have less tendency towards pitting, but they are expensive and soft. Therefore, only aluminium can be used in pure form.

- Improved corrosion resistance can be obtained by adding corrosion resistant elements. For example inter-granular corrosion occurs in stainless steel. This tendency can be reduced by addition of small amount of *titanium*.

- Nickel, copper and their alloys are used in non-oxidizing environment, whereas

chromium containing alloys are used in oxidizing environment.

- Materials those are close in electrochemical series should be used for fabrication.
- Corrosive materials are taken with suitable material of construction:

Table : List of materials of construction that can withstand the respective corrosivematerials

Corrosive material	Suitable material
Nitric acid	Stainless steel
Hyfrofluoric acid	Monel metal
Distilled Water	Tin
Dilute sulphuric acid	Lead
Caustic	Nickel

2. Proper Design of Equipment

Corrosion can be minimized in the following conditions:

- Design for complete drainage of liquids.
- Design for ease of cleaning.
- Design for ease of inspection and maintenance.
- A direct contact between two metals should be avoided. They may be insulated from one another.

3. Coating and lining: The metals are more prone to corrosion. To combat corrosion in metals, non metals coating or lining should be used. Electroplating, cladding, organic coating can also be used. Galvanic corrosion can be controlled by applying barrier coatings or insulating both the anodes and cathodes to prevent the flow of electrons across the joint. Organic coatings are also used as lining of tanks, piping and shipping containers. Cladding is the bonding of dissimilar metals. It is achieved by rolling of two sheets of metal together. Cladding is also done for steel with an alloy is another approach to combat corrosion.

4. By changing the environment: Corrosion can be prevented by removing air from boiler feed water which prevent steel from the corrosive effect of water. In case of nickel based alloy the pumping of inert gas reduce air or oxygen content. The corrosive effect of acidic media on stainless steel alloys can be minimized by aeration. Corrosion can also be reduced by decreasing the temperature, by reducing the moisture and also by decreasing the exposure time.

5. Use of Corrosion Inhibitors: Corrosion inhibitors are used to decrease corrosion of metals. The inhibitors are used in critical amount (less than 0.1% by weight). For example: Chromates, phosphates and silicates are used to protect iron and steel in aqueous solutions. Organic sulphides and amines are used to protect iron and steel in acidic medium. Copper sulphate is

used to protect stainless steel from corrosion in hot diluted solution of sulphuric acid.

6. Cathodic and Anodic protection: Cathodic protection is achieved by two methods as follows.

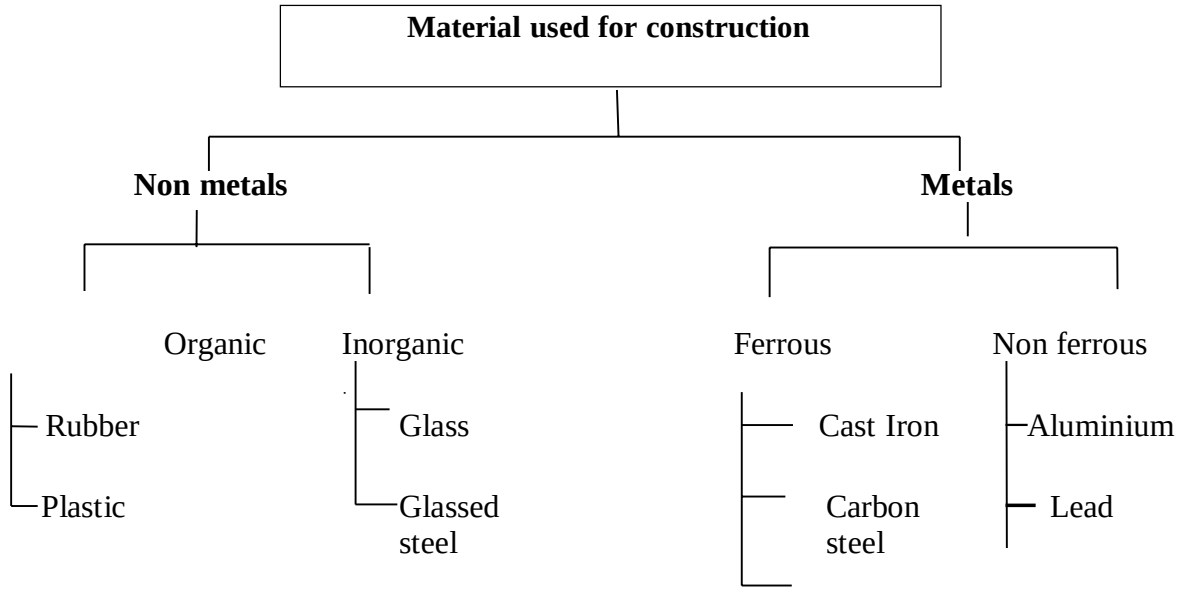
a. **Sacrificial anode methods:** As the name indicates, anodes are kept in contact with protected metal (cathode), this cause scarification of anode. For example: zinc, aluminium, magnesium and their alloys are used as sacrificial anode for protection of iron and steel tanks.

b. **Impressed emf methods:** In this method, external voltage is applied between tanks and electrodes. The anode is maintained always at positive. The natural galvanic effect is avoided. Thus anode is non-consumed. So any metal or non corrodible alloys are used. For example: in case of sulphuric acid and deionised water, anodes are buried in ground while graphite and high silicone steel are compressed. The advantage of this method is: simple, most effective, inexpensive and used to store mild corrosive liquors.

In contrast to cathodic protection, anodic protection is one of the more recently developed methods for controlling corrosion. In anodic protection, predetermined potential is applied to metal. At initial stage, as current increases, metal dissolution or corrosion occur. At critical point passivation occurs. The potential develop at critical point is called passivating potential. Above the passivating potential, current flow decrease to minimum value. This is called passivating current. The main advantage of anodic protection is that it requires small current. This is used in transportation of concentrated sulphuric acid.

CLASSIFICATION OF MATERIALS FOR PLANT CONSTRUCTION

The material used for construction of plant is classified as metals (ferrous and non ferrous) andnon metals (organic and inorganic).



Stainless steel

Fig. : Classification of materials used for construction

1. Non Metals-Organic

a. Rubber

It is used as a lining material

- **Latex:**

Advantages: The latex is ready to use directly outside the container. Latex is economical, exhibits good abrasion resistance and is an elastic mouldable rubber. Latex moulds are also good for casting wax and gypsum.

Disadvantages: Low-cost latex products generally shrink. Making moulds with latex rubber is slow and time consuming. Latex moulds are generally not suitable for melting resins.

- **Polysulfide rubbers:**

Advantages: Polysulfide moulds are very soft, elastic and long lasting, even some have a usefullife of 40 years.

Disadvantages: It has an offensive smell. The polysulfides must be accurately mixed by weight other wise they will not work. Polysulfide rubber costs more than latex.

- **Silicone rubbers:**

Advantages: Silicone rubber has the best release properties of all mold rubbers. The combination of good release properties, chemical resistance and heat resistance makes silicone the best choice for the production of resin castings.

Disadvantages: The silicones generally have a high cost.

- **Polyurethane rubbers**:

Advantages: Polyurethanes are easy to use. They are less expensive than silicones and polysulphides.

Disadvantages: As silicone rubber has the best release properties, urethane rubber has the worst release properties and adheres to almost anything. They have limited shelf life after opening.

b. Plastic

Plastic is commonly used material. It is light in weight. In plastic there are no chances of contamination as in metallic containers. They are available in variety of shapes. But plastic is not preferred in case of higher temperature. Generally pipes and tubing are made of plastic material. They are used for storage of inorganic salt and weak acid. They can be easily cut as per requirement. Plastic do not corrode in air or water. It is also used as insulating material.

Types:

- **Thermoplastic**: They get softened with application of pressure and heat but regain theiroriginal shape on cooling.

Table 2: Different type of thermoplastics and their uses

Thermoplastics	Uses
polyethylene	Cables, buckets, pipes

polypropylene	Milk, carsons, ropes
Teflon	Gaskets, coating
Polyvinyl chloride (PVC)	Manufacturing of gloves

- **Thermosetting**: They are permanently shaped to rigid structure when pressure and heat isapplied. e.g. Phenol-formaldehyde. They cannot withstand on severe abrasion.

2. Non Metals-Inorganic

a. Glass

Glass container is widely used in daily life. It is composed of sand (pure silica), soda ash (sodium carbonate), limestone (calcium carbonate), and cullet (broken glass). Cullet acts as fusion agent for whole mixture. Glass is its solid state is considered as super cooled liquid.

Types:

There are different varieties of glasses used such as

- **Soft glass**: They are made of sodium silicate and calcium silicate. It is used for making glassbulbs and window glasses.
- **Hard glass**: They are made of potassium silicate and calcium silicate. They are used formaking glass apparatus.
- **Flint glass**: They are made of potassium silicate and lead silicate.
- **Quartz glass**: They are made of pure silica. They are used for making silica crucible.
- **Pyrex glass and Jena glass**: They are generally used for laboratory glasswares. The ironoxide is added to give amber colored glass but iron oxide could leach into stored products.

There are 4 types of glasses used in pharmaceutical industries according to I.P.

- **Type1 (Borosillicate glass**): It is highly resistance to alkali leaching. In this alkali and earth cations are replaced by boron. They are less brittle. Easy to clean and sterilize.
- **Type II (Treated soda lime glass**): In this type of glass, surface alkali is neutralized by sulfur dioxide vapors. They are used for making containers for buffered aqueous solution having pH below 7.
- **Type III (Soda lime glass):** It release 10 times more alkaline than type1 and type II. It offers moderate hydrolytic resistance. It is used for dry powder and oleaginous solutions.
- **Type IV (General purpose soda lime glass**): It is not used for parenterals. It is used as container for tablets, oral solutions, suspensions, ointment and liquid for external use.

When glasswares are stored over a month in damp atmosphere having variation in temperature, it leads to Blooming or weathering. As a result salt leach out of glass and appear as fine crystals. In this case salt is washed off with water and acid. Pharmaceutical glass containers should comply with official test for hydrolytic resistance.

Advantages of glass container:

Physical aspect

- They are quite strong and rigid.
- They are transparent which allows the visual inspection of the contents; especially inampoules and vials.
- They are available in various shapes and sizes. Visually elegant containers attract thepatients.
- Borosilicate (Type-I) and Neutral glasses are resistant to heat so they can be readily sterilizedby heat.
- Glass containers can be easily cleaned without any damage to its surface e.g. scratching orbruising.

Chemical aspect

- Borosilicate type of glass is chemically inert. Treated soda lime glass has a chemically inertsurface.
- As the composition of glass may be varied by changing the ratio of various glass constituentsthe proper container according to desired qualities can be produced.
- They do not deteriorate with age, if provided with proper closures.
- Photosensitive drugs may be saved from UV-rays by using amber colour glass.**Economical aspect**
- They are cheaper than other packaging materials.

Disadvantages of glass container:

Physical aspect

- They are brittle and break easily.
- They may crack when subject to sudden changes of temperatures.
- They are heavier in comparison to plastic containers.
- Transparent glasses give passage to UV-light which may damage the photosensitive drugsinside the container.

Chemical aspect

- Flaking: From simple soda-lime glass the alkali is extracted from the surface of the container and a silicate rich layer is formed which sometimes gets detached from the surface and canbe seen in the contents in the form of shining plates – known as 'flakes' and in the form of needles. they are known as 'spicules'. This is a serious problem, especially in parenteral preparations.
- Weathering: Sometimes moisture is condensed on the surface of glass container which can extract some weakly bound alkali leaving behind a white deposit of alkali carbonate toremain over there, further condensation of moisture will lead to the formation of an alkaline solution which will dissolve some silica resulting in loss of brilliance from the surface of glass – called weathering. To prevent weathering, the deposited white layer of alkali carbonates should be removed as early as possible by washing the containers with dilute solution of acid and then washing thoroughly with water.

b. Glassed steel

It is an organic product of fusion. It is cooled to rigid condition without crystallizing. They are used in heavy vessels. It has excellent resistant to all acids. This is suitable in case of transparent pipes.

3. Metals-Ferrous

They are widely used as construction material because it is mechanically strong, easily available and

economical.

a. Cast iron

It is the combination of iron with carbon content greater than 2%. It is cheap and available easilyso greater in demand. It is resistance to concentrated sulfuric acid, nitric acid and dilute alkalis. It has low thermal conductivity. The main disadvantages of cast iron are hard and brittle. Gray cast iron contains carbon, silicone, manganese and selenium. It is easy to mould into any shape. Gray cast iron prevents material from corrosion but it is not preventive against dilute acids. Malleable iron (white cast iron with carbon content 2.5%) is also available and it is also corrosion resistant. Nickel resistant cast iron has also superior toughness, easy to weld, corrosion and as well as heat resistant. A number of cast iron alloys like Duriron and Durichlor are available in market.

Uses:

- It is used to jacketed steam pans.
- It is used as lining material with plastic.

b. Carbon steel

It is an iron alloy having low percentage of carbon content. It is cheapest and easy fabricate. It is most versatile metal used in industry. It is easily weldable and excellent ductility. But carbon steel has limited resistant to corrosion and it also react with caustic soda. Low alloy steel has high mechanical strength. It contains 0.4% Carbon, 0.7% Manganese, 1.85% Nickel, 0.8% Chromium and 0.25% Molybdenum. The properties of carbon steel can be altered by alloying with nickel, chromium and silicone. Carbon steel-Nickel alloy is tough and corrosion resistant. Carbon steel-chromium alloy increases hardness and more resistant to corrosion. At elevated temperature strength of carbon steel can be enhanced by preparing carbon steel-Molybdenum alloy.

Uses:

- It is used for construction of pipes and plates.
- It is used as supporting structure for plant vessels.
- It is used as fabricating material for large storage tanks for water, sulfuric acid and organicsolvents.

c. Stainless steel

It is an alloy of iron. It contain 12 to 30 % Chromium, 0 to 2% Nickel, low percentage of Carbon, Columbium, Copper, Molybdenum, Selenium, niobium, titanium. It is widely used in industries because it is heat resistant, corrosion resistant, easily fabricated, and has high tensile strength.

There are different types of stainless steel available

- **Martensitic (type 410):** It contains 12 to 20% chromium, 0.2 to 0.4% carbon and 2% nickel. It is mild resistant to corrosion and organic exposure. It is less ductile. It is used to prepare sinks, bench tops, storage tanks and mixing elements.
- **Alpha-Ferritic (type 430**): It contains 15 to 30% chromium and 0.1% carbon. It is better resistant to corrosion. It also resistant to oxidation and temperature. It is easy to machine. It is not good against reducing agents and hydrochloric acids. It is used in tower lining, baffles, beat exchangers, tubing, condensers, pump shafts and furnace parts.

- **Austenitic**: It contain 13 to 20% chromium, 0.1% is less than to 0.25% carbon and -22% nickel. It is highly corrosion resistant, east to weld, easily clean and sterilized. It can be easily weld. It is used in fomenters, evaporators, storage vessels, and extraction vesssels.
- **Others**: Type 316, 316L and 317 with 2.5 to3.5% Molybdenum are most corrosion resistant.

4. Metals-Non-Ferrous

a. Aluminium

It is available in large number of alloys. Aluminum is cheap and light in weight. It has adequate mechanical strength. Their maintenance and cleaning is easy. Thermal conductivity ofaluminium is 60% of pure copper. Its tensile strength is 10000 lb/sq. in. It is resistant to corrosion. It can also used for concentrated nitric acid and acetic acid. It is used in wide variety of chemical equipments. But mechanical strength of aluminium decreases greatly above 150 ^{0}C. For food and pharmaceutical uses super grade of aluminium is used. It is used as container for storage of meat. It is used in heat transfer applications. Aluminium alloy with improved mechanical properties and qualities are available which is also corrosion resistant. Aluminium- clad alloy is used for greater mechanical strength. Hot dipped aluminized steel is preferred when sulfur is present. Aluminium is used in biosynthetic processes because it is non toxic to microorganism.

Uses: It is used for manufacturing of container (tank), rail tankers and barrels.

b. Lead

In pharmaceutical industry, Lead is used in less percentage because in large amount it produces toxicity. It is cheap. It is generally used for non food products. The addition of silver (Ag) and copper (Cu) makes lead corrosion resistant and fatigue resistant. Lead has poor structural quality due to low melting point. Therefore antimony is added to harden the lead. Lead pipes are used for solution containing sulfuric acid. The main disadvantage of lead is high coefficient of expansion which may cause permanent deformation.

c. Others

Copper and its alloy are also used in chemical processing because it has high temperature resistance properties. Nickel and its alloy are also used for handling alkalis and storing and shipping of high purity caustic soda and potash. It is also used to store chlorinated solvents and phenols. Titanium is also used as construction material due to strong, corrosion resistant, resistant to hot chloride solutions and nitric acid. But it is costly.