NOTES ON
PHARMACEUTICAL ENGINEERING

THIRD SEMESTER B.PHARM

(As per syllabus of PCI and Kerala University of Health Sciences)

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Contents

FLOW OF FLUIDS

Fluid flow can be defined as the flow of substances that do not permanently resist distortion (pull or twist out of shape). The term fluid here means both liquids and gases. *Fluid is considered as a mass of substance formed by a series of layers.* When any attempt is made to change its shape, these layers may slide over one another until a new shape is attained. That means the fluid is under stress and after sometime it is relived.

IMPORTANCE of flow of fluids in Pharmaceutical Industries

1. In the handling of liquids: Transpiration of materials in the form of solution is simple and cheaper and trouble free than handling solids in Industries. Ex: solvents, solutions and suspensions
2. Handling of solids: Solids can be handled in finely divided state in the form of suspension of fluids so that transportation becomes easy. These kinds of mixtures are called as "fluidized solids."

During handling, the behaviour of liquids changes transiently. (lasting only for a short time/£Wnlamb) Such changes have great influence on heat transfer process, energy losses during pumping of fluids, energy changes in pumping of fluids etc.

Flow of fluids has number of applications in Pharmaceutical Industries. Some of them are,

1. Transfer of reactants in the form of liquids or gases in to the reaction systems
   Ex: In the production of bulk drugs
2. Transfer of air and nutrient broth into the fomenter in fermentation processes.
3. Bottling of liquid dosage forms into suitable containers.
4. Transport of sterile air and water in the production of parenterals
5. Mixing of solids and liquids in the production of suspensions.
6. Filling of semi solid dosage forms in to containers. Ex: Ointments, Pastes and creams

The equipments used for the flow of fluids depend on the flow characters of that particular fluid. Since pipes and channels are widely used for flow purpose, the flow characteristics through pipes and channels are relevant. The rate of flow also is very important in quantify (measure the quantity) additives in an industrial process.

Fluid flow can be divided broadly into two:

1. **Fluid statics** - Study of fluids at rest or equilibrium
2. **Fluid dynamics** - Study of fluids in motion

FLUID STATICS

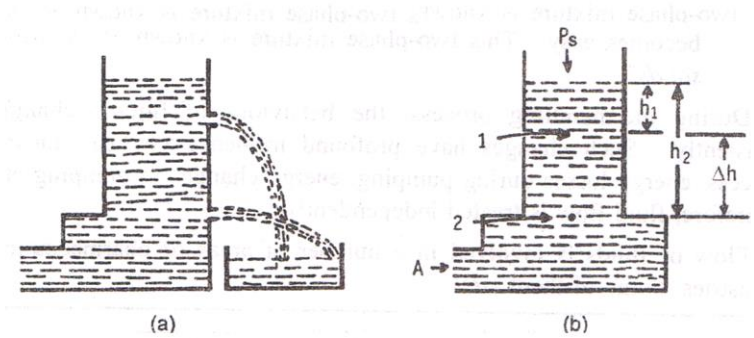
The behaviour of a liquid at rest, the nature of pressure exerts by it (Commonly called as ***Pressure head***) and the variation of pressure at different layers in the liquid etc.are some of the relevant aspects in pharmaceutical field.

Pressure difference between layers of liquids

Consider a column of liquid as show in figure. Two openings are provided to the wall of the vessel at different heights. The rate of flow from these openings is different. This is due to the difference in pressures exerted at different heights.

Consider a stationary column of fluid as shown in figure. The pressure *Ps* pascals *is acting on the surface of the liquid. The stationary column is maintained at constant pressure by applying pressure *P* pascals at point A

Let the cross section of the column be *S* meter square and is uniform from top to bottom. The force or further pressure acting on the liquid at different levels of the liquid column can be determined. The forces (Newton) acting on each side (horizontal components) of the point 1 are mutually nullified. The forces in Newton acting below and above the point 1 are evaluated.



Hydrostatic pressures observed at different layers of a stationary column

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**The unit of Pressure is Pascals (Pa) in S.I Unit*

Force acting on the liquid at point 1 = Force on the surface + force exerted by the liquid above point 1 ……………. (1)

Substituting the force with pressure x area of cross section*(S)* in equation (1) gives:

Pressure at point 1 x surface area = (pressure on the surface x surface area) + mass x acceleration …... (2)

$P_1S = PsS$ + volume x density x acceleration due to gravity

$$= P_1S = P_sS = h_1S\rho g \qquad \text{………………} (3)$$

Since cross sectional surface area is the same, equation (3) may be written as:

$$P_1 = P_s = h_1\rho g \qquad \text{………………} (4)$$

Similarly, the pressure acting on the liquid at point 2 may be written as:

$$P_2 = Ps = h_2\rho g \qquad \ldots\ldots\ldots\ldots\ldots\ldots (5)$$

The difference in pressure can be obtained by subtracting equation (4) from equation (5) as:

$$P_2 - P_1 = g\,(P_s + h_2\,\rho) - (P_s + h_1\rho)g \qquad \ldots\ldots\ldots\ldots\ldots\ldots (6)$$

$$\Delta P = (P_s + h_2\,\rho) - (P_s - h_1\,\rho)\mathrm{g}$$

$$\Delta P = (h_2 - h_1)\,\rho\mathrm{g} = \boldsymbol{\Delta h\rho}\mathbf{g}$$

The pressure difference (Δp pascals) between any two points can be measured by the distance between those points in a fluid. If the density of fluid (p kilogram per meter cube) varies with variation of pressure, an average density could be used. The variation in densities is quite negligible for liquids and gases. Since the difference in the heights (*Δh meter)* is necessary for the measurement, height can be measured from the bottom of the stationary column.

Δ– delta
ρ– rho

APPLICATIONS

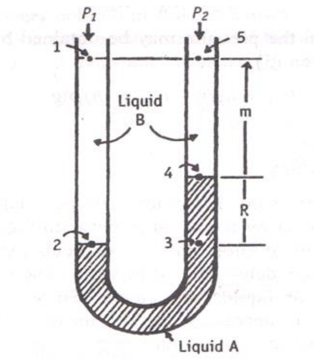
The principles of fluid statics are employed in the working of manometers. They are devices used for measuring the pressure differences.

MANOMETERS

Manometers are devices used for measuring the pressure difference. The different types of manometers are,

1. Simple manometer
2. Differential manometer
3. Inclined manometer (only has limited application and hence not important presently)

Simple manometer



Construction of a simple manometer

Principle and Construction

This is the most commonly used manometer. The construction of a simple manometer is Shown in figure. It consists of a glass U tube filled with a liquid (A) of density, ρ_A, kilogram per meter cube. Above liquid A, the arms are filled with liquid B of density ρ_B kilogram per meter cube. If two different pressures are applied on the two arms, the meniscus of liquid A will be higher in one arm than the other (Figure). Let the pressure at point 1 is P_1 pascals in left hand-side of the limb.
Let the pressure at point 5 is P_2 pascals in the right hand-side of the limb.

From the principles of fluid statics, the pressure difference (Δp) can be finally derived as,

$$\Delta p = R(\rho_A - \rho_B) g$$

Where, g- acceleration due to gravity

ρ_A- Density of liquid A

ρ_B– Density of liquid A

R –Distance from 3 to 4 in meters

- It is easy to measure R value (meters) that is the difference in the levels of liquid A in the two limbs of the instrument.
- The value Δp pascals are independent of the value of m and dimensions of U tube.

When using simple manometer, if the pressure differences of the fluids are large, mercury (high density liquid A) can be used as manometric liquid. If the pressure differences are small, liquids like alcohol, water (for gases) and carbon tetrachloride can be used.

APPLICATIONS

In the measurement of,

1. Consumption of gases in chemical reactions
2. Flow of fluids (in combination with flow meter) Ex: Venturi meter and orifice meter are used for the measurement of pressure head using a manometer. Pitot tubes measures the velocity head.

Differential manometers

Principle and Construction

Differential manometers are useful in measuring small differences and commonly employed in measuring small gas pressures (heads). The construction of a differential manometer is given in figure.

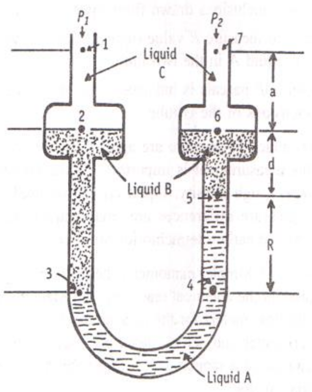
The differential manometer is also known as *two-fluid U-lube manometer*. It contains two immiscible liquids A and B having nearly same densities. The U tube consists of enlarged chambers on both limbs. Hence, the meniscus of the liquid in these enlarged chambers does not change appreciably with changes in the reading *R*.

Using the principle of simple manometers, the pressure difference (ΔP pascals) can be written as:

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

The above equation indicates that the smaller the difference $(\rho_C - \rho_A)$ the larger will be reading on the manometer (R meters) for a given value of ΔP.

Micro manometers based on liquid column principle are available commercially. They measure the reading with extreme precision and sensitivity. These are free from errors due to capillarity and require no calibration, apart from checking the micrometer scale.



Construction of a differential manometer

FLUID DYNAMICS

It deals with the study of fluids in motion

Importance of fluid dynamics in Pharmacy

The study of flow properties of fluid sis important in the manufacturing of dosage forms such as liquid orals, injections, gels, ointments, creams, pastes etc. These systems can change their flow behavior when exposed to different stress conditions. Some of the stress conditions are,

1. In the manufacture of dosage forms: When the materials undergo mixing, flowing through pipes and filled in containers etc
2. At the time of drug administration: The syringeability of the medicines, pouring of liquids, extrusion of ointments from tubes etc. depend on the flow property of dosage forms.

Therefore, the performance of a pharmaceutical product depends on the net effect of all the above mentioned process.

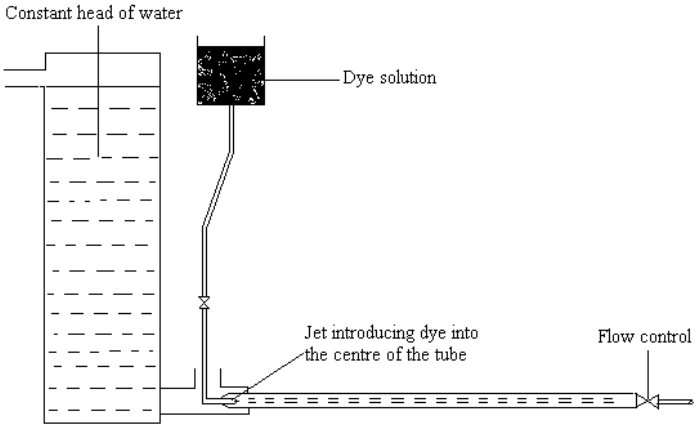
This Study is concerned with the characteristics of fluids that affect their flow properties through pipes and channels as that will occur in a process plant. (Industry) Usually the experimental works are with water, the considerations may be applied to any fluid and the study may include liquid, gases and vapours

MECHANISM OF FLUID FLOW

When a fluid flows through a pipe or channel, the flow characters can be varied according to the concentration. This can be best demonstrated by an experiment with water through a circular glass tube or pipe. (figure)

Osborne Reynolds was the first man who has studied the flow properties of water by this method in 1883. In his experiment, a long glass tube was connected to a reservoir providing a constant head of water, with a control outlet so that the rate of flow could be varied. In the inlet of the tube, a jet was inserted which allowed a coloured liquid (dye) to be injected into the center of the tube.

Reynolds studied the character of flow by varying the conditions based upon the appearance of the thread of coloured liquid. This can be further illustrated by varying the velocity of water through the tube.



Reynold's Experiment

1 When the velocity is low, the thread of coloured liquid remains undisturbed in the centre of the water stream moves steadily along the tube, without mixing. This condition is known as *streamline, viscous* or *laminar flow*.

2. At moderate velocities, a point is reached (Critical velocity) at which the thread begins to wave, although no mixing occurs. This phase is called as the phase of *transitional flow.*

3. As the velocity is increased to high values, eddies begin to occur in the flow, so that the coloured liquid mixes with the bulk of water immediately after leaving the jet. Since this is a state of complete turbulence, this phase is known as *turbulent flow*.

Imagine if the whole system is magnified millions of times, so that the smooth surface of the tube can be considered as a cobbled street and the molecules become rubber balls. Large numbers of these rubber balls are moving the street and when the movement is slow, they will roll along with little disturbance of their relative positions and the flow is *streamlined*. When the movement is rapid, the balls bounce off the cobbles and of each other and their relative positions change continually, giving *turbulent flow*.

Reynolds found that flow conditions were affected by four factors.

1. Diameter of the pipe (D)
2. Velocity of the fluid (υ)
3. Density of fluid (ρ)
4. Viscosity of fluid (η)

All these four parameters were connected in a particular way.

$Re = D\upsilon\rho / \eta$ Re- Reynolds number

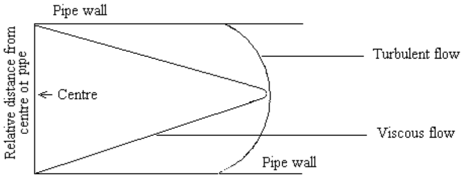
Significance of Reynold's number

1.It is very important in practice to know whether the flow is turbulent or streamline

2.Reynolds number can be used to predict this character of the fluid.

Variation in the velocity of flow across the cross- section of a pipe

1. The flow of the pipe in the middle of the pipe is faster than the fluid near the wall.
2. The velocity of the fluid approaches zero as the pipe wall is approached
3. At the actual surface of the pipe wall, the velocity of the fluid is zero.



Distribution of fluid velocities across the cross section of a pipe

$* \ \eta - eta$

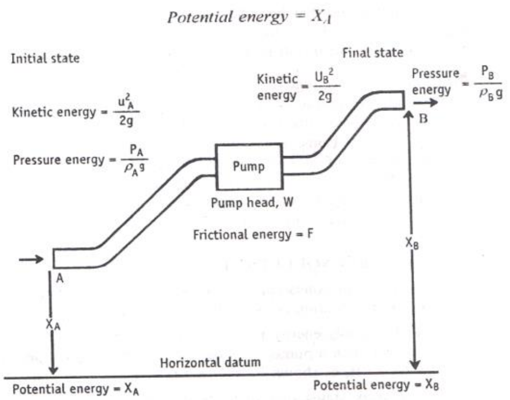
Under viscous conditions, the average velocity over the whole cross section is 0.5 times the maximum. In turbulent flow, it is about 0.8 times the maximum. Changes in roughness, direction, temperature or cross section of the pipe distort the shape and proportions of the velocity distribution curves. Since the velocity of the fluid at the wall surface is zero, there should be some layers in viscous flow near the pipe wall even if the flow is turbulent near the center of the pipe. Thus a buffer layer (a layer which lessens or moderates the impact of) exists between these viscous and turbulent streams which oscillate between viscous and turbulent flow

Bernoulli's theorem

When the principle of conservation of energy is applied to the flow of fluids, the resulting equation is called Bernoulli's theorem.

Pumps are generally employed in conveying fluids from one point to another. Consider such a pump working under isothermal conditions (a line on a diagram connecting points having the same temperature) between points *A* and *B* as shown in figure-6

Bernoulli’s theorem states that in a steady state, ideal flow of an incompressible fluid, (the total energy per unit mass, which consists of pressure energy, kinetic energy), at any point of fluid is constant.



Development of Bernoulli’s theorem

At point *A*, 1 kg (unit mass) of liquid is assumed to be entering. At this point, liquid experiences pressure energy, which can be expressed as follows:

Pressure energy = $P_A / g\rho_A$

P_A- Pressure at point A, *Pa*

g- Acceleration due to gravity m/s

ρ_A – Density of the liquid, kg/m^3

Potential energy (Datum energy) of a body is defined as the energy possessed by the body by virtue of its position or configuration.

Kinetic energy of a body is defined as the energy possessed by the body by virtue of its motion.

Bernoulli’s theorem can be expressed as follows:

$$P_A / g\rho_A + X_A + \upsilon_A^2 / 2g - F + W = P_B / g\rho_B + X_B + \upsilon_B^2 / 2g$$

*υ–

*** *Bernoulli’s theorem is employed in the working of centrifugal pumps, where kinetic energy is converted to pressure head. Also it is applied in the measurement of rate of fluid flow using orifice meter and venture meter.*

Pressure head (Head) and the concept of heads

The pressure head is defined as the height of a column of a liquid of known density, which is numerically equal to the pressure energy term. There are different heads in Bernoulli's equation namely,
Potential heads (X)
Velocity heads $(\upsilon^2/2g)$
Pressure heads $(P / g\rho)$
Similarly, F is known as the friction head and W is known as the head added by the pump.

Energy losses

There are several energy losses experienced by a fluid while flowing through a pipe. Some of them are,

1. Friction losses
2. Losses in fittings
3. Enlargement losses
4. Contraction losses

Friction losses

During the flow of fluids, frictional forces cause a loss in pressure (ΔP_f pascals) Viscous or turbulent nature of the flow also will influence the losses. In general, the pressure drop (ΔP_f) due to friction in a fluid is,

- directly proportional to the velocity of the fluid (υ), m/s
- directly proportional to the density of the fluid (ρ), kg/m^3
- directly proportional to the length of the pipe (L) ,m
- inversely proportional to the diameter of the pipe (D),m

Using the above relations, friction loss in viscous or turbulent flow can be calculated using *Fanning equation:*

$\Delta P_f = 2f\upsilon^2 L\rho / D$ *where* ΔP_f is the pressure drop and f is the friction factor.

The friction factor (f) depends on:

- Nature of flow of the fluid (viscous or turbulent) and
- Roughness of the inner surface of the pipe

The roughness factors for some pipe conditions are given below.

Condition of pipe	Roughness factor
Smooth brass, copper or lead pipe	0.6
New steel or cast iron pipe	1.0
Old steel pipe	1.6
Badly rusted cast-iron pipe	2.5

Losses in Fittings

Normally a large number of fittings are included in a pipeline like tee bows, elbows, couplings unions etc. Fanning equation is applicable when the fluid is passing through a straight pipe. When there are disturbances due to various fittings, there will be additional energy loss due to increased turbulence. It is difficult to specify these losses since a variety of fittings are employed in a pipe line. Instead; it is easy to express these losses as the equivalent length of straight pipe which is given as certain number of pipe diameters

Friction loss of some screwed pipe fittings (Values for turbulent flow)

Fitting	**Equivalent length, pipe diameters**
90 ° elbow ,standard radius	32
Tee(used as elbow, entering branch)	90
Couplings and unions	Negligible
Open globe valve	300
Water meters ,disc	400

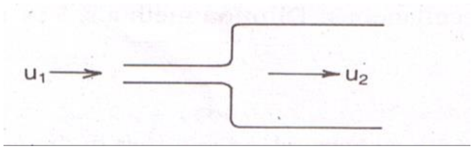
Ex: If a globe valve is fitted to a pipeline having an internal diameter of 5 cm ,the frictional loss due to this fitting will be equivalent to 300 x 5 = 1500 cm = 15 m of the straight pipe.

Enlargement losses

If the cross section of the pipe is increased or decreased gradually, the fluid adapts itself to the changed section without disturbances and there will be no energy loss. However, if the change in cross section is sudden, energy loss occurs due to the formation of eddies. For sudden enlargement with turbulent flow, the loss is given by,

$$\Delta H_e = (\upsilon_1 - \upsilon_1)^2 / 2g_e$$

Where ΔH_e is the loss in head due to enlargement in meters and υ_1 and υ_1 are velocities in smaller and larger cross section in meters per seconds respectively.

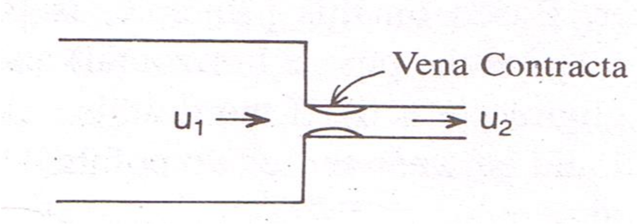


Sudden Enlargement

Contraction losses

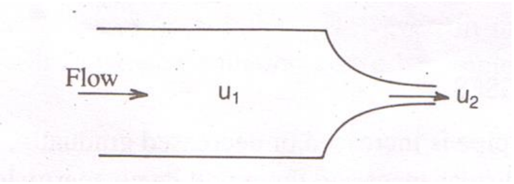
When the cross section is reduced suddenly, the loss in energy is given by,

$\Delta H_c = K \upsilon_2^2 / 2g_c$, where K is a constant. The value of K depends on the relative areas of the two sections. For example, when the ratio of the areas is 0.5, K = 0.3.



Sudden Contraction

For rounded entrance, K for turbulent flow is about 0.04. For laminar flow, it is negligible.



Rounded entrance

MEASUREMENT OF THE RATE OF FLOW OF FLUIDS

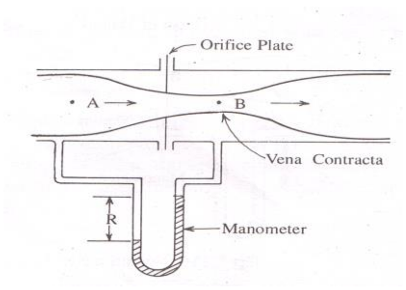
Measurement of the rate of flow of a fluid is important in industry since the materials are handled in the form of liquids or solutions wherever possible. Measurements are required for the purpose of auditing and to calculate the cost of usage also. The methods available can be classified as follows:

1. Direct weighing or measuring methods – *Obsolete and not widely used now.*
2. Hydro dynamic methods
    a. Orifice meter
    b. Venturi meter
    c. Pitot tube
    d. Rota meter
3. Direct displacement meter (Flow meters)

Orifice meter

Principle

The orifice meter is a thin plate containing a narrow and sharp aperture. When fluid stream is suddenly allowed to pass through the narrow constriction, the velocity of the fluid at the orifice meter increases compared to the velocity of the fluid in the upstream. This result in corresponding decreases in pressure head between two points. The differences in pressure can be read out from a manometer.



Orifice meter

Principle and Construction

The orifice meter is considered to be a thin plate containing a sharp aperture through a fluid flows. Normally orifice plate is fixed between long straight pipes, so that other fittings will not alter the flow rate that is being measured. A manometer is connected to the points A and B, the pressure at point B will be less than that of A; (Because the velocity is more at point B) this difference in pressure can be read out from the manometer.

Working

Orifice meter is referred to as a variable head meter. It measures the variation in the pressures across a fixed constriction placed in the path of flow consisting a constant area. The pressure at point A s higher than the pressure at point B (because of more velocity at this point)

Here, the Bernoulli's theorem will be reduced to,

$$\upsilon_B^2 - \upsilon_A^2 = 2g_c(P_A-P_B)/\rho = 2g_c\,\Delta P/\rho = 2g_c\,\Delta H \text{ or } \sqrt{\upsilon_B 2-\upsilon_A 2} = \sqrt{2g_c\,\Delta H}$$

Diameter of the liquid stream would be minimum at point B and would be less than the diameter of the orifice. The point t is known as vena contracta. The diameter of the stream at the vena contracta is not known but the diameter of the orifice is known. Hence if a constant included in the above equation to correct the difference in liquid velocities at point A and B and at the orifice, it can be written as,

$\sqrt{\upsilon_0 2-\upsilon_A 2} = C_0\sqrt{2g_c\,\Delta H}$ where υ_0 is the velocity through the orifice and C_0 is a constant termed as the *coefficient of the orifice*.

Applications

Normally used for testing of steam lines etc.

Venturi meter

Principle

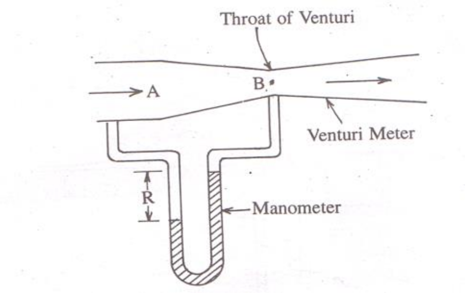
Venturi meter consists of two gradually tapered sections (*throat*) to prevent sudden contraction, which is a disadvantage of orifice meter. When fluid is allowed to flow through the narrow constriction, the velocity of the fluid increases which will results in corresponding decrease in pressure head. The difference in pressure head may be read from a manometer. The velocity of the fluid at the narrow constriction can be written as,

$\upsilon_v = C_v\sqrt{2g.\,\Delta H}$ where υ_v is the velocity at the throat of the venturi *m/s*

C_v - Coefficient of the venturi meter and ΔH - difference in head from the manometer, *m*

Construction

Venturimeter consists of two gradually tapered smooth sections inserted in a pipe line. (Figure-11) Normally venturi meter is placed between long straight pipes, so that other fittings will not alter the flow rate that is being measured. The upper stream cone is shorter than the downstream. There is no eddies formation and hence no power loss. A manometer is connected at points A and B as shown in the figure.



Construction of venturi meter

Working

Venturi meter is referred to as variable head meter. It measures the variable differential pressure across a fixed constriction placed in the path of flow consisting of a constant area. In a venturi meter, the velocity of the fluid at the throat is increased due to narrow constriction. These results in decreased pressure in the upstream cone. This pressure drop are used to measure the rate of flow using a manometer. The equation for venturi meter can be written as,

$\sqrt{\upsilon_v 2 - \upsilon_A 2} = C_v\sqrt{2g.\,\Delta H}$ where, υ_v is the velocity at the throat of the venture *m/s*

υ_v- Velocity at point A (venturi throat) *m/s*

C_v- Coefficeint *(= 0.98)*

This equation can be simplified as,

$\upsilon_B = C_v\sqrt{2g.\,\Delta H}$ *(If the diameter of the smaller cross section is one fifth of the diameter,* $\upsilon_A 2$ *is considered to be small compared to* $\upsilon_v 2$ *. Therefore, the term* $\upsilon_A 2$ *can be disregarded.)*

Applications

Venturi meter is commonly used for liquids, especially water. It can be also used for the measurement of gases.

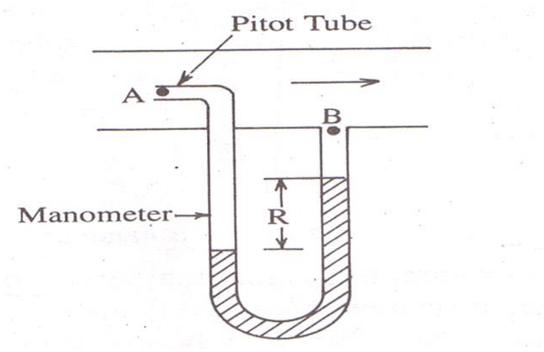
Differences between orifice meter and Venturi meter

S.No.	Orifice meter	Venturi meter
01	Cheap	Expensive
02	Easy to install	Fabrication is highly technical
03	Head losses are more	Insignificant
04	Power losses are more	Power losses are less
05	Used for testing purposes. For example, steam lines	Used in online installations
06	Greater flexibility	Permanent
07	Reading from orifice meter is larger under identical conditions	Reading from venturi meter Comparatively smaller

Pitot tube

Here, while point A measures both kinetic and pressure heads of the flowing fluid, point B which is at right angles to flow measures only the pressure head. (Figure-12) Thus, the manometer measures the velocity head as per the following equation, which is derived from Bernoulli's equation as before.

$\Delta H = \upsilon^2/2g_c$ where is the velocity head of the fluid corresponding to R.



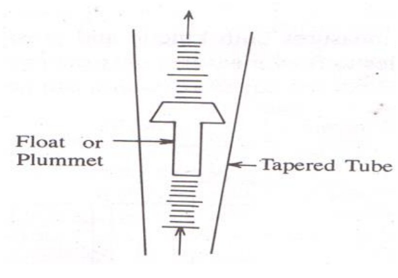
Construction and assembly of Pitot tube

While orifice and venturi meters measures the average velocity of the whole stream of the fluid, pitot tube measures the velocity at one point only.

Rota meter

The orifice and venturi meters measure the pressure difference across a fixed constriction placed in the flow of a fluid and the pressure difference varies with the rate of flow. Hence these are called as variable head meters. In a rotameter, the area of flow is varied in such a way that the head differential is a constant. Hence rotameter is called as variable area meters.

A rotameter (Figure-13) consists of a vertical, slightly tapered tube within which a solid plummet (*a lead ball or other heavy object attached to a line for finding the depth of water or determining the vertical on an upright surface)* or a float smaller in diameter than the narrowest part of the tube is placed. As the flow of the liquid is increased or decreased, the plummet rises and falls freely, thereby varying area of the annular space between it and the tube in such a way that the head loss across this annulus is equal to the weight of the plummet. The tube is usually made of glass and a linear scale is etched on it so that the volumetric flow of the fluid may be directly read the upper edge of the plummet as the index.



The construction of a Rotameter

One great advantage of rotameters is that they give a direct visual index of the flow rates and they have wide range. Instruments are available to record and integrate the rotameter reading and control the flow rates. The scale is nearly linear and the head loss is small and substantially constant over the whole flow range. It requires no straight pipe runs be after the meter. For high precision, a rotameter has to be calibrated with the fluid which is to be measured.

Rotameters are most popular flow meters in chemical process industries like bulk drug manufacture. They are useful in both liquids and gases at low or high pressures. Rotameters are available with pneumatic, electric and electronic transmitters for actuating remote recorders, integrators, and automatic flow controllers.

SIZE REDUCTION

Size reduction is the process of reducing the particle size of a substance to a finer state of subdivision to smaller pieces to coarse particles or to powder. Size reduction process is also referred to as comminution and grinding. When the particle size of solids is reduced by mechanical means it is known as milling.

Raw materials often occur in sizes that are too large to be used and, therefore, they must be reduced in size. The size reduction operation can be divided into two major categories depending on whether the material is a solid or a liquid. If the material is solid, the process is called grinding and cutting, if it is liquid, emulsification or atomization. All depend on the reaction to shearing forces within solids and liquids. Different raw materials vary in their size, shape, brittleness and toughness and the product required may vary from a coarse powder to a powder of the micron size. Different types of machinery are available to suit the specific process requirements.

Objectives of Size Reduction

1. Size reduction increases the surface area of drugs that help in rapid solution formation in the case of chemical substance.
2. The extraction from animal glands such as liver and pancreas and from crude vegetable drugs is facilitated with an increase in surface area because solvent can easily penetrate into the tissues resulting in quick extraction of their active constituents.
3. To increase the therapeutic effectiveness of certain drugs by reducing the particle size e.g., the dose of griseofulvin is reduced to half than that of originally required.
4. The mixing of several solid ingredients is easier and more uniform if the ingredients are reduced to same particle size.
5. In the case of powdered pharmaceutical dosage forms the crystalline drugs are powdered before mixing them with other drugs in order to mix all the drugs uniformly and to avoid recognition of crystalline drugs by the patients.
6. In the manufacture of tablets relatively few drugs can be compressed directly, generally they are mixed with excipients and made in the form of suitable size granules which are then compressed.
7. The stability of emulsions is increased by decreasing the size of the oil globules.
8. Particle size plays an important role in the case of suspensions. If the size of the particles is too small, they may form a cake which may not re-disperse easily but on the other hand large size particles settle quickly but form a loose cake which may re-disperse easily on shaking.
9. The physical appearance of ointments, pastes and creams can be improved by reducing its particle size.
10. All the ophthalmic preparations and preparations meant for external application to the skin must be free from gritty particles to avoid irritation of the area to which they are applied.
11. The rate of absorption of a drug depends on the dosage form, route of administration and particle size. The smaller the particle size, quicker and greater will be rate of absorption.

Factors Affecting Size Reduction

1. **Hardness:** Hardness is a surface property of the material, although it is frequently confused with a property that could be better described as strength. Thus, it is possible for a material to be very hard, but if it is brittle also then size reduction may present no special problems. In general, the harder the material the more difficult it is to reduce in size.

2. **Toughness:** Toughness of a material is sometimes more important than hardness, a soft but tough material may present more problems in size reduction than a hard but brittle substance. Toughness is encountered in many pharmaceutical materials, particularly in fibrous drugs, and is often related to moisture content.

3. **Abrasiveness:** Abrasiveness is a property of hard materials (particularly those of mineral origin) and may limit the type of machinery that can be used. During the grinding of some very abrasive substances the final powder may be contaminated with more than 0.1 percent of metal worn from the grinding mill.

4. **Stickiness:** Stickiness is a property that causes considerable difficulty in size reduction, for material may adhere to the grinding surfaces, or the meshes of the screen may become choked. If the method used for size reduction produces heat, then substances that are gummy or resinous may be troublesome to the size reduction process.

5. **Softening temperature:** During size reduction process, sometimes heat is generated which may cause some substances to soften, and the temperature at which this occurs can be important. Waxy substances, such as stearic acid, or drugs containing oils or fats are examples that may be affected. Some methods can be used to overcome this like cooling the mill, either by a water jacket or by passing a stream of air through the equipment. Another alternative is to use liquid nitrogen.

6. **Material structure:** Some substances are homogeneous in character, but the majority show some special structure, for example, mineral substances may have lines of weakness along which the materials splits to form flake-like particles, while vegetable drugs have a cellular structure often leading to long fibrous particles.

7. **Moisture content:** Moisture content influences a number of properties that can affect size reduction, for example, hardness, toughness or stickiness. In general materials should be dry or wet and not merely damp. Usually, less than 5 percent of moisture is suitable if the substance is to be ground dry or more than 50 if it is being subjected to wet grinding

8. Physiological effect: Some substances are very potent and small amounts of dust have an effect on the operators. To avoid this dust, enclose mills must be used, systems that can extract air are also desirable, and wet grinding also, if possible, as it eliminates the problem entirely.

9. 10. Purity required: Certain types of size reduction apparatus cause the grinding surfaces to wear, and such methods must be avoided if a high degree of purity of product is needed. Similarly, some machines will be unsuitable if cleaning between batches of different materials is difficult.

11. Ratio of feed size to product ratio: Machines that produce a fine may be necessary to carry out the size reduction process in several stages with different equipment, for example, preliminary crushing, followed by coarse grinding and then fine grinding.

12. Bulk density: The capacity of most batch mills depend on volume, whereas processes usually demand solid materials by weight. Hence, all other factors being equal, the output of the machine is related to the bulk density of the substance.

Mechanism and Principles of Size Reduction

The mechanism of size reduction may vary with the nature of the material and each drug may require separate treatment. Fracture occurs preferentially along lines of weakness. Fresh surfaces may be created or existing cracks and fissures may be opened up, the former requiring much more energy. Most of the time there is a tendency towards agglomeration of particles after formation. From the point of view utilization of energy size reduction is a very inefficient process as only a small percentage of the energy supplied is utilized in subdividing the particles. Much of the energy is spent in overcoming friction and inertia of machine parts, friction between particles and deforming the particles without breaking them. This energy is released as heat.

Laws governing energy and power requirement of Size Reduction process

Grinding is a very inefficient process and it is important to use energy as efficiently as possible. Unfortunately, it is not easy to calculate the minimum energy required for a given reduction process, but some theories have been advanced which are useful. A number of theories have been advanced to predict the energy requirements of a size reduction process, but none give accurate results. These theories depend upon the basic assumption that the energy required to produce a change dL in a particle of a typical size dimension L is a simple power function of L:

Kick's theory

$$dE/dL = KL^n \qquad (1)$$

Where dE is the differential energy required, dL is the change in a typical dimension; L is the magnitude of a typical length dimension and K, n, are constants.

Kick assumed that the energy required to reduce a material in size was directly proportional to the size reduction ratio dL/L. This implies that n in equation. (1) is equal to -1. If

$$K = Kf$$

Where K_K is called ***Kick's constant*** and f is called the crushing strength of the material, we have:

$$dE/dL = K_{Kc}L^{'1}$$ Which, on

integration gives:

$$E = Kf_c \log_e(L_i/L_2) \qquad (2)$$

Equation (2) is a statement of ***Kick's Law***. It implies that the specific energy required to crush a material, for example from 10 cm down to 5 cm, is the same as the energy required to crush the same material from 5 mm to 2.5 mm.

Rittinger's theory

Rittinger, on the other hand, assumed that the energy required for size reduction is directly proportional, not to the change in length dimensions, but to the change in surface area. This leads to a value of -2 for *n* in equation. (1) as area is proportional to length squared. If we put:

$K = K_{fc}$

and so

$dE/dL = KfL_{-2}$

Where K_R is called ***Rittinger's constant***, and integrate the resulting form of eqn. (1), we obtain:

$E = Kf_c(1/L- 1/L_1)$ (3)

Equation (3) is known as ***Rittinger's Law***. As the specific surface of a particle, the surface area per unit mass, is proportional to 1/L, eqn. (3) postulates that the energy required to reduce *L* for a mass of particles from 10 cm to 5 cm would be the same as that required to reduce, for example, the same mass of 5 mm particles down to 4.7 mm. This is a very much smaller reduction, in terms of energy per unit mass for the smaller particles, than that predicted by Kick's Law.

It has been found, experimentally, that for the grinding of coarse particles in which the increase in surface area per unit mass is relatively small, Kick's Law is a reasonable approximation. For the size reduction of fine powders, on the other hand, in which large areas of new surface are being created, Rittinger's Law fits the experimental data better.

Classification of Size Reduction equipments

A wide variety of size reduction equipment is available. Equipment may be classified into three classes according to the nature of the forces applied. In the first class, size reduction is accomplished by application of continuous pressure and this class includes equipment for coarse crushing. In the second class, reduction is effected by blow or impact. An example of impact is breaking of a brittle lumpy material by throwing against a wall when the material breaks into pieces due to sudden release of force. In the third type shearing forces are applied by grinding or abrasion and this class gives fine grinding. There is no sharp dividing line between these classes since some mills use a combination of these forces for size reduction. Thus a broad classification could be a crushing, impact and grinding mills.

Before size reduction, materials must be thoroughly dried to avoid accumulation in the mill and possible jamming with wet material and to prevent agglomeration of the particles after size reduction. Crystalline inorganic and organic medicinal compounds which are isolated by normal precipitation and crystallization methods do not require any form of preliminary treatment but they must be thoroughly dried. On the other hand vegetable drugs, due to their wide variation in physical state, may require an initial reduction to small pieces. Materials like camphor and spermaceti, the particles of which tend to cohere as quickly as they are produced, need wetting with alcohol before size reduction to avoid this difficulty.

On the small-scale initial size reduction of vegetable drugs may be done by slicing, rasping or contusion. Slicing or cutting may be done both transversely and longitudinally so that the tissues may be laid open as completely as possible for quicker drying of the material. Rasping or grating can be done with a nutmeg grater and is mainly used for soaps and waxes that are normally required in coarse state. Contusion or bruising is accomplished by beating the drug in a heavy motor. The shape and material of construction of mortars and pestles vary. They may be made of iron, marble, porcelain, glass, steel etc. The bottom surfaces of the mortar and pestle may be shallow or round. Shallow mortars give more grinding effect and are more efficient for size reduction of dry materials and for preparation of fine emulsions.

Grinding and Cutting

Grinding and cutting reduce the size of solid materials by mechanical action, dividing them into smaller particles. The most extensive application of grinding in the food industry is in the milling of grains to make flour, but it is used in many other processes, such as in the grinding of corn for manufacture of corn starch, the grinding of sugar and the milling of dried foods, such as vegetables.

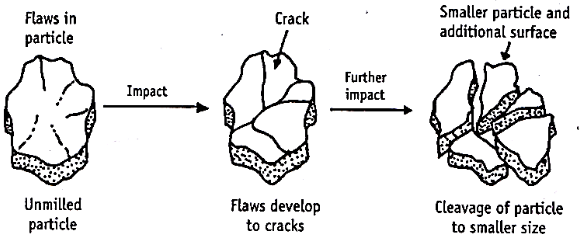
Cutting is used to break down large pieces of food into smaller pieces suitable for further processing, In the grinding process, materials are reduced in size by fracturing them, the material is stressed by the action of mechanical moving parts in the grinding machine and initially the stress is absorbed internally by the material as strain energy. When the local strain energy exceeds a critical level, which is a function of the material, fracture occurs along lines of weakness and the stored energy is released. Some of the energy is taken up in the creation of new surface, but the greater part of it is dissipated as heat. Time also plays a part in the fracturing process and it appears that material will fracture at lower stress concentrations if these can be maintained for longer periods. Grinding is, therefore, achieved by mechanical stress followed by rupture and the energy required depends upon the hardness of the material and also upon the tendency of the material to crack.

The force applied may be compression, impact, or shear, and both the magnitude of the force and the time of application affect the extent of grinding achieved. For efficient grinding, the energy applied to the material should exceed, by as small a margin as possible, the minimum energy needed to rupture the material. Excess energy is lost as heat and this loss should be kept as low as practicable.

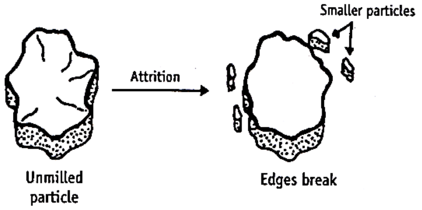
The important factors to be studied in the grinding process are the amount of energy used and the amount of new surface formed by grinding.

Mechanism of Size Reduction

1. **Cutting -** Here the material is cut by means of a sharp blade or blades.
2. **Compression -** In this method, the material is crushed by application of pressure.
3. **Impact -** Impact occurs when the material is more or less stationary and is hit by an object moving at high speed or when the moving particle strikes a stationary surface. In either case, the material shatters to smaller pieces. Usually both will take place, since the substance is hit by a moving hammer and the particles formed are then thrown against the casing of the machine.
4. **Attrition** -In attrition, the material is subjected to pressure as in compression, but the surfaces are moving relative to each other, resulting in shear forces which break the particles.



(a) Mechanism of size reduction when impact type of stress is applied.



(b) Mechanism of size reduction when attrition type of stress is applied.

Mechanisms of size reduction.

Methods of size reduction

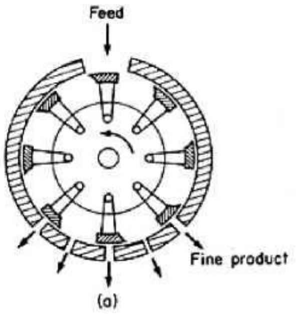
Cutting and compression have limited uses in Pharmaceutical practice, impact and attrition are used much more widely, both separately and in combination, and there is a great variety in each type. The machines used for size reduction are often termed as mills.

Equipments based on the mechanism of Impact

1. Hammer mill- Principle and Construction

In a hammer mill, swinging hammerheads are attached to a rotor that rotates at high speed inside a hardened casing. The principle is illustrated in the figure.

The hammer mill consists of a steel casing in which a central shaft is enclosed to which a number of swinging hammers are attached. When the shaft is rotated the hammers swing out to a radial position. On the lower part of the casing a screen of desired size is fitted which can be easily replaced according to the particle size required. The material is crushed and pulverized between the hammers and the casing and remains in the mill until it is fine enough to pass through a screen which forms the bottom of the casing. Both brittle and fibrous materials can be handled in hammer mills, though with fibrous material, projecting sections on the casing may be used to give a cutting action. The hammer mills are available in various designs and shapes.



Hammer mill

The hammer mill is capable of producing intermediate grades of powder from almost any substance, apart from sticky materials that choke the screen. Applications include the powdering of barks, leaves, roots, crystals and filter cakes. With cutting edges to the hammers, the method has proved to be especially useful for granulation, the damp masses being cut to granules by the hammers when they are found to produce greater uniformity than is obtained by sieve granulation.

Uses

In pharmaceutical industry they are used for grinding dry materials, wet filter cakes, ointments and slurries.

Advantages

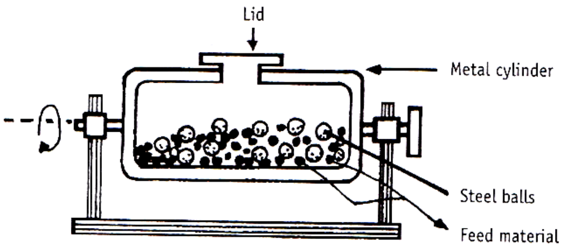
1. It is rapid in action, and is capable of grinding many different types of materials.
2. They are easy to install and operate, the operation is continuous.
3. There is little contamination of the product with metal abraded from the mill as no surface move against each other.
4. The particle size of the material to be reduced can be easily controlled by changing the speed of the rotor, hammer type, shape and size of the screen

Disadvantages

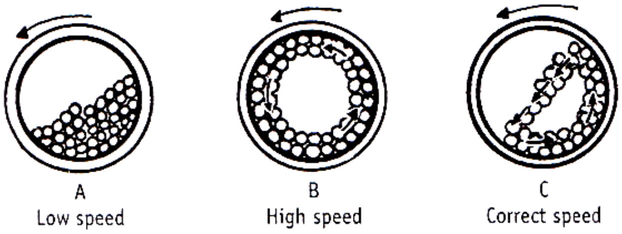
1. The high speed of operation causes generation of heat that may affect thermolabile materials or drugs containing gum, fat or resin. The mill may be water-cooled to reduce this heat damage.
2. The rate of feed must be controlled carefully as the mill may be choked, resulting in decreased efficiency or even damage.
3. Because of the high speed of operation, the hammer mill is susceptible to damage by foreign objects such as stones or metal in the feed. Magnets may be used to remove iron, but the feed must be checked visually for any other contamination.

3. Ball mill -Principle and Construction

Ball mill is a type of crusher, is a cylindrical device used to grind (or mix) materials like ores, chemicals, ceramics and paints. Ball mills rotate around a horizontal axis, partially filled with the material to be ground plus the grinding medium. Different materials are used for media , including ceramic balls, flint pebbles and stainless steel balls. An internal cascading effect reduces the material to a fine powder. Industrial ball mills can operate continuously fed at one end and discharged at the other. Large to medium ball mills are mechanically rotated on their axis, but small ones normally consist of a cylindrical capped container that sits on two drive shafts (pulleys and belts are used to transmit rotary motion). A rock tumbler functions on the same principle. High quality ball mills are potentially expensive and can grind mixture particles to as small as 0.0001 mm, enormously increasing surface area and reaction rates.



Ball Mill



Different speeds of ball mill

The material to be ground is added so that it is slightly more than fills the voids between the pellets. The shell is rotated at a speed which will cause the pellets to cascade, thus reducing particle sizes by impact. It has been proposed that in the plastics industry the term ball mill be reserved for metallic grinding media, and the term pebble mill for non-metallic grinding media.

Ball mills are very effective for grinding smooth, aqueous or oily dispersions by wet grinding since it will give particles of 10 microns or less.

Advantages

1. Ball mill is capable of grinding a wide variety of materials of differing character and of different degrees of hardness.
2. It can be used in a completely enclosed form, which makes it especially suitable for use with toxic materials.
3. It can produce very fine powders.
4. It can be used for continuous operation, and a classifier can be used in conjunction with the mill, so that particles of suitable size are removed while oversize particles are returned.
5. It is equally suitable for wet or dry grinding processes.

Disadvantages

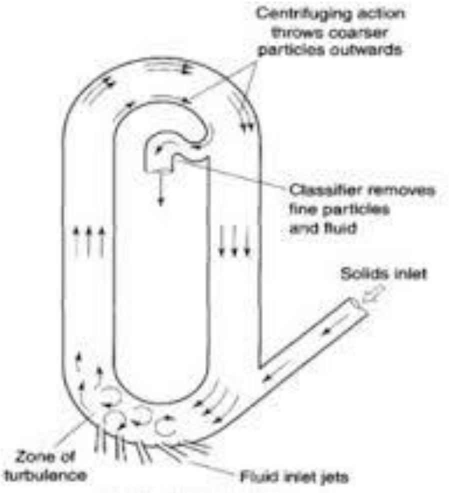
1. Wear occurs, principally from the balls, but partially from the casing and this may result in the product being contaminated, with abrasive materials this may exceed 0.1 percent, but even ordinary substances may be contaminated with 0.03 percent metal after grinding.

1. In some cases, this may not be significant, but in others it may be of great importance.
2. Soft or sticky materials may cause problems by caking on the sides of the mill or by holding the balls in aggregates.
3. The ball mill is very noisy machine, particularly if the casing is of metal, but much less so if rubber is used.
4. Relatively long time of operation.

Ball mills are applicable to a wide variety of materials, large ones being used for grinding ores prior to manufacture of pharmaceutical chemicals and small versions for the final grinding of crude drugs or for grinding suspensions.

Fluid energy mill-Principle and Construction

It consists of a loop of a pipe, which has a diameter of 20 to 200 mm, depending on the overall height of the loop which may be up to about 2 meters, a fluid, usually air, is injected at high pressure through nozzles at the bottom of the loop, giving rise to a high velocity circulation in a very turbulent condition, as shown in figure. Solids are introduced into the stream and, as a result of the high degree of turbulence, impact and attritional forces occur between the particles.



Fluid energy mill

The feed to the mill needs to be pre-treated to reduce the particles size to the order of 100 mesh, enabling the process to yield a product as small as 5 micrometers or less. Despite this, mills are available which are capable of outputs up to 4 mg per hour. A classifier is incorporated in the system, so that particles are retained until sufficiently m fine.

Advantages

1. The particle size of the product is smaller than that produced by any other method of size reduction.
2. Expansion of gases at the nozzles leads to cooling, counteracting the usual frictional heat which can affect heat-sensitive materials.
3. Since the size reduction is by inter-particulate attrition there is little or no abrasion of the mill and so virtually no contamination of the product.
4. For special cases with very sensitive materials it is possible to use inert gases.
5. Having a classifier as an integral part of the system permits close control of particle size and of particle size distribution.

Uses

The method is used where especially fine powders are required, and antibiotics, sulphonamides and vitamins are typical examples.

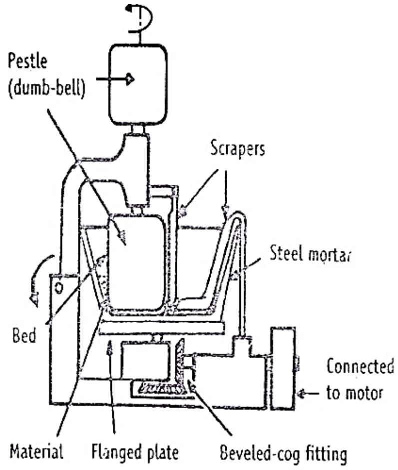
End runner mill

Principle: Crushing due to weight of pestle and shearing also involves.

Construction

1. Consists of mechanical mortar and pestle which can rotate at high speed.
2. Steel mortar, which is fixed to a flanged plate is present.
3. Pestle is dumb-bell shaped.

4. Pestle can be raised from the mortar to facilitate emptying and cleaning



End runner mill

Working

1. The material to be ground is placed inside the mortar.
2. Mortar revolves at high speed the revolving mortar causes the pestle to revolve.
3. Size reduction is achieved by shearing and crushing.
4. The material is passed through a sieve for desired size.

Uses

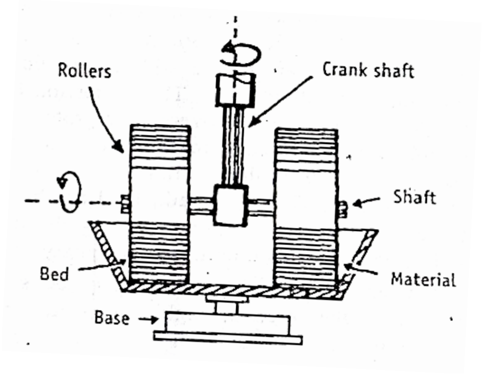
Suitable for fine grinding. Nowadays these mills are outdated.

Edge runner mill

Principle: The size reduction is done by crushing (compression) due to heavy weight of stones. Shearing force is also involved during the movement of stones.

Construction:

1. It consists of two heavy rollers.
2. The rollers move on bed. which is made of stone or iron
3. Each roller has a central shaft and revolve on its axis.
4. The rollers mounted on a horizontal shaft & move around the bed.



Edge runner mill

Working:

1. The stones revolve on its own axis, and at the same time travel round the shallow stone bed.
2. Size reduction achieved by shearing as well as crushing.
3. It is a batch process

Uses:

1. Grinding of tough materials and plant based products to fine powder. More sophisticated mills are used now.

Advantages

Does not require attention during operation.

Disadvantages:

1. Occupies more space.
2. Contamination is more.
3. Time consuming process.
4. Cannot be used for sticky materials
5. Energy consumption is high

The following table shows general characteristics of various types of mills.

General characteristics of various types of mills

S.No.	Name of the mill	Mechanism of action	Uses
1	Hammer mills	Impact	Used for almost all the drugs
2	Roller mills	Attrition &compression	Soft materials
3	Colloid mills	Imapct & attrition	Used for almost all the drugs
4	Ball mill	Imapct & attrition	Brittle drugs
5	Fluid energy mil	Imapct & attrition	Moderately hard and friable materials

Factors influencing choice of Size Reduction machinery

1. **Nature of the raw material:** Drugs must be thoroughly dried before they are subjected to size reduction. Drugs like belladonna, gentian, liquorice and squill are returned to the drying rooms to the drying rooms after they have been partially comminuted, as they rapidly absorb moisture from the atmosphere and as the moisture may not be removed from all parts of the drug during the initial drying stage. In the case of water insoluble substances, wet grinding of the material can be done. Fibrous material seed tearing apart of the fibres and this can be achieved in high-speed impact mills. The replacement of air by an inert gas is necessary when grinding readily oxidizable materials and ball mills and high-speed impact mills are generally used in this way.

2. **Nature of the product:** A powdered drug produced in a ball mill differs from the product in a micronizer or a disintegrator even though both powders may be screened to the same fineness. Differences exist in the shape of the particles, their toughness and their internal pore structure. Powder obtained from grinding mills like ball mill is more compact and less porous than that obtained in high speed impact mills like a disintegrator. For the grinding of a sterile material under aseptic conditions, a mill is required that can be easily sterilized and sealed to prevent contamination during the grinding operation. When iron and copper contamination of pharmaceutical products is to be avoided stainless steel or ceramic material may be used for the surfaces of a mill coming into contact with the drug.

3. **Degree of comminution required:** The degree of comminution of materials varies according to the purpose for which they are required. For the preparation of galenicals, the size of the vegetable drug powder employed for extraction varies from coarse to fine powder. Tincture preparation requires bruised to moderately coarse powders. For percolation, drug powders should not contain a large proportions of fines to avoid uneven extraction. Coarse to moderately coarse powder, with a minimum of fine powder, are all most easily obtained by using high speed mills of the impact type. Materials such as cascara, liquorice, belladonna leaf and root and ginger are all easily broken down in such mills. The rate of absorption of sparingly soluble drugs, either through the Gastro-intestinal tract or when administered parenterally, depends on particle size. Penicillin in a medium of aluminium monostearte and arachis oil appears to be most effective when 90 % of the particles are smaller than 5 micrometers. The relatively insoluble sulphonamides attain their maximum antibacterial activity at crystal sizes of about 1 micrometer or below. For insufflations the drug should be smaller than about 5 micrometers.

Pharmaceutical Applications of Size Reduction

1. Size reduction increases the surface area per unit weight also known as the specific surface area. This increased specific surface affects the therapeutic efficiency of medicinal compounds that possess a low solubility in body fluids by increasing the area of contact between the solids and the dissolving fluid. For example, the control of fineness of griseofulvin led to an oral dosage regimen half that of the originally marketed product.

2. Particle size control influences the duration of adequate serum concentration, rheology, and product syringeability of a suspension of penicillin G procaine for intramuscular injection.

3. The rectal absorption of aspirin from a theobroma oil suppository is related to particle size.

4. There is an increase in antiseptic action for calomel ointment when the particle size of calomel is reduced.
5. The size of particles used in inhalation aerosols determines the position and retention of the particles in the broncho pulmonary system.

6. Size may affect texture, taste and rheology of oral suspensions in addition to absorption.

7. The time required for dissolution of solid chemicals in the preparation of solutions is shortened by the use of smaller particles.

SIZE SEPERATION

Size separation is a unit operation that involves the separation of a mixture of various sizes of particles into two or more portions by means of screens. It is also known as ***sieving, classifying or screening.*** This technique is based on physical differences between the particle such as the size, shape and density etc.

Screening is a method of separating particles according to the size. Sieves can be used for this purpose. The material that remain on the given surface (sieve) is known as ***oversize or plus material***. The material that passes through the screen is known as ***undersize or minus material***.

Applications of size separation

1. To determine particle size and size distribution, which are useful in the production of tablets or capsules.
2. As a quality control tool for the analysis of raw materials such as griseofulvin (antibiotic) and aspirin (NSAID)
3. To test the efficiency of a size reduction equipment or process.
4. To optimize the process conditions such as method of agitation, time of screening, feed rate etc.

OFFICIAL STANDARDS FOR POWDERS

Powders are generally classified into two

1. Coarse powder
2. Fine powder

As per I.P 1996, powders are classified as follows.

S.No	Grade of powder	Sieve through which the particle must pass	Nominal mesh aperture size in microns	Sieve through which 40% particles pass	Nominal mesh aperture size in microns
01	Coarse Powder	10	1.7	44	355
02	Moderately Coarse Powder	22	710	60	250
03	Moderately fine Powder	44	355	85	180
04	Fine Powder	85	180	-	-
05	Very fine Powder	120	125	-	-

When fineness of a powder is indicated by means of a number, it means that all particles of the powder shall pass through the sieve of which the mesh aperture in microns is equal to that number.

Normally, fine powders are used for the preparation of compound powders. Moderately coarse powders are used for the preparation of tinctures. Coarse powders without fines are employed in percolation process. In general powders of above mentioned terminology is applied in the production of galancials. I.P has prescribed lower and upper limits for three coarse powders and for two fine grades, it has prescribed only the upper limit.

Examples of crude drugs and nature of powder required

Liquid extract	Useful parts	Grade of powder
Ashoka	Stem, bark	Coarse
Nux vomica	Seeds	Moderately coarse
Rauwolfia	Roots	Moderately coarse
Ergot	Sclerotia	Moderately fine
Ipecac	Root	Fine
Ephaedra	Stem	Fine

SIEVES

Sieves are the simplest and most frequently used equipment for size separation.

Construction

1. Sieves for pharmaceutical testing are constructed from wire cloth with square meshes, woven from wire of brass, bronze, stainless steel or any suitable materials.

2. Sieves should not be coated or plated.

3. There should not be any reaction between the material of the sieve and the substance to be sieved.

Types of sieves

The size and shape of aperture opening is the most important factor to be considered in a sieve. Commonly used pharmaceutical sieves include,

- **Woven wire sieves**
- **Bolting cloth sieves**
- **Closely spaced bars(Screens)** and **- Punched plates**

Woven wire sieves

They are general purpose sieves and widely used in pharmaceutical practice. The types of woven sieves include,

Plain weave and Twilled weave

The nature of sieve surfaces is shown in the following figure. For sieving of fine powders metal wire woven sieves are used. Ex: Hand sieves. They can be attached in roller mill, ball mill etc. during milling. In case of coarse sieves, the wire is generally given a double crimp to preserve the alignment of the wire.

(a) Plain weave sieve (b) Twilled weave sieve

Bolting cloth sieves

Silk, nylon and cotton are generally woven from twisted multi strand fibres. These are mainly used for the separation of fine powders.

Ex: **Hummer screens.**

Bar Screens

Usually used in the handling of large and heavy materials. These bars are fixed in parallel positions and held by cross bars and spacers.Ex: **Grizziles**

Perforated Screens or punched plates

Used to separate coarse powders. These are made of sheet of metals with perforated clothes. The holes may be round, oval, square or rectangular. Ex; Hammer mill uses this type of screens

Perforation shape	Recommended use	Characteristics
Round holes	Fibrous materials	Clogs more quickly; lower hole size is limited because of structural strength
Herringbone(Slot) screens	Amorphous and crystalline substances	Slightly coarse powder than equal diameter round perforations
Cross slots	Amorphous materials and slurries with coarse particles	Same grind size as of equal sized round perforation; finer slot size attainable than round perforations.

STANDARDS OF SIEVES

1. Tyler standard sieve series(U.S.A)
2. US standard sieve series
3. British standard sieve series(U.K)
4. German DIN(Deutsche Industrienormein) in Germany and Europe
5. I.P Standard sieve series in India
6. International test sieve series (ISO) world wide

01. Sieve number

Sieve number indicates the number of meshes per linear length of 25.4 mm.

02. Nominal size of apertures

This indicates the distance between two adjacent wires. IP 1996 gives the nominal mesh aperture size in m or μm.

03. Nominal diameter of the wire

Wire mesh sieves are made from wires having a specific diameter in order to give a suitable aperture size and sufficient strength to avoid distortion of the sieve.

04. Approximate percentage sieving area

Generally, the sieving area is kept within the range of 35 to 40 percent in order to give suitable strength to the sieve.

05. Aperture tolerance average size

Some variation in aperture size is unavoidable. The variation is expressed in terms of aperture tolerance average. (Refer I.P standards for sieves specifications)

IDEAL AND ACTUAL SCREENS

An ideal screen sharply separates the feed mixture in such a way that the smallest particle in the oversize would be just larger than the largest particle largest particle in the undersize.

Ideal separation defines a diameter called cut diameter, D_{pc} that makes a point of separation between the fractions. Usually D_{pc} is equal to the mesh opening of the screen.

Actual screen does not give perfect separation about the cut diameter. Here the overlap between the smallest particle in oversize and the largest particle in undersize is more pronounced, when particles are,

- Needle like fibers
- Tend to aggregate
- Tend to strike the screen surface endwise and pass through
- Tend to strike the screen sidewise and retained

MODES OF MOTION IN SIZE SEPERATION

Screening is a method of separating particles according to the size alone. The basic technique is passing the particle through a series of sieves of uniform size. Here 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. This can be hastened by inducing some type of motion (movement) to the particles.

Size separation is basically assisted by three methods.

1. **Agitation**
2. **Brushing**
3. **Centrifugal forces.**

These modes of shaking help to shake the material so that sieving will be quick and entire sieving area can be utilized.

AGITATION

Sieves are agitated in a number of ways. Some of them are discussed below.

Oscillation

The sieve mounted in a frame that oscillates back and forth in a reciprocal motion. The motion is parallel to the plane of the sieve. The sieves can be slightly inclined. The reciprocating motion is induced by means of an ordinary eccentric on a rotating shaft.

Vibration

- The sieve is vibrated at high speed by means of an eccentric device (electric vibrator)
- Rapid vibration is imparted into the particles that help the powder to pass through the sieve.

Gyration

Here the sieve is on rubber mounting and connected to an eccentric flywheel. This give a rotary movement of small amplitude to the sieve, which in turn gives spinning motion to the particles that helps them to pass through the sieve.

ADVANTAGES

- Agitation methods are simple, rapid and inexpensive

DISADVANTAGES

- Agitation methods have the lower limit of particle size.
- If the powder is not dries, apertures become clogged with particles leading to improper sieving.
- During agitation, attrition occurs causing size reduction.

BRUSHING METHOD

A brush is used to move the particle son the surface of the sieve and to keep the meshes clear. The brush is rotated in the middle in the case of a circular sieve, but spiral brushes are rotated on the longitudinal axis in case of horizontal cylindrical sieve. **Ex: Brush Swifter**

CENTRIFUGAL METHOD

A high speed rotor is fixed inside a cylindrical sieve, so that on rotation the particles are thrown outwards by centrifugal force. The air current can be generated by means of a jet of air into the equipment, which helps in separating the particles. Ex: Cyclone separator, Air separator

Testing of a Powder-Sieve Analysis-Theory and Principle

Pharmaceutical powders ideally should contain particles of one size (mono size), which is not possible by any size reduction method. Al least, the powder should contain narrow particle size distribution. Therefore, these materials are controlled to meet official specifications. For this evaluation, sieving method is used. The basic principle involved in this method is size separation using standard sieves or screens.

Size separation is a unit operation that involves the separation of various sizes of particles into two or more portions by means of screening surfaces.

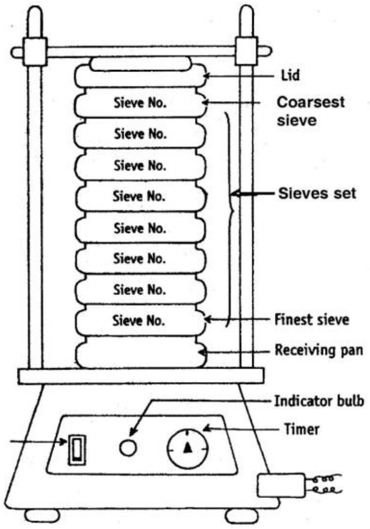
Size separation is also known as *sieving, sifting, classifying,* or *screening.* From the theoretical considerations, screen may be ideal or actual.

An *ideal screen* would sharply separate the feed mixture (material) in such a way that the smallest particle in the oversize would be just larger than the largest particle in the undersize.

An *actual screen* is the one that does not give perfect separation about the cut diameter.

Sieving method involves size separation process. Standard sieves of different sieve numbers are available commercially as per the specifications of IP and USP (see table). Sieves are constructed from wire cloth with square meshes, woven from wire of brass, bronze, stainless steel, or other suitable materials. These sieves are arranged as a nest in the gyratory sieve shaker.

Sieve Shaker Machine



Applications

Sieving method finds applications in the production **of** tablets and capsules. Normally, 15% of fine powder (passed through 100-mesh sieve) should be present in a granulated material to get a proper flow of material and achieve good compaction in tabletting. Therefore, per cent of coarse and fine powder can be quickly estimated using sieving method.

Milled material is subjected to size separation in order to obtain the
Powder of desired size or size distribution. Size distribution analysis is
Important in different areas as mentioned below:

* Quality control tool for the analysis of raw materials.
* Testing the efficiency of a size reduction equipment or process.
* Optimizing the process conditions such as method of agitation, time of screening etc.
* Selecting the sieve system for commercial equipment.

Errors can arise if the sieves are overloaded or if insufficient allowed for the particles to pass through. The size distribution in powdered drug also influences the absorption of drug.

Advantages

1. Sieving method is inexpensive, simple, and rapid with reproducible results.

2. This method is useful when particles are having *50* to I size range.

Disadvantages

1. Lower limit of the particle size is *50* mm.

2. If the powder is not dry, apertures become clogged w des, leading to improper sieving.

3. During shaking, attrition (particles colliding with ea occurs causing size reduction of particles. This leads to estimation.

Factors Influencing Sieving

Factors influencing sieving are weight of sample, duration of shaking, and type of motion. The types of motion influencing sieving are vibratory motion (most efficient), side-tap motion, bottom-pa rotary motion with tap, and rotary motion. The type of motion and intensity of the shaker are fixed and standardized. Care should in order to get reproducible results.

Principle

Sieving method directly gives weight distribution analysis. From the data, d_{sieve}, can be calculated.

Sieve diameter is defined as the diameter of a sphere that through the sieve aperture as the asymmetric particle.

Sieve diameter is applied to those particles that pass through a sieve. It is useful for establishing size distribution analysis. On the other hand, powder is expressed by average particle diameter.

The powdered drug is separated according to its particle size number of sieves in a nest. These are subjected to different types of agitation in sieve shaker, so that size separation is rapid. Sieves are arranged in a nest with the coarsest at the top. A sample (100 powder (or granules) is placed on the top sieve. This sieve set is fixed to the mechanical gyratory shaker and shaken for a period of time (05 minutes). The powder retained on each sieve is weighed. Then normal weight distribution curve is constructed.

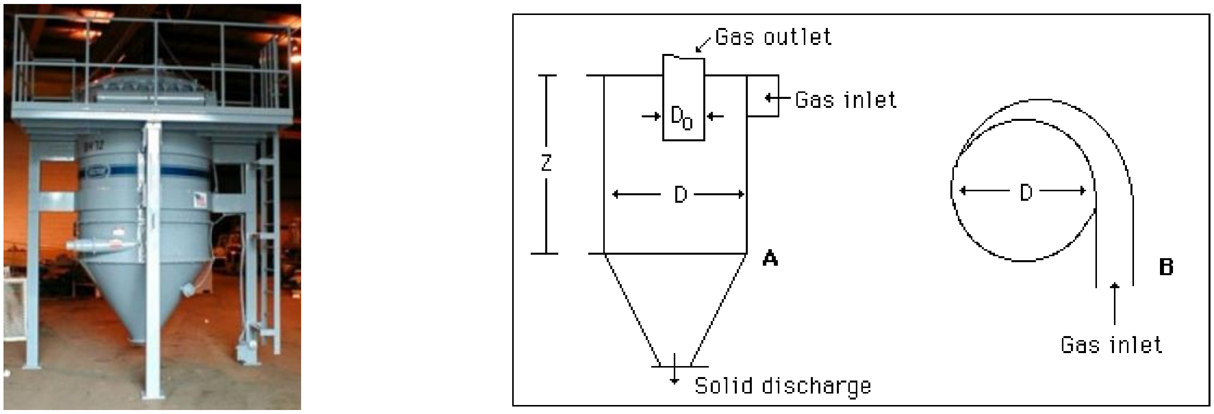
Cyclone seperator

Principle

Centrifugal force is used to separate the solids from fluids. Depend on the fluid velocity, the cyclone separator can be used separate all types of particles.

Construction

- ✓ Consists of a short, vertical, cylindrical vessel with a conical base.
- ✓ The upper part of the vessel is fitted with a tangential inlet. The outlet is arranged at the base.
- ✓ Fluid outlet is provided at the center of the top portion, which extends inwardly to the separator.



Cyclone separator

Working

1. The solids to be separated are suspended in a stream of gas (usually air)
2. Such a feed is introduced tangentially at a very high velocity, so that rotary movement takes place within the vessel
3. The centrifugal force and vortexing throw the solids to the wall
4. As the speed of air diminishes the particles fall to the conical base and are discharged through the solid outlet.
5. The fluid air can escape from the central outlet at the top.

Uses

1. Cyclone separator is used to separate solids from gases

2. Size separation of solids in liquids
3. Separation of heavy or coarse fraction from fine dust.

Air separator

It works on the same principle as that of cyclone separator. But in this case the air movement is obtained by means of rotating disc and blades. To improve the separation, the stationary blades are used. By controlling these blades and the speed of rotation, it is possible to vary the size at which separation occurs.

Construction

It consists of a cylindrical vessel with a conical base. In the upper part of the separator the vessel is fitted with feed inlet, and at the base there are two outlets, one for light particles and other for heavy particles. The rotating disc and rotating blades are attached to the central shaft, to produce air movement.

Working

As shown in the Fig. 6-3, the sample powder is passed through the feed inlet, which falls on the rotating disc. The rotating blades are attached to the same shaft. These produce a current of air as shown by the arrows. The fine particles are picked up and are carried into space, where air velocity is sufficiently reduced. The fine particles are dropped and ultimately collected at an outlet meant for fine particles. The heavy particles which fall downward are removed at an outlet meant for heavy particles.

Uses

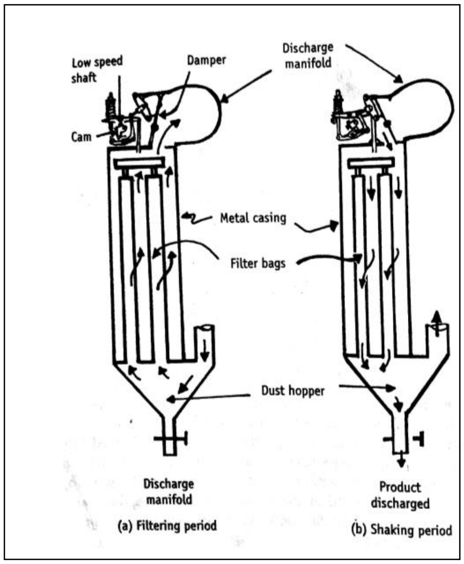
Air separator is often attached to the ball mill or hammer mill to separate and return oversized particles for further size reduction.

Bag filter

Principle: In bag filter, size separation of fines or dust from the milled powder is achieved in two steps. In the first step, the milled powder is passed through a bag filter (cloth by applying the suction on the opposite side of the feed entry). This facilitates the separation. In the next step, pressure is applied to in order to shake the bags so that powder adhering to the bag falls off, which is collected from the conical base.

Construsction

1. It contains a number of bags made up of fabric or wool.
2. These are suspended in a sheet metal container
3. A hopper is arranged at the bottom of the filter to receive the feed
4. An exhaust is arranged on the top of the container.
5. Adjacent to this, a bell crank arrangement is made to bring the filters to normal atmospheric conditions.



Bag filter

Working

Two steps are there.

In the first step, the feed is separated from the air by passing it through the cloth bags. In the subsequent step, the bags are shaken to collect the fines that are adhered to the bags. These two steps follow in succession and are controlled at different intervals with the help of a bell crank lever arrangement.

Bell crank lever arrangement: In this mechanism, a shaft with a cam is allowed to rotate at a low speed. During rotation, the cam can either press the bell crank lever or does not come into contact. Depending on this mechanism, the damper changes its position.

Filtering period: The exhaust fan positioned at the top keeps the bags under less pressure than atmospheric pressure. The gas containing fine particles (or dust) enters the hopper, as shown with arrows in Figure, and passes up. The gas feed passes through the fabric of bag. During this process, the fines (or dust) are retained in the bags, while the gas reaches the top of the casing. Because of air, the bag remains taut during filtering operation.

Shaking period: Since vacuum is cut off in the chamber, air from outside enters the casing and passes through the bags. This results in violent shaking of the bags, so that the dust and fine particles are displaced from the bags and falls into the conical base. It is then removed at intervals. Such devices are entirely automatic in their action and can be designed to affect very large filtering surface per unit floor space.

Uses

Bag filters are used along with other size separation equipment, for example, a cyclone separator. Bag filters are used to remove the fines from cyclone discharge. Bag filter is connected to the discharge end of the fluidized energy mill.

Advantages

1. Extremely useful in removing fines, which cannot be separated by other methods.
2. These can be used even to remove dust. Ex: Household vacuum cleaner

Disadvantages

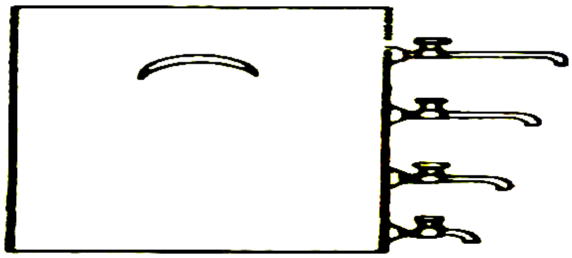
It is not a pure size separation equipment as such.

Elutriation tank

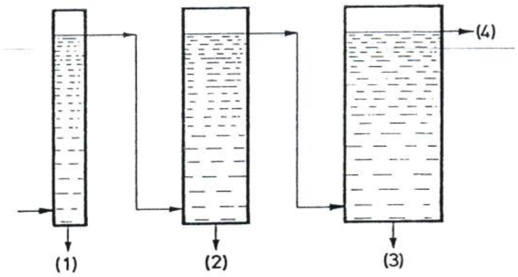
Principle and construction: The size separation of powder is based on the low density of fine particles and high density of the coarse particles. Elutriating tank is used to separate the coarse and fine

particles of powder after levitation. The dry powder or paste made by levitation process is kept in an elutriating tank and mixed with a large quantity of water. The solid particles are distributed in the liquid by stirring and then it is allowed to settle down. Depending on the uniformly density of solid particles, it will either settle down or remain suspended in water.

The sample is withdrawn at different heights through the outlets. These are dried and thus the powder with various size fractions are collected Nowadays in elutriation process, the particles are suspended in a moving fluid, generally water or air. The apparatus consists of a vertical column with an inlet near the bottom for suspension, an outlet at the base for coarse particles and an overflow near the top for fluid and fine articles. One column will give single separation into two fractions. If more than one fraction is required a number of tubes of increasing area of cross section can be connected in series. The velocity of the fluid decreases in succeeding tubes as the area of cross section increases, thus giving a number of fractions. These fractions ar separated and dried.



Elutriation tank



Multi-stage elutriator (1) to (4) are fractions of decreasing particle size

Advantages

1.The process is continuous.
2.Depending on the number of fractions required, the same number of tubes of different area of cross section can be connected.
3.The separation is quick as compare to other methods of separation
4.The apparatus is more compact than as that used in sedimentation methods. The main disadvantage of this method is that the suspension of solid particles has to be diluted which may not be desired in certain cases.

Disadvantage

The suspension has to be dilute; which may sometimes be undesirable.

MIXING

Solid-solid mixing

Mixing is a process that tends to result in a randomization of dissimilar particle s within a system. Mixing means to put together in one mass

Blending: Mix smoothly and inseparably together (This process need minimum energy utilization)

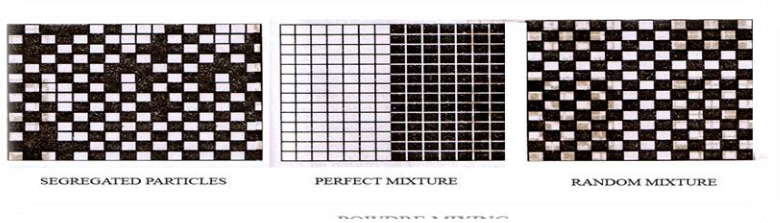
Types of solids

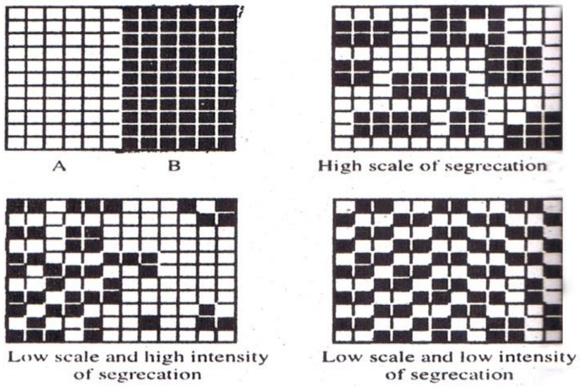
1. **Cohesive solids-** Wet clay
2. **Non-cohesive solids-** Dry sand, plastic chips etc.

***Cohesion** - sticking together of particles of the same substance*

Applications of mixing

1. Wet mixing in the granulation step in the production of tablets and capsules
2. Dry mixing of several ingredients ready for direct compression as in tablets
3. Dry blending of powders and capsules, dry syrups, and compound powders (insufflations)
4. Production of pellets for capsules





Scale and intensity of segregation

Inter Particle Interactions-Segregation

Particle characteristics like size, shape, distribution and surface influence the inter particle interactions in a powder bed

Inertial forces

Inertial forces like van der Waal's forces, electrostatic and surface forces etc. These forces tend to hold neighboring particles in a fixed relative position.

Surface or interfece forces

Cohesive Forces which prevent intimate mixing of particles. Cohesive forces and Frictional forces promotes the inter particulate movement due to gravitational forces resulting in lumps. These depend on surface area, surface roughness, surface polarity, surface charge and adsorbed surfaces such as moisture.

Gravitational forces

Gravitational forces tend to improve the movement of two adjacent particles or group of particles. Tumbling action (fall suddenly) promotes interparticulate movement due to gravitational forces. Continuous exchange or distribution of momentum between translational and rotational mode is necessary for effective mixing. The efficiency of momentum transfer depends on, Elasticity of collision between particles, coefficient of friction, surface area of contact, surface roughness, centrifugal forces etc.

Mechanism of mixing in solids

1. **Convective mixing**-A large mass of material moves from one part to another. This is also termed as *macro mixing*. -Ex: Inversion of a powder bed using blades or paddles or screw element.
2. **Shear mixing**- Here the forces of attraction are broken down so that each particle moves on its own between regions of different composition and parallel to their surfaces.
3. **Diffusive mixing**- The random motion of particles within the power bed, thereby particles change their position relative to one another (*micro mixing*)

In solid-solid mixing, mainly four steps are involved.

i. Expansion of bed of solids

ii. Application of three dimensional shear forces to the power bed

iii. Mix long enough to permit true randomization of particles

iv. Maintain randomization After mixing, no randomization)

The law of mixing appears to follow first order

$$M = A\,(1-e^{-kt})$$

Where M = degree of mixing after time t

t = *time* in minutes

A and k are constants which depend upon the mixer geometry, physical characteristics, of the powders and proportion of the materials being mixed

Degree of mixing

Degree of mixing is also known as degree of homogenecity.The possible degree of mixing can be obtained provided each particles move freely to every spot of the equipment. It requires the movement of particle sin three directions. The degree of mixing is important in calculating the time of mixing and to obtain an acceptable randomization.

Ideal mixing/Perfect mixing

Any mixing process will never yield an ideal or perfect mixture. Perfect mixing means each particle of one component is lying nearly adjacent to a particle of another component. Degree of mixing is indicated by its standard deviation.

Since perfect mixing is not possible, other acceptable methods of mixing must be considered.

Random mixing -Same ratio of components in the entire mixture

Ordered mixing: It is described as the usage of mechanical, adhesion and coating forces. Ordered units in the mix should be such that ordered unit will be the smallest possible sample to the mix.

Statistical parameters

The analysis of samples before and after mixing provides information about the degree of mixing. Arithmetic mean, Standard deviation (*SD*), relative standard deviation (*RSD*) etc. are commonly used.

Mixing index

The selection of a mixer or blender depends upon the degree of mixing which it can provide and this is commonly referred as mixing index.

Mixing index refers to the degree of uniformity achieved during mixing. It is calculated by statistical procedure. With the data obtained, mixing index is calculated using the following formula:

$$M_s = \sqrt{\frac{\Sigma(y - \bar{y})^2}{n(1 - \bar{y})\bar{y}}}$$

Where Ms = mixing index

n = number of samples

$\bar{y}$ = true average composition of component A in the mixture

y = actual composition of component A in a single sample.

The M_s values are calculated at different time intervals. Based on

the results, optimum time required for actual mixing can be estimated.

Sampling

While sampling a bed of powder, it should be sampled uniformly. It is assumed that there are many zones in a bed of powder. Among the zones, the composition is different.

Sample size guidelines

The number samples required should be not less than 20, preferably 30 and more ideally 100. But analysis of these samples is time consuming and therefore, economic considerations suggest 20 samples.

Collection of samples

1. A powder should be sampled when it is in motion
2. Sampling after completion of mixing
3. In case, sampling cannot be done from a moving stream of powder, it can be done after completion of mixing
4. After sampling, the samples are subjected to statistical evaluation.

Method of collection

1. Scoop sampling of bulk mixture
2. Thief probing of the bulk mixture

Factors influence mixing

a. Nature o surface- Rough, smooth etc
b. Density of particles
c. Particle size
d. Particle shape Ex: Spherical shape is easy for mixing
e. Particle charge
f. Proportion of materials.

MIXING EQUIPMENTS FOR SOLID-SOLID MIXING

Twin Shell Blender Or V-Cone Blender

Principle and Construction

It is made of stainless steel or transparent plastic. Larger models with a charging capacity of one tonne and rotate 15 revolutions per minute. The material is loaded through either of the shell hatches. Emptying of the blend is normally done through an apex port. 50 - 60% of total volume can be loaded with the powder.

V-Cone Blender

Advantages

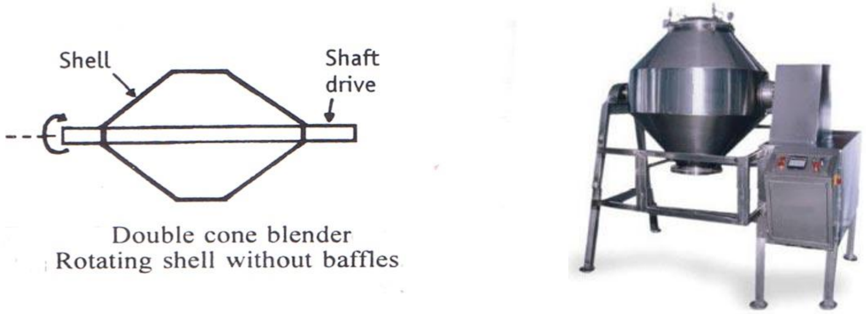
1. Fragile granules can be handled because of minimum attrition
2. They can handle large capacities
3. Easy to clean, load and unload
4. Requires minimum maintenance.

Disadvantages

It needs high head space for its installation

Double cone blender

It is usually charged and discharged through the same port. Different density powders can be mixed very efficiently. Commonly the range of 30 to 100 RPM is used. The method remains same as that of V-cone lender.



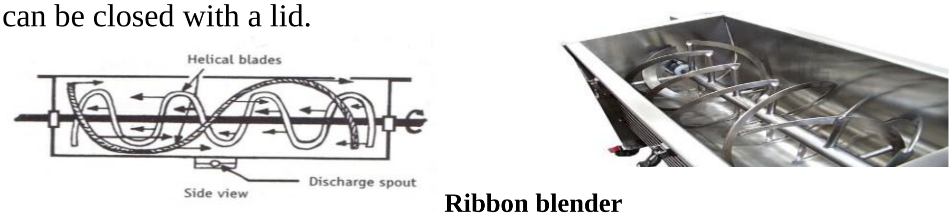
Double cone blender

Ribbon blender

The mechanism of mixing is shear. Shear is transferred to the bed of powder by ribbon shaped moving blades in a fixed non-moving shell. Convective mixing also occurs. An equilibrium state of mixing can be achieved.

Construction

1. Consist of a non-movable cylindrical shell (trough) usually open at the top.
2. It is fitted with two helical blades which are mounted on the same shaft through a long axis of the trough/shell
3. The blades have both right hand and left hand twists. The blades are connected with a speed drive
4. The trough can be closed with a lid.



Ribbon blender

Working

Ribbons are allowed to rotate. One blade moves the solid slowly in one direction and the other moves them quickly in the opposite direction

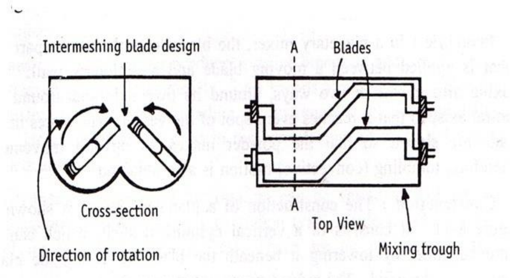
Different powders are introduced through the top of the trough. The powders are lifted by a centrally located vertical screw allowed to cascade to the bottom of the shell (tumbling action) The counter acting blades set up high shear and are effective in breaking lumps. Helical blades move powder from one direction to other.The blend is discharged from the bottom opening.

Uses

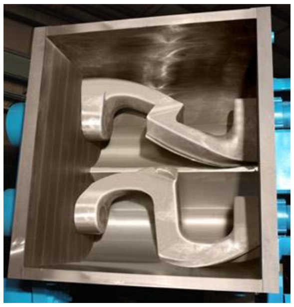
In the mixing of finely divided solids, wet solid mass, sticky and plastic solids. Uniform size and density material can be easily mixed. It is used for liquid-solid and solid-solid mixing.

Sigma blade mixer

The mechanism of mixing is shear. The inter-meshing of sigma shaped blades creates high shear and kneading actions. Convective mixing is achieved by cascading the material.



Sigma blade mixer. Stationary shell and rotating blades.



Sigma Blade Mixer

Working

Different powders are introduced from the top of the trough. The body is covered because considerable amount of dust may be evolved during dry blending and granulating solution may evaporate during the granulation. Through the fixed speed drive, the sigma blades are allowed to rotate. The blades move at different speeds, one usually about twice the speed of the other, resulting in lateral pulling of the material. Cascading and shear actions can be achieved. The perforated blades can break up the lumps. By means of a rack and pinion drive the bowl is tilted to empty the blend

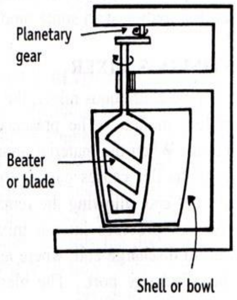
USES

In wet granulation process in the manufacture of tablets, pills, ointments etc. It is mainly used for liquid-solid mixing although it can be used for solid-solid mixing.

Planetary Mixer

Principle: Here the blade tears the mass apart and shear is applied between a moving blade and a stationary wall. The mixing arm will move around its axis and around the central axis so that it reaches every spot of the vessel. Convective (tumbling) action is also obtained

Planetary Mixer



Planetary mixer.

Working

1. In the planetary mixer, the agitator has a planetary motion.
2. It rotates its own and around the central axis so that it reaches all parts of the vessel.
3. The blade/beater is shaped to pass with close clearance over the side and bottom of the mixing bowl.
4. The blade tears the ass by shear between the moving blade and the stationary wall.
5. The plates in the blades are sloped so that the power makes san upward movement. Therefore, tumbling or convective motion also is obtained.
6. Initially the blade moves slowly for pre mixing and finally increased speed for active mixing. Thus high shear can be applied for mixing.
7. Emptying the bowl may be done by hand (scooping) or dumping mechanism.

Uses

1. In wet granulation in tablet manufacturing
2. Steam jacketed bowls can be used in the manufacture of sustained release products and ointments.

Advantages

Speed of the rotation can be varied at will, which is helpful in wet granulation process.

Disadvantages

✓ Mechanical heat is built up with in the powder mix.

✓ Requires high power

Size is limited and hence useful for batch work only.

LIQUID-LIQUID MIXING

Liquid-Liquid mixing is considered as a simple mechanism when compared to liquid-solid mixing. This also involves the application of shear.

***AGITATION**:* Induced motion of a material in a specified way, usually in a circulatory motion in a container.

***MIXING**:* Refers to the random distribution into or through one another of two or more separate phases.

Classification of Liquids according to Theory of solutions

1. **MISCIBLE LIQUIDS:** Miscible in all proportions Ex: Ethyl alcohol and water. These can be mixed easily by employing mechanisms of bulk transport and shear. After Shave lotions, elixirs etc. belong to this class.
2. **PARTIALLY MISCIBLE LIQUIDS:** Miscible in one another in particular proportion. Their miscibility depends upon temperature and pressure. Ex: P-cresol and water
3. **IMMISCIBLE LIQUIDS:** These are not miscible at all.

Ex: Vegetable oil and water. they can form emulsions. Mixing requires high shear.

Applications of liquid mixing

In the preparation of

- Suspensions
- Emulsions
- Solutions
- Aerosols
- Heat transfer operations.

Mechanism of mixing

- **BULK TRANSPORT**-Movement of large portion of a material from one location to another location in a given System-Mixing devices such as rotating blades and paddles.
- **TURBULENT MIXING**-Mixing due to turbulent flow. Eddies can be resulted.
- **LAMINAR MIXING**-Mixing of two dissimilar liquids through laminar flow. Suitable for liquids which need moderate mixing.
- **MOLECULAR DIFFUSION**-Mixing at molecular level in which molecules diffuses due to thermal motion.

MIXING VESSELS

Principle and Construction

The mixing apparatus consists of a container (tank) and a mixing device. These are assembled and used for a batch process. A mixing device is called ***impeller***, which is mounted with the help of a shaft. The shaft is driven by a motor. The tank is made up of stainless steel vessel. The top may be open or closed. The size of the tank depends upon the agitation method. The tank bottom is rounded to eliminate sharp corners into which fluid would penetrate. It also carries an oulet, coils, jackets, temperature measuring device etc. Wherever necessary.

Mixing vessel or tank

Mixing Tanks

Baffles

Containers can be either *baffled* or *un baffled*. Baffled Containers are those in which metal strips or baffles are attached vertically to the internal surface. When bulk transport is important in mixing, baffles can be used.

If solids are to be mixed with the liquid, baffles are fixed with a gap of about 25 mm between the baffle and the tank surface.

Flow pattern in mixing devices

Mixing devices are used to supply energy to the system so long as to bring about reasonably rapid mixing. Flow currents are responsible for transporting un mixed materials to the mixing zone.

Mixing devices are called as ***IMPELLERS*** Three main types of impellers are used namely, ***Propellers, Turbines and Paddles***

Propellers

- ☐ Normally they consist of many blades. A three bladed design is most common for liquids.
- ☐ Propellers may be either right or left handed.
- ☐ Four bladed propellers are used for special purposes
- ☐ In a deep tank, push-pull propeller is used in which two or more propellers are attached to the same shaft, which will work in opposite direction to create high turbulence.
- ☐ Propeller produces axial movement of the liquid.

Propeller type of mixing devices. A three bladed design.

Propellers

Propellers

Uses

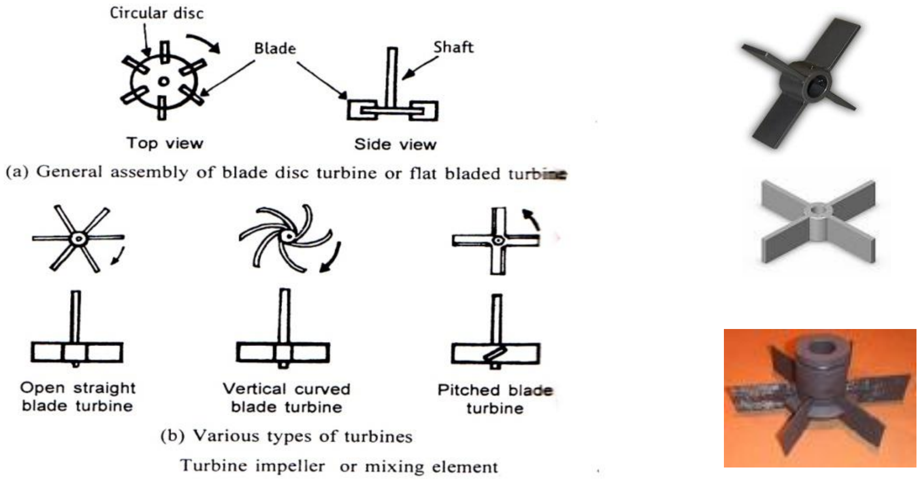
1. Propellers are used when high mixing capacity is needed.
2. Effective in liquids having viscosity of 2.0 Pascal second and slurries having up to 10% of solids.
3. Multi vitamin elixirs, disinfectant solutions are manufactured using propellers.

Disadvantage: Not suitable for high viscosity liquids like glycerin, castor oil etc.

Turbines

A turbine which consists of a circular disc to which a number of short blades are attached. It rotates at lower speed than propellers.

Uses: In mixing highly viscous solutions up to 700 Pascal seconds. Ex: syrup, liquid paraffin, glycerin etc. They can handle slurries up to 60% solids.



(a) General assembly of blade disc turbine or flat bladed turbine

(b) Various types of turbines

Turbine impeller or mixing element

Turbines

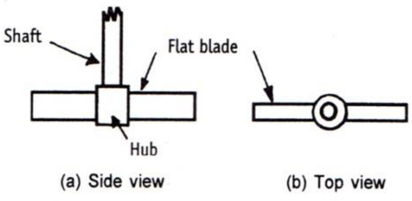
Paddles

A paddle consists of a hub centrally with two long flat blades attached to it vertically.

Paddles with two blades and four blades are common.

USES:

Paddles are used in the preparation of antacid suspensions. (Al Hydroxide gel and magnesium hydroxide), agar and pectin related purgatives, anti-diarrheal mixtures such as bismuth kaolin.



Paddle type of agitator or impeller

Paddles

Flow pattern during mixing

Liquids are mixed by using impellers. Mixing takes place due to the resultant effect of three components acting on the liquid. These are radial, longitudinal and tangential components.

Tangential or circular component

It acts in a direction tangent to the circle of rotation around the impeller shaft. If shaft is placed vertically and centrally, tangential flow follows a circular path around the shaft and creates a vortex in the liquid.

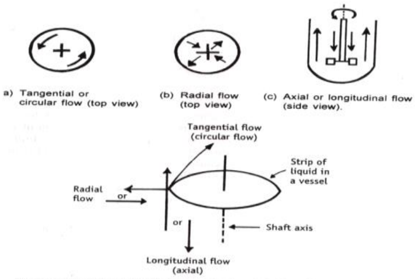
Radial Component

It acts in a direction vertical to the impeller shaft. Excessive radial flow takes the material to the container wall and then material falls to the bottom and rotates as amass beneath the impeller.

Axial/longitudinal/vertical components

It acts in a direction parallel to the impeller shaft. Inadequate longitudinal component causes the liquid and solid to rotate in layers without mixing.

These may occur singly or in various combinations. A satisfactory flow pattern depends on the balance of the components.



(d) Different components of flow at a horizontal strip of liquid section.

Diagrammatic representation of a cylindrical tank with tangential, radial and axial flow. Each diagram represents one type of flow (a, b, and c). In usual situations, one or more or a combination of these flow patterns occur simultaneously (d).

Flow pattern of impellers

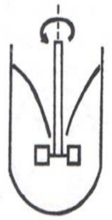
Impeller type	Flow component
Propellers	Axial
Turbines	Axial or tangential or both
Paddles	Radial and tangential, not axial
Paddles with pitch	Radial, Tangential and axial

Vortex *formation*

A strong circulatory flow pattern sometimes manifests into formation of a *vortex* near the impeller shaft.

It can be formed when:

- The shaft is mounted vertically in the centre of the tank.
- Blades and turbines are arranged
- Perpendicular to the central shaft.
- At high impeller speeds
- In un baffled tanks.

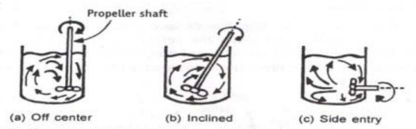


Vortex formation with a turbine type impeller
(Similar pattern is observed with propeller type also).

Disadvantage: Vortex formation reduces mixing intensity by reducing the velocity of impeller

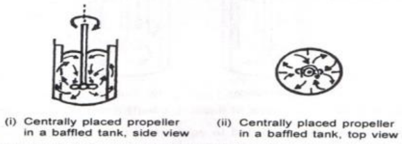
Prevention of vortex

- ✓ Avoiding symmetry in mounting impellers(figure)
- ✓ By using baffled containers
- ✓ Two or more impellers can be used when greater depth is desired. This is known as push pull system.
- ✓ Tanks other than cylindrical shape can avoid vortex formation.

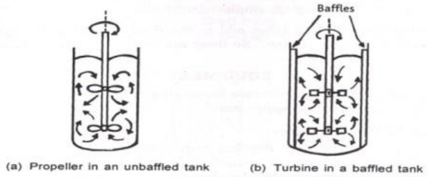


(a) Off center (b) Inclined (c) Side entry

1. Positions of propellers with flow pattern in a vessel to prevent vortex.


(i) Centrally placed propeller in a baffled tank, side view (ii) Centrally placed propeller in a baffled tank, top view

2. Arrangement of baffles and flow pattern in a vessel to prevent vortex.



(a) Propeller in an unbaffled tank (b) Turbine in a baffled tank

3. Arrangement of push-pull mechanism with flow patterns in a tank to prevent vortex.

Positions of impellers with flow pattern in a vessel to prevent vortex formation and gas entrapment.

Equipments for mixing liquids

The following factors influence liquid mixing in tanks or vessels.

1. Density, viscosity (Newtonian/non-Newtonian) and miscibility of the materials to be mixed.
2. Shape of the Impeller-Propeller type, straight, vertical curved or pitched.,
3. Position of the Impeller-Central, off-center, side entry, vertical or inclined etc.
4. Shape and size of the container-Cylindrical or geometric, presence of baffles or not etc.

Mixing of immiscible liquids

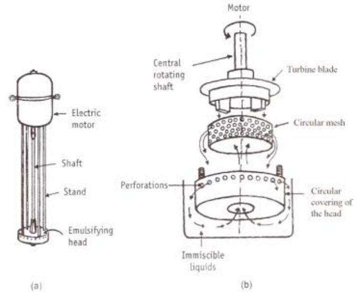
Silverson mixer/emulsifier

Principle

Silverson mixer-emulsifier produces intense shearing forces and turbulence by the use of high speed rotors (the rotating part of a turbine). This turbulence causes the liquid to pass through fine interstices formed by closely placed perforated metal sheets. Circulation of materials takes place through the head by the suction produced in the inlet at the bottom of the head. Circulation of the material ensures rapid break down of the dispersed liquid into smaller globules.

Construction

Silverson emulsifier consists of long supporting columns connected to a motor which give support to the head. The central portion contains a shaft, one end of which is connected to the motor and the other end to the head. The head carries turbine blades. The blades are surrounded by a mesh which is covered by an enclosure



Construction of Silverson emulsifier.

Silverson emulsifier

Working

The head of the emulsifier is placed in the vessel with immiscible liquids. When the motor is started, the central rotating shafts rotate the head, which in turn rotates turbine blades a very high speed. This creates a pressure differences a result, liquids are sucked into the head from the center of the base and subjected to intense mixing action. Centrifugal forces expel the contents of the head through the mesh on to the cover.

The intake and expulsion of the mixture set up a pattern of circulation to ensure rapid breakdown of the bigger globules into smaller globules.

Uses

Silverson mixer is used in the preparation of emulsions and creams of fine size particles

Disadvantage

Occasionally there is a chance of clogging of pores.

EVAPORATION

Evaporation is a process of vaporizing large quantities of volatile liquid to get a concentrated product, preferably a liquid.

Evaporation is a type of vaporization of a liquid, that occurs only on the surface of a liquid. Evaporation is a type of phase transition; it is the process by which molecules in a liquid state (e.g. water) spontaneously become gaseous (e.g. water vapour). The molecules in a water do not have enough heat energy to escape from the liquid. With sufficient heat, the liquid would turn into vapour. Evaporation is caused when water is exposed to air and the liquid molecules turn into water vapour which rises up and forms clouds

Distinguishing factors of evaporation

Residue is a concentrated liquid. Evaporating liquid is only one component. No attempt is made to separate the mixture of vapour. The Purpose of evaporation is to get concentrated product /liquid only

Applications

1.Manufacturing of bulk drugs

2. Manufacturing of biological products

3.Miscellaneous – In the manufacture of demineralized water

Differences between evaporation and other heat processes

S.No.	Evaporation	Other Heat process
01	The residue is a concentrated liquid residue is a concentrated.	Residue is a solid in drying
02	Evaporating liquid is only one component in most of the cases.	Evaporating liquid is a combination of two or more components in Distillation
03	No attempt is made to separate the mixture of vapor	It is compulsory to do so in Distillation
04	The purpose is to get a concentrated liquid only.	The purpose of concentrating is to get crystals in crystallization

Factors affecting evaporation

Rate of evaporation depends on several factors

$M = K S P (b – b')$

M = mass of vapour formed per unit time, m3/s

S = surface area of the liquid exposed,

m2 P = atmospheric pressure,

Kpa b = maximum vapour pressure at the temp of air

b' = pressure due to vapour of the liquid

K = constant

1. **Temperature:** Higher the temperature, greater will be the evaporation Ex – Alkaloids, Hormones, Enzymes, antibiotics – heat sensitive **Vapour pressure:** Rate of evaporation is directly proportional to the vapour pressure of the liquid Lower the pressure, greater will be the evaporation
2. **Surface area:** Greater the surface area of the liquid, greater will be the evaporation
3. **Time of evaporation:** Exposure time is longer – more evaporation
4. **Density:** The higher the density, the slower a liquid evaporates
5. **Concentration:** Low concentration of the substance – faster evaporation
6. **Moisture Content of Feed:** Some drug constituents undergoes hydrolysis readily in presence of moisture at high temperature. To prevent the decomposition, the material is exposed to low temp. initially, then exposed to higher temp.
7. **Time of evaporation: I**f time of exposure is longer, greater will be the evaporation, provided the constituents are thermo stable. Exposure of a drug to a relatively high temperature for a short period of time may result in less decomposition of active principle than a lower temp. with long exposure time
8. **Film & Deposits:** When vegetable extracts are concentrated in steam pan, a film may be formed on the surface and/or precipitate matter may deposit on the heating surface. Film reduces the heating surface and precipitated matter hinders the transfer of heat.
9. **Economic Factors:** Economics of labour, fuel, floor space & materials are primary considerations. The recovery of solvent & utilization of waste heat are also important as they involves considerable reduction of cost.

Classification of evaporators

1. Evaporators with heating medium in Jacket

Ex: Heat jacketed kettle or evaporating pan

2. Vapour Heated evaporators with tubular heating surfaces

a. Evaporators with Tubes placed horizontally

Ex: Horizontal Tube Evaporator

b. Evaporators with Tubes placed vertically

i. With short tubes

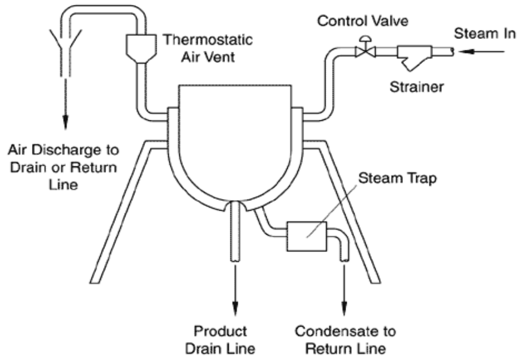
Single effect evaporation

1. Short Tube vertical evaporators
2. Short Tube vertical evaporator with propeller
3. Basket type evaporator
4. Triple Effect evaporator

ii.Evaporators with Long tubes

1. Climbing film(Rising Film) evaporator
2. Falling Film evaporator
3. Forced Circulation Evaporator

Evaporating Pan (Steam Jacketed Vessel/kettle)



Evaporating pan

Construction

Evaporating pan Material for construction: - Should be good conductor of heat.

Copper →as a kettle, excellent material because of good conductivity, but if acidic materials, some quantity of copper would have dissolved. So tinned copper is used.

Iron →as a jacket, because minimum conductivity, to prevent rusting of jacket, tinned or enameled iron is used.

Inlet of steam & outlet for non-condensed gas at the top of kettle. Outlet for the condensate at the bottom of the jacket Outlet for the concentrated product at the bottom of the kettle.

Working

Aqueous extract is placed into a evaporating pan Steam is supplied through the inlet. Steam gives out heat to the contents & the condensate leaves through the outlet. The contents must be stirred manually for small volumes & mechanically for large volumes. Rate of evaporation is fast at initial stage & decreases gradually as the liquid gets concentrated. Room where evaporation is carried must be fully ventilated, otherwise the room is quickly filled with fog of condensed vapor & water falls from the roof & runs down the walls. Fans fitted over pan remove vapor, prevent condensation in room, & accelerate the rate of evaporation by quickly removing saturated air from the surface of the liquid

Uses

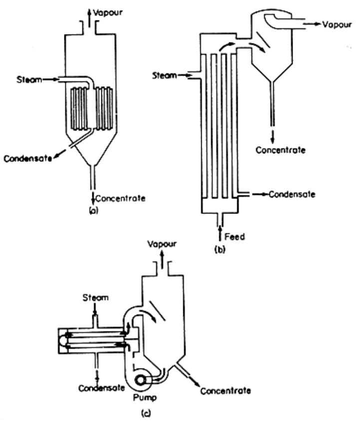
- ✓ Concentration of aqueous & thermos table liquors.
- ✓ For liquid extracts containing water as a menstruum. E.g. liquid extract of liquorice.

Disadvantages

- ❖ Heat economy is less, so, cost per unit material production is more.
- ❖ Not suitable for heat sensitive material due to long time of exposure.
- ❖ Heating area decreases as the product gets more concentrated.
- ❖ It is open type, so vapor passes into the atmosphere, which can lead to saturation of the atmosphere, slowing evaporation as well as causing discomfort.
- ❖ B.P, of water can’t be reduced, so reduced pressure can’t be created in open type evaporator.

HORIZONTAL TUBE EVAPORATOR

Principle: Steam is passed through the horizontal tubes, which are immersed in a pool of liquid to be evaporated. Heat transfer takes place through the tubes & the liquid outside the tubes gets heated. The solvent evaporates & escapes from the top of the evaporator. The concentrated liquid is collected from the bottom.



Horizontal Tube evaporator

Construction

Large cylindrical body with conical or dome-shaped top & bottom. Made up of cast iron or plate steel. Body range from 1.8 to 2.4 meters diameter & from 2.4 to 3.6 meters height. Lower part of body consists of a steam compartment with an inlet for steam at one end & a vent for non condensed gas on the other end. Condensate outlet from the bottom of the steam compartment. In the steam compartment, 6 to 8 stainless steel horizontal tubes are placed. The tubes are cut long enough so that they project about 25 mm beyond the tube sheet on the both ends. The width of steam compartment is usually half the diameter of the body. At one convenient point inlet for feed is provided. One outlet for vapor is placed at the top of the dome. Another outlet for thick liquid is placed at the centre of the conical bottom of the body.

Working

- The feed is introduced into the evaporator.
- Steam is introduced into the steam compartment.
- The horizontal tubes receive heat from the steam & conduct it to the liquid due to temp. gradient.
- Steam condensate passes through the corresponding outlet.
- The feed absorbs heat & solvent gets evaporated.
- The vapor then escapes through the outlet placed at the top.
- This process is continued until a thick liquid is formed, which can be collected from the bottom outlet.

Uses

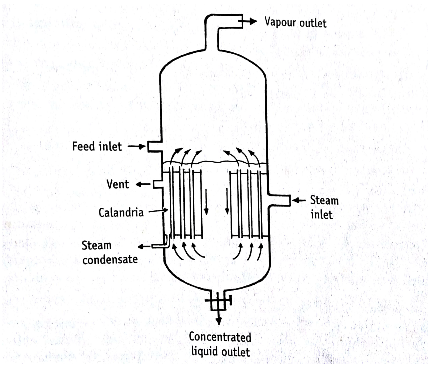
Best suited for non-viscous solutions that do not deposit scales or crystals on evaporation. E.g. cascara extract.

Advantages

Cost per square meter of heating surface is usually less.

Vertical Tube Evaporator
(Short Tube Evaporator)

Principle: Liquid is passed through the vertical tubes & the steam is supplied from outside the tubes. Heat transfer takes place through the tubes & the liquid inside the tubes gets heated. The solvent evaporates & escapes from the top of the evaporator. The concentrated liquid is collected from the bottom. vertical tube evaporator.



Short tube evaporator

Construction

Large cylindrical body made up of cast iron with dome shaped top and bottom. Inside the body, calandria is fitted at the bottom. Calandria consists of a number of vertical tubes, whose diameter ranges from 0.05 to 0.075 meters & length of 1-2 meters. About 100 such tubes are fitted in a body measuring 2.5 meters or more diameter. Inlet are provided for steam & feed. Outlet are provided for vapor, concentrated product, non-condensed gas and condensate.

Working

Steam is introduced outside the tubes. The condensate is passed through the corresponding outlet & non-condensed gas escapes through the vent. The feed is introduced in such a way as to maintain the liquid level slightly above the top of the tubes. The liquid inside the tubes is heated by the steam & begins to boil. As the liquid boils, it spouts up through the tubes & returns through the central down-take. It sets up a circulation of hot liquid, which enhances the rate of heat transfer. The vapor escapes through the top outlet. The steam is supplied until required concentration of the product is obtained. Finally the product can be withdrawn from the bottom outlet.

Uses

In the manufacture of cascara extract, sugar, salt, caustic soda.

Advantages

Tubes increases the heating surface nearly 10-15 times when compared with steam jacketed kettle. Vigorous circulation enhances the rate of heat transfer. It can be connected to a condenser & receiver, which further increases rate of evaporation. Also used for volatile solvents. No. of units can be joined to obtain more efficient effect. (Multiple effect evaporator)

Disadvantages

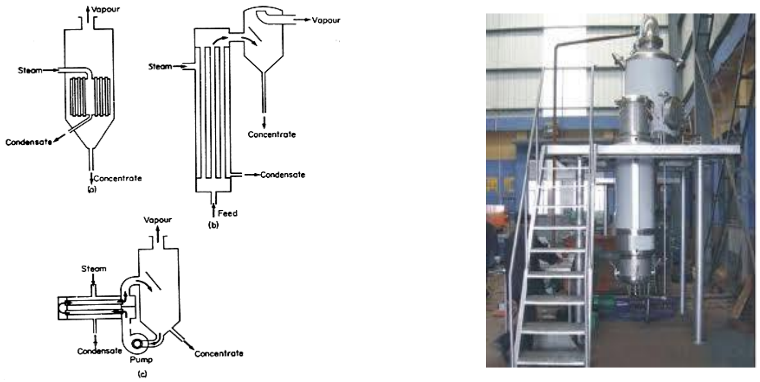
The liquid is maintained above the level of the calandria. So the upper layers of the liquid need a long time for getting heated. This problem can be minimized by removing concentrated liquid slowly from the bottom. Complicated construction, so installation cost increases. Cleaning & maintenance is difficult. During operation, the pressure inside the evaporator increases. In large evaporators, where the liquid depth may be of the order of 2 meters, the pressure increases to 25 kilopascals, leading to elevation of the boiling point by 5 to 60 C. this reduces the effective temp. gradient & may affect heat sensitive materials.

Variants

- Short tube vertical evaporator with Propeller
- Basket type evaporator

Climbing film evaporator

Principle: The tubes are heated externally by steam. The preheated feed enters from the bottom & flows up through the heated tubes. The liquid gets heated, the liquid near the wall becomes vapor & forms small bubbles. These tend to fuse to larger bubbles, which travel up in the tubes along with entrapped slug. The liquid films are blown up from the top of the tubes & strikes entrainment separator (deflector) kept above. This throws the liquid concentrate down in to the lower part from where it is drawn. Rising tube evaporator



Climbing film evaporator

Construction

The heating unit consists of steam jacketed tubes. Tubes (long & narrow) are held between two plates. A deflector is placed to the top of the vapor head. The evaporator carries steam inlet, vent outlet & condensate outlet. The feed inlet is from the bottom of the steam compartment.

Working

The preheated liquid feed is introduced from the bottom of the unit. The height of the liquid column is maintained low, i.e. 0.6-1.2 meters above the bottom tube sheet. Steam enters in to the spaces outside the tubes through the inlet. Heat is transferred to the liquor through the walls of the tubes. The liquid becomes vapor & forms smaller bubbles, which tend to fuse to larger bubbles. They travel up in the tubes along with slug. As more vapor is formed, the slug of liquid is blown up in the tubes facilitating the liquid to spread as a film over the walls. Finally the mixture of liquid concentrate & vapor eject at high velocity from the top of the tubes.

The deflector prevents entrainment, and acts as a foam breaker. Vapor leaves from the top, while concentrate is collected from the bottom.

Uses

Thermo labile sub. Can be concentrated. E.g. insulin, liver extract, vitamins. Clear liquids, foaming liquids & corrosive solutions in large quantities can be evaporated. Deposited film can be removed quickly by increasing feed rate or reducing steam rate.

Advantages

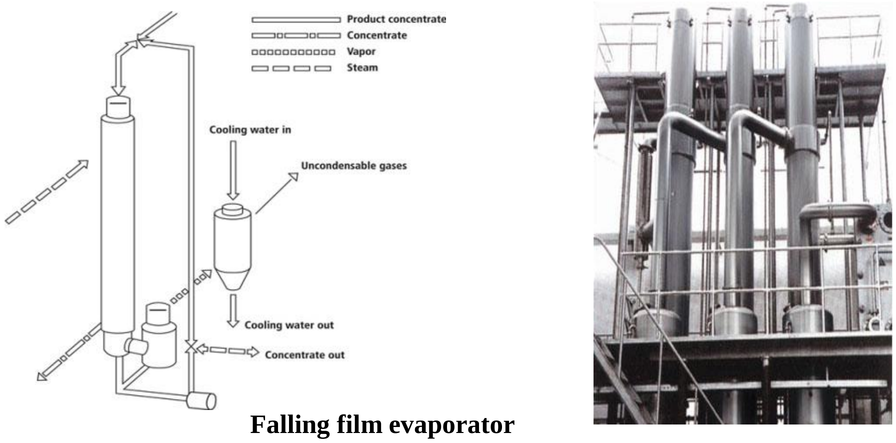
Large area for heat transfer. Liquid flows at a high velocity, so resistance for heat transfer at the boundary layers is reduced. So heat transfer enhanced. Time of contact between the liquor & heating surface is very short. (1 second for heater & 20 seconds in evaporator). So suitable for heat sensitive materials. Unlike short tube evaporator, the tubes are not submerged, so there is no elevation of B.P. due to hydrostatic head. Suitable for foam-forming liquids, because of deflector. Low hold up & small floor space.

Disadvantages

Expensive Complicated construction Difficult to clean & maintain Large head space is required Not for viscous liquids, salting liquids and scaling liquids If high feed rate, liquor may be insufficiently concentrated. If low feed rate, film can't maintained. Dry patches may form on the tube walls.

Falling film evaporator

Principle: Feed enters from the top and flows down the walls of the tubes. The liquid gets heated rapidly due to heat transfer from steam. The liquid boils & becomes vapor, which forms small bubbles. They tend to fuse to form larger bubbles, which travel down the tubes. Concentration takes place during this downward journey. Vapor and liquid are separated at the bottom.



Falling film evaporator

Construction

It resembles climbing film evaporator, but it is inverted. The heating unit consists of steam jacketed tubes. The feed inlet is from the top of the steam compartment. The other provisions are steam inlet, vent & condensate outlet remain same. The outlet for the product is provided at the bottom & is connected to a cyclone separator

Working

Steam is supplied into the steam compartment. Feed enters from the top of the tubes. The temp. of the boiling liquid is same as that of the vapor head. The feed flows down the walls of the tubes. The liquid gets heated rapidly. The liquid boils & become vapor, which forms small bubbles. These tend to fuse to form large bubbles, which travel down the tubes. Concentration takes place during this downward journey. Vapor & liquid are separated in the cyclone separator.

Uses

To separate volatile & non-volatile material, when the feed is of low viscosity. For the concentration of yeast extract, manufacture of gelatin, extract of tea & coffee. Used for heat sensitive materials such as fruit juices.

Advantages

Suitable for high viscous liquids, because the flow of vapor film is assisted by gravity. Liquid hold up is less & hold up time is very small. Not over heated during passage, heat transfer coefficient is high even at low boiling temp. Highly acidic & corrosive feeds can be concentrated using impervious graphite tubes & rubber lined vapor heads

Disadvantages

Easy distribution of feed is by using the perforated plate or by spray nozzles, so not useful for the suspension as the solids clog the perforated plate. Not suitable for salting or scaling liquids. Feed distribution is poor. For continuous supply, liquid may re circulated or rate of feed is kept high.

Forced circulation evaporator

Principle: Liquid is circulated through the tubes at high pressure by means of a pump. Boiling does not takes place because boiling is elevated. Forced circulation of liquid creates form of agitation. When the liquid leaves the tubes & enters the vapor head, pressure falls suddenly This leads to flashing of super heated liquor. Thus evaporation is affected.

Forced circulation evaporator

Construction

The steam jacketed tubes are held between two tube sheets. Tubes are 0.1 m inside diameter & 2.5 m long. The part of tubes projects into the vapor head. Which consists of a deflector. Vapor head is connected to a return pipe, which runs downward and enters into the inlet of a pump.

Working

Steam is introduced into the calandria. Pump sends the liquid to the tubes with a positive velocity. As the liquid moves up through the tubes, it gets heated & begins to boil. As a result the vapor & liquid mixture rushes out of the tubes at a high velocity. This mixture strike the deflector which throws the liquid downward. This result in an effective separation of liquid & vapor. The vapor enters the cyclone separator & leaves the equipment. The concentrated liquid returns to the pump for further evaporation. Finally the concentrated product is collected.

Uses

It is operated under reduced pressure, so suitable for thermo labile substance. For concentration of insulin & liver extract. Suitable for crystallizing operations where crystals are to be suspended at all times. Advantages :- High heat transfer coefficient due to rapid liquid movement. Salting, scaling & fouling are not possible due to forced circulation. Suitable for thermo labile substance, high viscous preparations because of pumping mechanisms.

Disadvantages

Hold-up of liquid is high. Expensive because pump is required.

Multiple Effect Evaporators

Evaporators are classified by the number of effects. In a single-effect evaporator, steam provides energy for vaporization and the vapor product is condensed and removed from the system. In a double-effect evaporator, the vapor product off the first effect is used to provide energy for a second vaporization unit. Triple- effect evaporator problems are familiar to generations of engineering students. This cascading of effects can continue for many stages. Multiple-effect evaporators can remove much larger amounts of solvent than is possible in a single effect.

In a multiple effect arrangement, the latent heat of the vapor product off of an effect is used to heat the following effect. Effects are thus numbered beginning with the one heated by steam. It will have the highest pressure. Vapor from Effect I will be used to heat Effect II, which consequently will operate at lower pressure. This continues through the train: pressure drops through the sequence so that the hot vapor will travel from one effect to the next.

Normally, all effects in an evaporator will be physically the same in terms of size, construction, and heat transfer area. Unless thermal losses are significant, they will all have the same capacity as well.

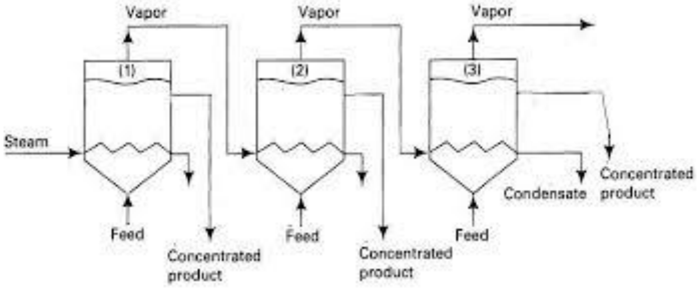
Evaporator trains may receive their feed in several different ways. The feed order is NOT related to the numbering of effects. Effects are always numbered according to decreasing pressure (steam flow).

Forward Feed arrangements follow the pattern I, II, III. These require a single feed pump (reduced fixed costs). They typically have reduced economy (higher operating costs) since the cold feed must be raised to the highest operating temperature. These also tend to have the most concentrated liquour, which tends to be the most viscous, in the lowest temperature effects, so there may be difficulties getting a good overall heat transfer coefficient.

Backward Feed arrangements go III, II, I. These need multiple pumps to work against the pressure drop of the system; however, since the feed is gradually heated they usually have better economies. This arrangement also reduces the viscosity differences through the system and so is better for viscous solutions.

Mixed Feed arrangements offer a compromise, with the feed entering in the middle of the system (i.e. II, III, I). The final evaporation is done at the highest temperature so economies are still better than forward feed, but fewer pumps are required than in a backward feed arrangement.

Parallel Feed systems split the feed stream and feed a portion to each effect. This is most common in crystallizing evaporators where the product is likely to be a slurry.



Multiple Effect Evaporators

USES OF STEAM AS HEATING MEDIUM

1. Steam is an invisible gas generated by adding heat energy to water in boiler.
2. The equipment which produce steam are called as boilers.
3. Enough energy must be added to raise the temperature of water o the boiling point.
4. The additional energy without further increase in temperature changes the water into steam

Steam is very efficient and easily controlled heat transfer medium. It is used to transport energy from a central location(Boiler) to number of locations in the plant or industry where it is used to heat air, water or process applications.

Heat required to change boiling water to steam is called as latent heat or heat of vaporisation. Heat flows from a higher temperature level to a lower level. . The process is known as heat transfer. Steam provides excellent heat transfer. Steam can be surrounded or be injected into the product being heated. Steam is sterile and thus popular process use in food, pharmaceutical and health industries. It is also widely used in hospitals for sterilization purposes. The typical applications of steam in industry includes the following:

1. Heating / Sterilization-Heat exchangers, interchangers and autoclaves
2. Propulsion / Drive
3. Motive (moving parts of machines)
4. Atomization
5. Cleaning
6. Moisturization
7. Humidification

HEAT TRANSFER (FLOW OF HEAT)

Heat

Heat is a form of energy. Whenever a physical/chemical transformation occurs, heat flows into or leaves the system.

Sources of heat in Industry

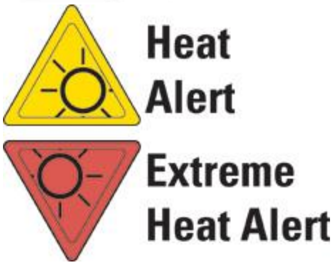
1. Steam
2. Electricity

Applications

I. Evaporation
II. Distillation
III. Drying
IV. Crystallization
V. Sterilization

Instruments

Air ovens
Incubators
Dryers
Refrigerators
Heat Exchangers



Mechanism of heat flow

1. Heat flows from a region of higher region to a region of low temperature.
2. Heat may flow by one or more of the three basic mechanisms, which are as follows.

1. Conduction

When heat flow in a body is achieved by the transfer of the momentum of individual atoms or molecule without mixing, such a process is known as conduction.

Ex: Flow of heat through a *metal* shell or *boiler*

2. Convection

When heat flow is achieved by actual mixing of warmer portions with cooler portions of the same material/molecules, it is called as convection.

Ex: *Coil type water heater* where heating of water takes place by heating the water by a hot surface.

3. Radiaton

When heat flow through space by means of electromagnetic waves, such energy transfer is known as Radiation

Ex: Solar water heaters, solar cookers, Microwave ovens, Microwave cookers etc.

Conduction

Heat transfer through conduction can be considered under the following three categories.

✓Fourier’s law and thermal conductivity
✓ Steady state heat transfer
✓ Unsteady state heat transfer

Fourier’s law

✓ Heat can flow only when there is a temperature gradient.
✓ i.e., from a hot surface to a cold surface.
✓This can be explained by Fourier’s Law
✓ Rate of heat transfer = $\frac{\text{Temperature difference}}{\text{Resistance of the medium}}$

Fourier’s Law states that the rate of heat transfer across a surface is proportional to the temperature gradient at the surface

Fourier’s law-Derivation (* Heat transfer through a metal wall or slab)

dQ/dq a dt/dx Fourier’s Equation

If A is the area to the heat flow, then

dQ/dq = -kA dt/dx

Where **dQ/d**q = Rate of heat transfer

A= Area of cross section of heat flow path

dt/dx= Temperature gradient

k= Thermal conductivity of the medium

(- Sign indicates that heat flow occurs from hot to cold region)

Thermal Conductivity

In Fourier’s equation, ***k*** is a proportionality constant which indicates the resistance per unit flow path which is independent of temperature gradient. Its value depends on the material of which the body is made and temperature.

But ***k*** can be considered as a constant because the change in its value is negligible

If temperature range is larger, then

k= a+ bT

*where **a** & **b** are constants; k- Proportionality constant/thermal conductivity, T-Temperature*

At a given temperature, thermal conductivity is a function of bulk density of the material. The unit of thermal conductivity is **BTU/ft^{2}hr(^{0}F/ft)** ***** *BTU-British Thermal Units*

Factors regarding Thermal Conductivity

1. Thermal conductivity is the *reciprocal* of thermal resistance
2. The ***coefficient**** of thermal conductivity is the quantity of heat that flows across a unit surface area in unit time at constant temperature
3. The coefficient of thermal conductivity depends upon the *material* which it is made and the *temperature*
4. Thermal conductivity of liquids and gases are very small compared to most of the solids

---

***-** *A multiplier or factor that measures some property*

THERMAL CONDUCTIVITY (k) OF SOME MATERIALS

Materials	Temperature(^{0}C)	k BTU/ft^{2}hr(^{0}F/ft)	Material	Temperature (^{0}C)	k BTU/ft^{2}hr(^{0}F/ft)
Silver	100	238	Mercury	0	4.8
Copper	100	218	Ethyl ether	40	0.080
Aluminium	100	119	Water	100	0.39
Cast Iron	100	20	Air	100	0.018
Mild Steel	100	26	Steam	100	0.0136
Stainless steel(	100	9.4	Nitrogen	100	0.018
Carbon	50	80	Carbon dioxide	100	0.013
Glass	50	0.63	Hydrogen	100	0.124
Glass wool	100	0.036			
Ice	0	1.2			

Applications of Thermal Conductivity

1. In case of steam jacketed vessels, the kettle (Inner surface) must have good conductivity so that maximum amount of heat passes from steam to the contents.
2. The high thermal conductivity of copper suggests that it is a suitable material for the construction of kettle.
3. At the same time, the metal used for jacket (outside) must have minimum conductivity to prevent loss of heat by conduction/ radiation.
4. The low thermal conductivity of iron suggests that it suitable for the construction of jacket
5. For the construction of evaporators and tubular heat exchangers, thermal conductivity values are helpful.
6. Thermal conductivity is very sensitive to changes in chemical composition and temperature and therefore, these values cannot be used in all situations.

Steady state heat transfer

When heat transfer remains constant and is unaffected by time, it is called as steady state heat transfer.

- Here **dQ/d**q in Fourier's equation is constant and hence called as *q*
- So Fourier's equation for steady state heat transfer can be written as,

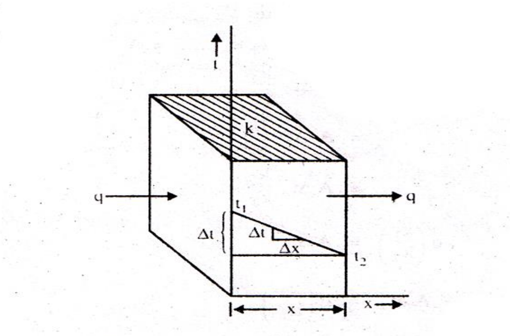
$$Q/\theta = q = -kA\ dt/dx$$

Heat Conduction through a slab[*]

Slab[*]: a large, thick, flat piece of solid material

- If we consider a slab of a material of which two faces have temperatures t_1 and t_2. If t_1 is greater, heat flows from higher temperature t_1 to other face having temperature t_2. If x is the thickness of the slab, then by Fourier's equation, rate of heat transfer is given by,
- $q = kA\ (t_1-t_2)/\ x$

  $q = k/\ x\ A\ \Delta t$

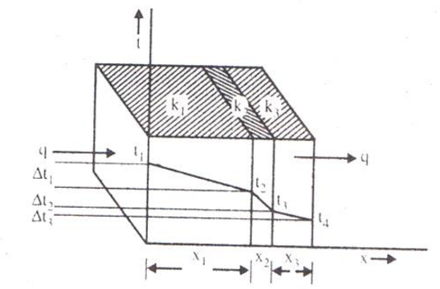


Heat conduction through a slab

Many times, heat flows through several consecutive layers of different materials in such situations, the above equation has to be applied for every layer separately. Thus if the same quantity of heat per unit time passes through each layer, then,

$$q = A1\ \Delta t_1 k_1/x_1 = A_2\ \Delta t_2 k_2/x_2 = A_3\ \Delta t_3 k_3/x_3$$

Heat Transfer through Series of Slabs (Series of layers)



Heat Transfer through series of slabs

If cross sectional areas of the layers are same

i.e., A1 = A2 = A3 =………………..= A

Then q= A $\Delta t_1 k_1/x_1$ = A $\Delta t_2 k_2/x_2$ = A $\Delta t_3 k_3/x_3$

$q(x_1/k_1) = A\ \Delta t_1$

$A\ \Delta t_2 = q(x_2/k_2)$

$A\ \Delta t_3 = q(x_3/k_3)$

Therefore, $A\ \Delta t_{1+}\ A\ \Delta t_{2+}\ A\ \Delta t_{3+\ldots =}\ q(x_1/k_1)+ q(x_2/k_2)+ q(x_3/k_3)+\ldots$

Sum of the temperature gradients across each layer is equal to the difference in temperature between inside and outside surface.

i.e, $\Delta t_{1+}\ \Delta t_{2+}\ \Delta t_{3+\ldots\ldots =}\ \Delta t$

Thermal conductivity per unit length is conductance(C).Therefore k_1/x_1 is the conductance.

C_1 for first layer,C_2 for second layer and similarly for subsequent layers, then,

$x_1/k_1+x_2/k_2+x_3/k_3= 1/C_1+1/C_2+/C_{3}+_{\ldots} =$ **1/U** where U is the overall conductance for combined layers

$A\ \Delta t = q\ (1/U)$

$$\boxed{q = UA\ \Delta t}$$

This is equation explains heat flow through the combination of layers and *U* is called as the ***overall heat transfer coefficient***

HEAT TRANSFER IN PIPES AND TUBES (Through Thick walled cylinder)

In heat exchangers and pharmaceutical equipments, heat transfer takes place across a tube or pipe. in such cases, the area changes. There fore it is necessary to determine the value of A and thickness of pipe. Consider a pipe having a length of l with internal radius r_1 and external radius r_2, and then the area *2*□*r* l will have to be determined as per the thickness of the wall

1. **For thin walled pipes, where r_1/r_2 = <1.5**

   Then mean radius, $r_m = r_1 + r_2/2$

$A = 2\Delta\, (r_1+r_2/2)\, l$

By substituting Value of A from equation $q = UA\,\Delta t$

We get, $q = k\, 2p\, (r_1+r_2/2)\, l\ \Delta t/r_2-r_1$

2. **For thick walled pipes, heat transfer area is calculated by logarithmic mean radius(r_m)**

$r_m = r_2-r_1/In\,(r_2/r_1)$

$q = k\, 2\Delta\, (r_2-r_1)/In\,(r_2/r_1)\, l\, \Delta t/r_2-r_1$

$= 2p\, kl\, \Delta t/In\, r_2/r_1$

et, $q = k\, 2\Delta\, (r_1+r_2/2)\, l\, \Delta t/r_2-r_1$

Unsteady heat transfer

- When temperature changes with time, such heat transfer is called as *Unsteady Heat Transfer.*

  Ex: Heating and Cooling of Pharmaceuticals

- Unsteady heat transfer analysis include complicated mathematical calculations including solving Fourier's equation written in terms of partial differentials in three dimensions but is out of scope of this discussion.

Convection

- When heat flow is achieved by actual mixing of warmer portions with cooler portions of the same material/molecules, it is called as convection
- Ex: *Coil type water heater* where heating of water takes place by heating the water by a hot surface.

Types of Convcetion

1. Natural convection- When movement of group of molecules occurs due to change in density resulting from temperature gradient, it is known as *natural (Free) convection*. Here the warmer fluid will be replaced by the cooler one generating convection currents. These currents start when forces like gravitational, centrifugal, electrostatic etc. acts on fluid in which there is density gradient. The force which include these convection currents are called as buoyancy force, which is due to the presence of density gradients.
2. Forced convection

   Mixing of fluid may be obtained by the use of a stirrer or agitator or pumping the fluid for re circulation. Such process in heat transfer is designated as forced convection.

   Ex: *Tubular evaporators*

Rate of heat transfer is given by the equation,

$q = h_c A(t_1-t_2)$

Where h_c is the convective heat transfer coefficient or surface heat transfer coefficient

Radiation

When heat flow through space by means of electromagnetic waves, such energy transfer is known as radiation

Ex: Solar water heaters, solar cookers, Microwave ovens, Microwave cookers etc.

Mechanism

Emission of energy will occur when an electron from a high energy orbit jumps to a low energy orbit and the energy released from it has the properties of electromagnetic waves. These waves upon striking a receiver the photons travel into it until they strike an electron or nucleus that is susceptible to the energy level of photon. This collision results in an increase in the energy of receiver. In solids, radiation is usually absorbed very close to the surface because of its dense nature, whereas penetration is very high in liquids and still high in gases.

Black body

Any body(material) above absolute zero temperature emits radiation. The radiation that results only due to temperature is called as thermal radiation. A part of the incident radiation is absorbed by the body, a part is reflected and a part is transmitted. The absorbed radiation is transformed into heat. The proportion of incident energy that is absorbed, reflected and transmitted depends on the properties of the receiver and to some extent wavelength of radiation and temperature of receiver. *A black body is one which will absorb all the incident radiation into heat and emits all thermal energy as radiation.*

$\mathbf{a + r + \tau = 1}$ τ *-tau*

a - Absorptivity, fraction of incident radiation that is absorbed

r – Reflectivity, fraction of incident radiation that is reflected

τ – Transmissivity, fraction of incident radiation that is transmitted

For most of the solids, transmissivity is zero; therefore the above equation reduces to

$\boldsymbol{a + r = 1}$

Laws in radiation heat transfer

1. **Kirchoff's law**
2. **Stefan -Boltzmann law**

Kirchoff's law

1. This law establishes a relation between the emissive powers of a surface to its absorptivity.
2. Emissive power of a body E is the radiant energy emitted from unit area in unit time.
3. If a small body is placed inside a large evacuated enclosure with wall temperature T, the heat will be exchanged between the body and enclosure until equilibrium is established.i. e, enclosure, wall and body will have the same temperature. Then the body will emit as much as energy as it is absorbing.
4. If E is the emissive power of the body, a is its absorptivity and G is the rate at which the energy falls from the wall on the body; then energy balance at this equilibrium state can be written as,
   a. $\mathbf{G \alpha E}$
   b. $\mathbf{G = E/\alpha}$

5. Rate of energy fall G is a function of temperature T and geometrical arrangement of both surfaces. But if the body is very small compared to the enclosure and its effect upon the irradiation field is negligible. Hence G remains constant at any temperature. This is stated by Kirchoff's law. It states that the ratio of emissive power to the absorptivity is same for all bodies in thermal equilibrium. Thus for two bodies at same temperature,
6. $E_1/\alpha_1 = E_2/\alpha_2$

7. E_1, E2 are the emissive power of the two bodies.
8. α_1, α_2 are the absorptivity of the two bodies in thermal equilibrium.
9. For a black body, α =1 and then according to Kirchhoff's law, Where E_b is the emissive power of the black body
10. $E_b /1 = E/ \alpha$
11. $\alpha= E/E_b$
12. As black body is a perfect radiator, it is used for the comparison of emissive powers. The ratio of emissive power (E) of a surface to the emissive power (E_b) of a perfect black body at the same temperature is known as emissivity (ε) of the surface.
13. $\varepsilon= E/E_b$
14. Therefore, emissivity of a body will be equal to absorptivity at thermal equilibrium.

Grey body

Although emissive power of a surface varies with wave length, for certain materials, it is constant. That is E/E_b of a perfect black body is a constant. Such *materials with constant emissivity are known as Grey bodies.* For grey bodies, it not necessary that the two bodies should be at thermal equilibrium to apply Kirchoff's law.

HEAT INTERCHANGERS & HEAT EXCHANGERS

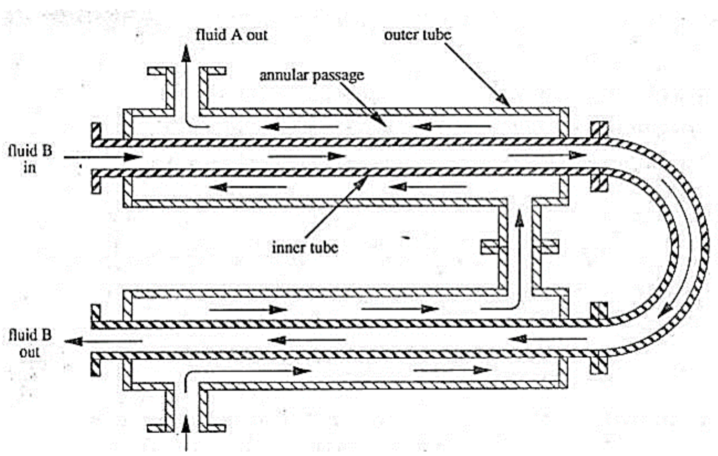
Heat exchangers are devices, which transfer heat from a hot fluid to a cold fluid usually across a metal tube wall. The most common heat exchangers used in the chemical industry are the SHELL AND TUBE types. In their simplest form, these consist of a bank, bundle, of small diameter tubes fitted inside a large diameter tube, usually referred to as the SHELL. One fluid flows through the tubes, while the other flows inside the shell, circulating around and between the small diameter tubes. Heat is transferred, from one fluid to the other, by CONDUCTION, across the tube walls, and then by FORCED CONVECTION, through the fluid. There are several different designs of shell and tube exchangers. A number of the more important ones are discussed in the following sections.

The use of heat interchangers are practicing in the commercial refrigeration industry for many years, often with virtually no consideration of both theoretical or practical concepts.

They can be found applied in refrigerant systems in many different ways for many different reasons. Here we cover the most common uses these refrigerant heat interchangers. Virtually all refrigeration systems will benefit from a heat interchanger.

Double pipe heat exchanger

One of the simplest types of heat exchanger is the double pipe design. It consists of two concentric pipes. Hot fluid in One fluid flows through the inner tube whilst the other flows through the annular space between the tubes. Heat passes from one fluid to the other by conduction through the inner pipe wall followed by forced convection through the fluid. The main advantages of this type of heat exchanger are that they are simple and cheap to make. They can often be made from standard diameter piping. Their main disadvantages are that they have low thermal efficiencies, and cannot handle large quantities of process fluid. The performance of the Double Pipe Heat Exchanger type can be improved by connecting two or more units together. Fluid A in Double pipe exchangers are sometimes referred to as ANNULAR or CONCENTRIC tube heat exchangers.



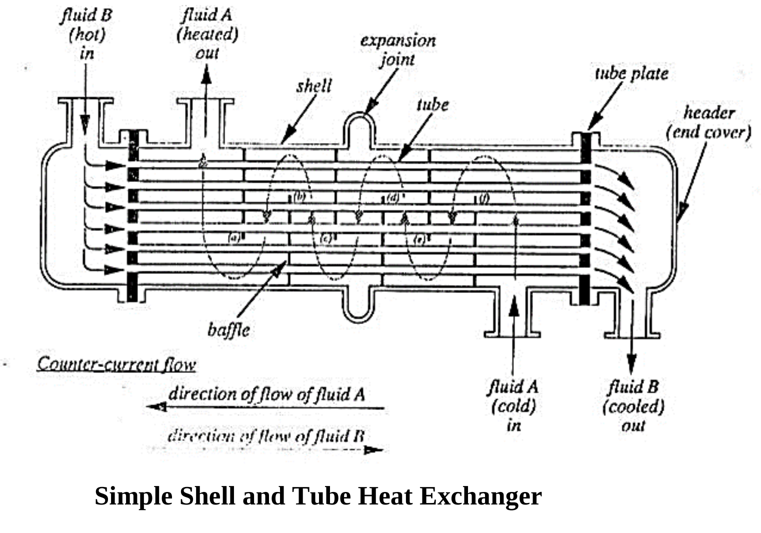
Double Pipe Heat Exchanger

Shell and tube heat exchanger

Simple shell and tube heat exchanger

The major features of this type of heat exchanger are:

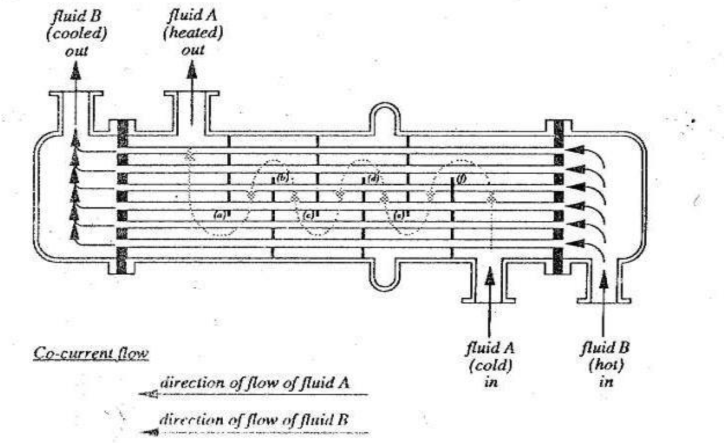
1. A large number of small diameter TUBES fitted into a TUBE PLATE to form a BANK or BUNDLE of tubes.
2. A large diameter tube called a SHELL into which the bank of tubes fits.
3. End covers or HEADERS, which are fitted over each end of the tube bundle.
4. INLET and OUTLET pipes fitted to the shell to allow fluid into and out of the shell.
5. INLET and OUTLET pipes fitted to each end of the cover to allow fluid to flow through the tubes.
6. An EXPANSION JOINT, fitted to the shell. This is included to relieve stresses due to thermal expansion. It allows the shell to expand and contract as the tube bundle expands and contracts.
7. A set of BAFFLES, i.e. plates set at right angles to the tube bundle. Their function is to cause shell side fluid to flow at right angles to the tubes. They also support the tubes within the bundle.



Simple Shell and Tube Heat Exchanger

Counter current and co-current flow

The heat exchanger is operating with CO-CURRENT FLOW since the two fluids flow through the exchanger in the same direction. Generally speaking, counter current flow provides the most efficient heat exchange since the temperature of the cold fluid can be raised to a temperature just below the temperature of the hot fluid. With co-current flow the heated fluid can be no hotter than the outlet temperature of the cooled heating fluid. However, the temperature distribution within a co-current heat exchanger is more even. For this reason, co-current is often preferred when heat-sensitive fluids are being heated.



Co-Current heat exchanger

OVERALL HEAT TRANSFER COEFFICIENT

The overall heat transfer coefficient U is the measure of overall ability of a series of conductive and convective barriers to transfer heat. It is commonly applied to the heat transfer in heat exchangers. (It can be applied to other situations too)

For the case of a heat exchanger can be used to determine the total heat transfer between the two streams in the heat exchanger by the following relationship.

$q = UA\ \Delta T_{LM}$

where q -Heat transfer rate(W-*watt*)

U-Overall heat transfer coefficient (W/(m^2.K)

A- Heat transfer surface area(m^2)

ΔT_{LM}-Logarithmic mean temperature difference (k)

The overall heat transfer coefficient takes into account the individual heat transfer coefficients of each stream and the resistance of the pipe material. It can be calculated as the reciprocal of the sum of a series of thermal resistances:

$1/UA = \Delta 1/hA = \Delta R$

R= Resistance(s)to heat flow in pipe wall (K/W) other parameters are as above.

The heat transfer coefficient is the heat transferred per unit area per kelvin. Thus area is included in the equation as it represents the area over which the transfer of heat takes place. The areas for each flow will be different as they represent the contact area for each fluid side.

R= A/k.x

x-Wall thickness

k-Thermal conductivity of the material(W/(m.k))

A-the total area of the heat exchanger (m^2)

This represents heat transfer by conduction in the pipe.

The thermal conductivity is a characteristic of the particular material. The thermal conductivity values are different for different materials.

DRYING

Theory

Drying is defined as the removal of small amounts of water or other liquid from a material by application of heat. Drying involves both heat and mass transfer operations. Heat must be transferred to the material to be dried in order to supply the latent heat required for vaporization of the moisture. Mass transfer involves the diffusion of water through the material to the evaporating surface and subsequent evaporation of the water from the surface

During the constant rate drying period, the moisture vaporized per time per unit area of drying surface remains constant. During the falling rate drying period, the moisture vaporized per time per unit area of drying surface decreases continuously. The two zones are determined by a breaking point called as critical moisture content (where the moisture content at which the constant rate drying period ends and the falling rate drying period starts. Equilibrium moisture content(EMC) can be defined as the moisture content of the solid when in equilibrium with the given partial pressure of vapour in the gas phase. Free moisture is the moisture content of the solid in excess of the equilibrium moisture content.

Applications in Pharmacy

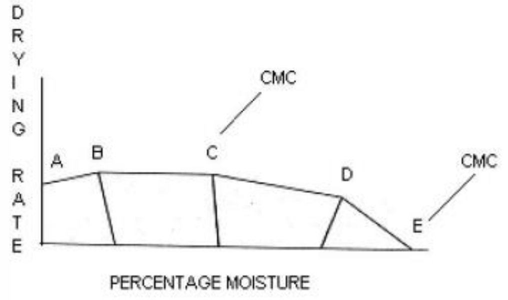
1. In the manufacture of bulk drugs, drying is the final stage of processing. Example is dried aluminium hydroxide gel.

2. Drying is necessary in order to avoid deterioration, for example, crude drugs of animal and vegetable origin

3. Granules are dried to improve the fluidity and compression characteristics.

4. Drying of viscous and sticky materials modifies the flow characteristics.

5. Removal of moisture makes the material light in weight and reduces the bulk.



TYPICAL DRYING RATE CURVE

AB- Constant Rate Period

BC- First Falling Rate Period

CD-Second Falling rate Period

CMC-Critical Moisture Content

Bound water is the minimum water held by the material that exerts an equilibrium vapour pressure less than the pure water at the same temperature.

Unbound water is the amount of water held by the material that experts an equilibrium vapour pressure equal to that of pure water at the same temperature.

Unbound water exists largely in the voids of the solid.

Equilibrium moisture content (EMC): It is the amount of water present in the solid which exerts a vapour pressure equal to the vapour pressure of the atmosphere surrounding it.

Free moisture content (FMC): Free moisture content (FMC) is the amount of water that is free (easy) to evaporate from the solid surface

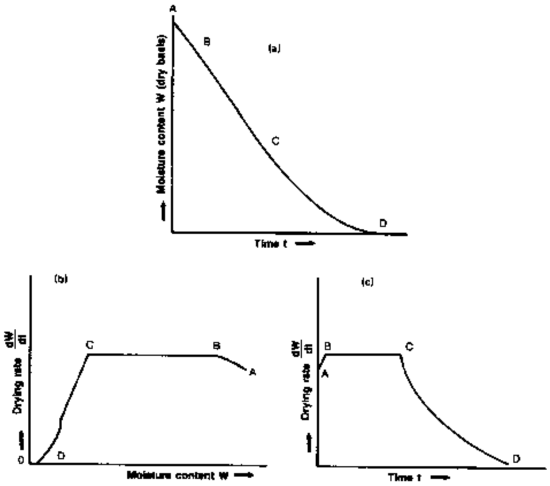
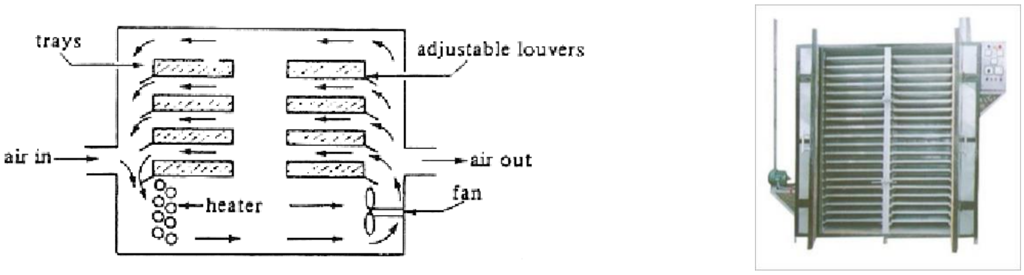


Figure 2 Drying profiles. (a) Moisture content versus time, (b) drying rate versus moisture control, and (c) drying rate versus time. (Adapted from Ref. 11.)

Tray Drier

Principle: In tray dryer, hot air is continuously circulated. Forced convection heating takes place to remove moisture from the solids placed in trays. Simultaneously, the moist air is removed partially.



Tray drier

Construction

It consists of rectangular chamber whose wall is insulated. Trays are placed in the chamber according to need.

Working

1. Wet solid is loaded into trays.
2. Trays are placed in the chamber.

3. Fresh air is introduced through inlet which passes through the heaters and heated up.
4. The hot air is circulated by means of fans at 2 to 5 meter per second.
5. The water is picked up by air and moist air is removed from outlet.
6. During the cycle of drying only 10 to 20 % of fresh air is introduced and 80 to 90% air is circulated back.

Uses

In the drying of:

1. Stcky materials
2. Plastic substances
3. Crysatalline and granular substances
4. Precipitates and pastes
5. Crude drugs, powders,tablet granules
6. Parts of machinaries

Advantages

1.It is for batch process.

2. Loading and unloading can be done with out loss of material

Disadvantages

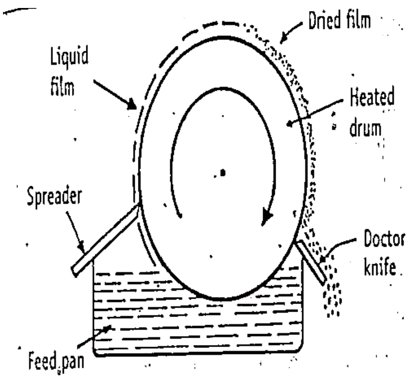
1. Material is over heated
2. It is long duration process
3. Loading and unloading, labour costs are more

Drum drier (Roller Drier/Film drier)

Principle: In drum dryer, a heated hallow metal drum rotates on its longitudinal axis, which is partially dipped in the solution to be dried. The solution is carried as a film on the surface of the dryer and dried to form a layer. A suitable knife scraps the dried material, while the drum is rotating

Construction

It consists of horizontally mounted hollow steel drum of 0.6 to 3m diameter and 0.6 to 4m length. Below the drum feed pan is placed in such a way that the drum dips partially into the feed. One side of the drum spreader and other side knife is placed. Storage bin is placed to collect the material,



Drum drier

Working

1. Steam is passed inside the drum is rotated at 1 -10 rotations per minute
2. The liquid present in feed pan adheres to the surface of drum. Material is dries during the rotation of drum and collected in storage bin by using knife.

Note: Drying process in drum dryer completes in less than one cycle so the time of contact of material with drum is only 6 to 15second

Uses

In the drying of:

1. Slurries, suspensions, solutions etc.
2. Milk products, starch, ferrous salts, zinc oxide suspension, kaolin suspension, yeast, malt extract, glandular extracts, antibiotics, calcium and barium chlorides.
3. DDT and other insecticides.

Advantages.

1. Drying time is less; so heat sensitive materials can be handled
2. Occupies less space
3. Since a thin film is formed, the rate of heat transfer and mass transfer are high.
4. Complete and perfect drying of the product.

Disadvantages

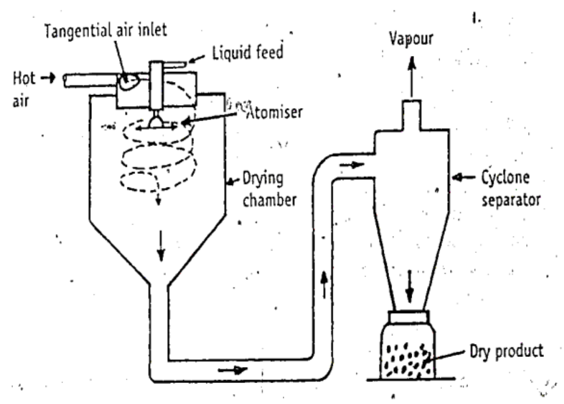
1. Maintenance cost is high
2. Skilled and trained operators are needed
3. Not suitable for solutions of salts with less solubility.

Spray drier

Principle: in spray dryer the fluid to be dried is atomized into fine droplets which are thrown radially into moving stream of hot gas. The temperature of the droplets is immediately increased and fine droplets get dried instantly in the form of spherical particles.

Construction

1. It consist of large cylindrical drying chamber with a short conical bottom made up of stainless steel.(diameter 2.5 to 9.0 m and height 25 m or more)
2. An inlet for hot air placed in the roof and another inlet carrying spray disk atomizer is also set in the roof.
3. The spray disk atomizer is about 300 mm in diameter and rotates at a speed of 3000 to 50,000 rpm.
4. Bottom of the dryer is connected to cyclone separator.



Spray drier

Working

Drying of material in spray dryer involves 3stages;

1. Atomization of liquids: The feed is introduced through the atomizer either by gravity or using suitable pump.

2. Drying of the liquid droplets: Fine droplets are dried in the drying chamber by supplying hot air through the inlet.

3. Recovery of the dried product: Centrifugal force of atomizer drives the droplets to follow helical path. Particles are dried and collected at the conical bottom.

All these processes are completed in few seconds. Particle size obtained is ranging from 2 to 500 mm. maximum size of spray dryer has capacity of 2000 kg per hour.

Uses

It Can be used if

1. The quantity of the material to be dried is too large
2. The product is thermolabile, hygroscopic or undergoes chemical decomposition.

Examples of products dried using spray drier: Acacia, Gelatin, Chloramphenicol, Plasma, vaccines, vitamins etc.

Advantages

1. Quick drying in a few seconds.
2. Economical in terms of labour cost.
3. Product uniformity and controlled size can be obtained by using proper atomizer.
4. Spray dried products shows excellent solubility
5. Suitable for sterile products
6. Globules of emulsion can be dried, which upon reconstitution will give excellent emulsion.

Fluidized bed drier (FBD)

Principle: In fluidized bed dryer hot air is passed at high pressure through a perforated bottom of the container. The granules are lifted from the bottom and suspended in the stream of air, this condition is called as fluidized state. The hot gas surrounding every granule to completely dry them.

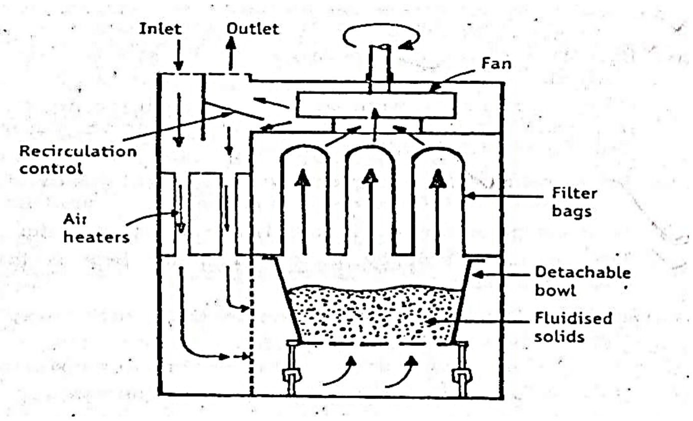
Construction

Fluidized bed dryer is available in two forms;

i) Horizontal FBD ii) Vertical FBD

The dryer is made up of SS or plastic.

1. A detachable perforated bowl is placed at the bottom of the dryer which is used for charging and discharging of material.
2. A fan is mounted in the upper part for circulating hot air.
3. Fresh air inlet, pre-filter and heat exchanger are connected serially to the required temperature.
4. Bag filters are placed over bowl for recovery of fines.



Fluidized bed dryer

Working

The wet granules to be dried are placed in detachable bowl and bowl is placed into the

dryer.

1. Fresh air is allowed to pass through pre-filter which subsequently gets heated by passing through heat exchanger.
2. Hot air passed through bottom of bowl and fan is allowed to rotate.
3. The granules rise in the container because of high velocity gas and later fall back in random motion.
4. The gas surrounds every granule to completely dry them. The air leaves the dryer by passing through bag filter.
5. The entrained particle remains adhere to the inside surface of bags. Periodically the bags are shaken to remove the particles.
6. The material is collected after some time so as to reach the ambient temperature.

Uses:

1. For drying granules.
2. It can be used for drying, mixing and granulation process.
3. It is modified for coating of granules

Advantages

1. Less time for drying; it is 15 times faster than tray drier
2. Drying capacity ranges from 5 to 200 kg per hour
3. Handling is simple and labour cost is less.
4. Mixing efficiency is high
5. High output from small floor size
6. Can be used for thermolabile substances since the drying time is short.
7. Can be used in batch and continuous process.

Disadvantages

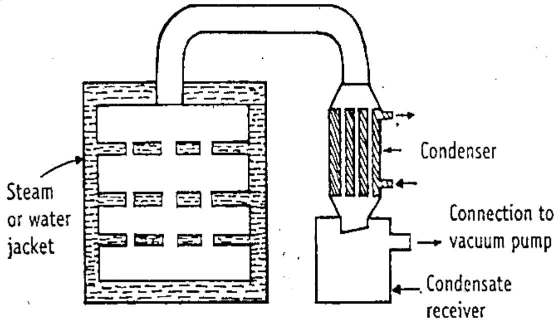
1. Some organic material may develop electrostatic charge so requires earthing.
2. Due to attrition fines are observed in large quantity.

Vacuum drier

Principle: In vacuum dryer, material is dried by the application of vacuum. When vacuum is created the pressure is lowered so that water boils at lower temperature. Hence water evaporates faster. The heat transfer become efficient.

Construction:

1. It is made up of cast iron heavy jacketed vessel.
2. The enclosed space (approx. 1 .5 meter cube) is divided into a number of portions by means of 20 hollow shelves which are part of the jacket.
3. These shelves provide larger surface area (about 45 to 50 m^2) for conduction of heat. Over these shelves metal trays are placed.
4. The oven is connected to vacuum pump by placing condenser in between.



Vacuum drier

Working

1. Material to be dried is placed on trays.
2. The trays are placed on shelves and pressure is decreased up to 30 to 60 kilopascals.
3. Steam or hot air is supplied into hollow space. Drying of material is done by presence of vacuum.
4. Water vapour passes into the condenser where it is condensed.
5. At the end of drying vacuum line is disconnected and material is collected from trays.

Uses

Vacuum dryer can be used in the drying:

1. Heat sensitive materials
2. Dusty and hygroscopic materials
3. Drugs containing toxic solvents
4. When the final product needed is porous type.

Advantages

1. Provides large surface area for heat transfer and thus drying
2. Handling of the equipment is easy
3. Hot water of desired temperature can be provided
4. Electrically heated hollow shelves can be used.

Disadvantages

1. Heat transfer coefficients are low in vacuum drier.
2. Limited capacity and can be used for batch process only
3. More expensive; labour and running costs are also high.
4. Chances of overheating of the material as they are in contact with steam heated surface for a long time.

Freeze Drier

In freeze drying water is removed from the frozen state by sublimation. (direct change of water from solid into vapour) It is also known as sublimation drying It is also known as lyophillization as the dried product has great affinity for water (lyophilic = water loving)

Construction

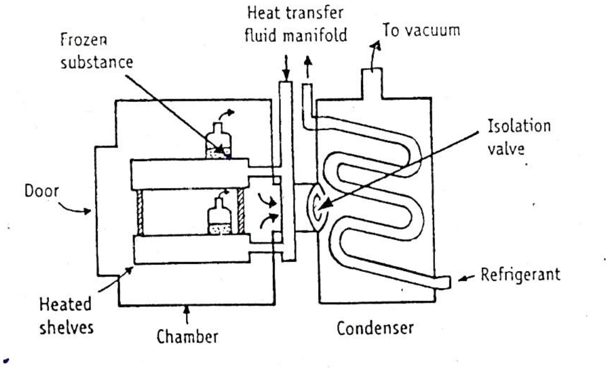
Freeze dryer is consisting of

1) Drying chamber in which trays are loaded

2) Heat supply in the form of radiation

3) Vapour condensing or adsorption system

4) Vacuum pump or steam jacket or both



Freeze Drier

Working

It involves following steps;

1) Preparation and pretreatment

2) Pre-freezing for solidifying water

3) Primary drying (sublimation of ice)

4) Secondary drying (removal of residual moisture)

5) Packing

Preparation and pretreatment

The solution is pre concentrated under normal vacuum drying. This reduces the actual drying time by 8 to 10 times. The final product becomes more porous.

Pre-freezing for solidifying water

Solutions are packed in vials, ampoules or bottles are frozen in cold shelves at about minus 50-degree C, during which the cabinet is maintained at low temperature and atmospheric pressure. During this time large ice crystals with wholes are formed on sublimation of ice, which is responsible for porous final product.

Primary drying (sublimation of ice under vacuum)

The material to be dried is spread as much large surface as possible for sublimation. The temperature and pressure are maintained below the triple point of water (0.0098-degree C and 0.533 kilo pascals)

The temperature should be brought below the eutectic point. The pressure and temperature at which frozen solids vaporizes without conversion into liquid is called as eutectic point. (minus 10 to 30-degree C usually)

Primary drying stage easily removes removable moisture (about 98 to 99%).Still traces of moisture may present in the sample.

Secondary drying (removal of residual moisture)

Traces of moisture is removed at this stage. Temperature of solid is raised as high as 50 to 60-degree C but vacuum is lowered below that is used in primary drying. (50 mm Hg) The rate of drying is very low and it takes about 10 to 20 hours.

Packing

After vacuum is replaced by inert gas, the bottles and vials are closed.

Uses

In the production of dosage forms like injections, solutions and suspensions. It is used for the drying of a number of products

1. Blood plasma and plasma products.
2. Bacterial and viral cultures
3. Human tissues
4. Antibiotics and plant extracts

5. Steroids, vitamins and enzymes.

Advantages

1. Thermolabile substances can be dried
2. Reconstitution of the product is easy; it is porous in nature
3. Denaturation does not occur
4. Loss of volatile materials are less
5. Moisture levels can be kept as low as to avoid de composition.
6. Materials can be dried in its final containers like vial.
7. Sterility can be maintained
8. The final product can be stored at ambient temperature.

Disadvantages

1. The product is prone to oxidation. Therefore, it should be packed in vacuum or using an inert gas.
2. Equipment and running costs are too high.
3. Difficult in solutions containing non aqueous solvents
4. The period of drying is high (Usually, rarely less than 10 hours)

DISTILLATION

Distillation can be defined as the process of separation of the components of a liquid mixture by a process of vaporization followed by condensation

Two steps in Distillation

1. Transfer the vapour to another place and condense it
2. Converting the liquid into vapour phase

If one component is volatile and others non volatile, it is easy to separate volatile components from nonvolatile components by distillation. So distillation can be considered as a *purification/separation* method. But it is a partial separation method because when heat is applied, more volatile components will be condensed than less volatile component.

Differences between distillation,evaporation and drying

Distillation	**Evaporation**	**Drying**
Used when condensed vapour is required as a product.	Used when the concentrated liquid residue is needed as a product. The temp. of the liquid is maintained below its B.P. further vapour is not condensed unless recovery is essential	Used when dried solid residue is required as a product.

Applications

- Separation of volatile oils
- Purification of organic solvents
- Manufacture of official preparations
- Refining of petroleum products
- Recovery of solvents
- Quality control methods
- Separation of drugs obtained from plant and animal resources.
- Purification of drugs obtained from chemical process.
- Separation of Volatile oil from clove, anise seeds, eucalyptus leaves, cardamom, cinnamon, fennel etc. by steam distillation.

- Absolute alcohol (100% ethanol) can be obtained by azeotropic distillation.
- Spirit of nitrous ether & aromatic spirit of ammonia by simple distillation.
- Distilled water & water for injection are prepared by simple distillation.
- Vitamin A is separated from fish liver oil using molecular distillation.

Theory of distillation

- ☐ Distillation is a process of separating and purifying the components in a liquid mixture.
- ☐ Vapour –liquid equilibrium relationship is very important in this process
- ☐ When two liquids are mixed together, they may be miscible with each other in all proportions. Such miscible liquids are known as ***binary mixtures.***

Example: Ethyl alcohol and water, Acetone and water, Benzene and carbon tetra chloride

Ideal and real solutions

- Ideal solution is defined as the one which there is no change in properties of the components other than dilution, when they are mixed to form a solution. They are characterized by vapour pressure, one of the important properties of liquid. Ideal solutions will obey ***Raoult's law.*** They are also known as ***Perfect solutions.***

Ex: Benzene and toluene, n-hexane and n-heptane, Ethyl bromide and ethyl iodide

Raoult's law

This law establishes a quantitative relationship between the concentration and vapour pressure of the mixture.

Raoult's law states that the partial vapour pressure of each volatile component is equal to the vapour pressure of the pure constituent multiplied by its mole fraction in the solution at a given temperature.

Consider a mixture of miscible liquids A and B. In this mixture,

Let the partial vapour pressure exerted by A= PAkPa

Let the partial vapour pressure exerted by B= PBkPa

Let the partial vapour pressure exerted by the pure component of A= p^0AkPa

Let the partial vapour pressure exerted by the pure component of B= p^0BkPa

Let the mole fraction concentration of liquid A = XA

Let the mole fraction concentration of liquid B = XB

Raoult's law may be mathematically expressed as ,

Partial vapour pressure of a liquid = Vapour pressure of pure liquid x Mole fraction of the liquid

$$PA = P^0A \ XA \quad \ldots\ldots\ldots(1)$$

$$PB = P^0B \; XB \quad \ldots\ldots\ldots(2)$$

A mixture of ethylene chloride and benzene obeys Raoult's law. When two liquids are mixed, the vapour pressure of each one is reduced by the presence of other to the extent of dilution of each phase.

Dalton's law

Dalton's law of partial vapour pressure states that the total pressure exerted by a mixture of ideal gases may be considered as the sum of the partial vapour pressure exerted by each gas ,if alone were present and occupied the total volume.

It can be mathematically expressed as,

Total vapour pressure=Partial pressure of A + Partial pressure of B

$$P = PA+PB \quad \ldots\ldots\ldots\ldots(3)$$

Substituting equations (1) and (2) in equation (3) gives

$$P= P^0A \; XA + P^0BXB \quad \ldots\ldots\ldots\ldots(4)$$

Applications

- ✓ In simple Distillation

According to an ideal solution, the component having relatively greater vapour pressure will be distilled first. This principle is used in Simple distillation.

Real solutions

Most system shows different degree of variation from Raoult's law, depending on the nature of the liquid and temperature. These solutions are known **as *Real solutions***.

Reasons for Deviations from Raoult's Law

Deviations are observed because of solute-solute, solvent-solute and solvent-solvent interactions in a system (mixture)are un equal.

Examples: Carbon tetra chloride and cyclohexane, Chloroform and acetone

Two types of deviations

1. Positive Deviation
2. Negative Deviation

Positive deviation

In some liquid systems, the vapour pressure is ***greater*** than the sum of the partial pressures of the individual components. Such systems are said to exhibit ***positive deviation*** from Raoult's law.

Ex: Carbon tetra chloride and Cyclohexane., Benzene and ethanol. The degree of deviation from Raoult's law decreases as the temperature increases, since the differences in the nature of components are reduced at higher temperatures. Similarly a decrease in temperature may lead to a decrease in miscibility of two components and phase separation.

Negative deviation

In some liquid systems ,the vapour pressure is ***lower*** than the sum of the partial pressures of the individual components. Such systems are said to exhibit *Negative deviation* from Raoult's law.

Examples: Chloroform and Acetone, Pyridine and acetic acid, Water and nitric acid

Negative deviation occurs when interactions such as hydrogen bonding ,salt formation and hydration occur between the components of the solution. As a result, the vapour pressure of each component lowers with respect to the behaviour of an ideal solution. Raoult's law does not apply over the entire concentration range in a non ideal solution.

Applications

- ✓ In fractional distillation of intermediates and drugs
- ✓ In the Purification of alcohol and organic solvents

Complete separation of components of a mixture may not be achieved if large positive or negative deviations from Raoult's law are observed. Such solutions will form *Azeotropic mixtures.*

Azeotrope *-A mixture of two liquids which has a constant boiling point and composition throughout distillation.*

Volatility

It can be defined as the equilibrium partial pressure of the substance in the vapour phase divided by the mole fraction of the substance in the solution.

Ex: If a substance A in a liquid mixture has partial pressure Pa and its concentration in the mixture is X_A on mole fraction scale. Then volatility of A(v_A) may be mathematically expressed as

$$\text{Volatility of component } A,\ v_A = \frac{\text{Partial vapour pressure of A}}{\text{Mole fraction of A in solution}}$$

$$= \quad P_A/X_A \ldots\ldots\ldots\ldots\ldots\ldots (5)$$

***** The Volatility of a material in its pure state is equal to the vapour pressure of the material.***

Relative volatility

Consider a liquid mixture containing two components A and B. In such a case, the volatility of one component is expressed in terms of the second. Relative volatility can be defined as,

$$\text{Relative volatility} = \frac{\text{volatility of component A}}{\text{volatility of component B}} = VA/VB \ldots\ldots\ldots\ldots (6)$$

Relative volatility is commonly expressed with the higher of two volatilities in the numerator. This means that the relative volatility should never have a numerical value less than 1.0

Since ,$v=p/X$ (eqn.5),it may be substituted in eqn.(6)

$$a = \quad P_A/X_A = P_AX_B \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (7)$$

$P_B/X_B \quad P_B X_A$

According to Dalton's law, the partial vapour pressures of *A* and *B* may be expressed as:

$$P_A = Y_A.P \text{ …………………………………….}(8)$$

$$P_B = Y_B.P \text{ …………………………………….}(9)$$

Y_A - *Mole fraction A in vapour state*

Y_B- *Mole fraction B in vapour state*

P- Total pressure of the vapour,kpa

Relative volatility may be expressed by substituting equations (8) and (9) in equation (7) gives:

$$a_{AB} = \frac{Y_A P.X_B}{Y_A P.X_B} = \frac{Y_A X_B}{Y_A X_B} \text{ ………………..}(10)$$

Equipments for distillation

- **STILL**-Liquid is heated in a vessel or container
- **CONDENSER**-Vapour occurs in still made to pass through an apparatus which cools the vapour
- **RECEIVER**-Where liquid is collected in suitable vessel .

Still

- It is a vaporizing chamber and used to place the materials to be distilled.
- If the still is too large, super heating and decomposition of liquid may occur.
- The still is heated by a suitable means.Ex: steam for vaporization of the volatile components
- The boiling temperature of the liquid is very important so provision for thermometer is provided in the still.
- A condenser is attached to the still using appropriate joints.
- Liquids which have the tendency to bump or froth, a *trap* is inserted between distillation flask and condenser.
- In Laboratories, round bottom flasks made of glass are commonly used as stills.

Condenser

- Condenser is a heat exchanger, which is kept cold by circulating water through water jacket.
- Condenser helps in condensing the vapour.
- The boiling point and volatility of a substance are the main two factors governing the choice of a condenser.

Main points in construction of a condenser

1. The condenser must be easy to clean
2. The cooling surface must be large enough, since rate of condensation is proportional to the rate at which the surface is cooled.
3. Condenser is placed in inclined position to prevent formation of film of condensed liquid which may affect the rate of condensation.

Cooling water is arranged to move on the counter current principle-i.e, its direction of flow is opposite to that of the flow of vapour to be condensed

The condenser is connected to the receiver through a suitable adapter. In vacuum distillation ,the adapter is provided with a facility to connect to the vacuum pump.

Different types of condenser

- I. Single-surface condensers Ex: Liebig condenser, Spiral or glass –worm condenser
- II. Double-surface condensers –High efficient condensers
- III. Multi-tubular condensers-For large scale operations like manufacturing distilled water and water for injection.

Reciever

- It is used to collect the distillate. It may be a simple flask or modified flasks.Ex: Florentine receivers
- Sometimes, the receiver is immersed in an ice bath or any other freezing mixture. This minimizes loss of volatilization.
- Florentine receivers are used for the separation of oil and water. These are of two types

1. Type I- For the separation of oil heavier than water
2. Type II- For the separation of oil lighter than water

Distillation methods

1. Simple distillation
2. Flash distillation
3. Fractional distillation
4. Azeotropic and extractive distillation
5. Distillation under rdeuced pressure
6. Steam distillation
7. Molceular distillation
8. Distructive distillation

9. Compression distillation

Simple Distilation

- **Simple Distillation** is the process of converting a single constituent from a mixture into its vapour, transferring the vapour to another place and recovering the liquid by condensing the vapour, usually by allowing it to come in contact with a cold surface
- Since the process depend on the volatilities and vapour pressure of components ,it is called as *differential distillation.*

Principle

If the liquid under study is volatile and remaining components are non volatile in nature, Simple distillation is the useful means of purification and separation of liquids

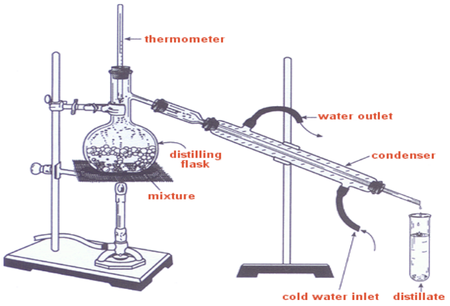
- Liquid boils when its vapour pressure is equal to the atmospheric pressure.
- Simple distillation is carried out at its boiling point.
- Heat is supplied to the liquid to boil it.
- The resulting vapour is transferred to a separate place and condensed.

Applications

1. In the preparation of distilled water and water for injection
2. In the preparation of volatile and aromatic waters
3. In the purification of organic solvents
4. In the official preparation of aromatic spirit of ammonia
5. In the separation of non-volatile solids from volatile liquids.

Lab Apparatus For Distillation

1. It consists of a distillation flask with a side arm sloping downwards.
2. Condenser is fitted into the side arm by means of a cork.
3. The condenser is usually a water condenser which is jacketed for water circulation.
4. The condenser is connected to a receiver flask using an adaptor.
5. In laboratories, the whole apparatus is made of glass.



Simple Distillation

Procedure

1. The liquid to be distilled is filled in the flask to one-half to two-third of its volume.
2. Bumping is avoided by using porcelain bits/porous pot pieces
3. A thermometer just below the level of the side arm is inserted into the cork and fixed
4. Water is circulated through the jacket of the condenser as shown in the diagram.
5. The contents are heated gradually-the liquid begins to boil after sometime.
6. The vapour rises up and passes down the side arm into the condenser.
7. The temperature rises gradually and reaches a constant value. *This temperature is equal to the boiling point of the liquid.*
8. The vapour is condensed and collected in to the receiver
9. The flame is adjusted so that the distillate is collected at the rate of one or two drops per second.
10. Distillation should be continued until a small volume of liquid remains in the flask.

Simple distillation-large scale equipment

- A simple still made up of copper, stainless steel or any other suitable material is used fore industrial purpose
- A thermometer is fixed to the still to note the temperature of the boiling liquid.
- An observation window is provided to monitor the process inside
- The still is connected to a condenser and receiver.
- The still is heated using steam
- A steam out let and inlet at the bottom of the still is provided for removing the condensed steam.

Working

1. The liquid to be distilled is filled in the STILL to one-half to two-third of its volume.
2. Bumping is avoided by using porcelain bits/porous pot pieces
3. A thermometer just below the level of the side arm is inserted into the cork and fixed
4. Water is circulated through the jacket of the condenser as shown in the diagram.
5. Steam is passed through the inlet and the contents are heated gradually-the liquid begins to boil after sometime.
6. The vapour rises up and passes down the side arm into the condenser.
7. The temperature rises gradually and reaches a constant value. *This temperature is equal to the boiling point of the liquid.*
8. The vapour is condensed and collected in to the receiver

9. The flame is adjusted so that the distillate is collected at the rate of one or two drops per second.
10. Distillation should be continued until a small volume of liquid remains in the still.

Preparation Of Purified Water Bp And Water For Injectioon Bp By Distillation

i. Gases dissolved in the raw water such as carbon di oxide, ammonia etc. must be removed. These should not be allowed to contaminate the distillate.
ii. The carryover of soluble materials in the droplets must be avoided ,particularly if the product is required for use as water for injection.
iii. Entrapment of liquid droplets by the vapour must be prevented .For this purpose, *baffles* are included in the path of the vapour between boiler and condenser.
iv. Contamination of the distillate by pyrogen from feed water must be avoided.
v. The residue of solids must not be concentrated to a point where hydrolysis occurs. Otherwise the distillate may be contaminated by volatile material produced during hydrolysis. For example, hydrolysis of chlorides produces hydrochloric acid.

Construction & Working

- Water enters at the base of the still and rises in the jacket, which contain a number of tubes.
- In the condenser tubes, the condensed liquid descends.
- The rising feed water gets heated on account of condensate in tubes.
- The rate of flow is adjusted in such a way that the water gets heated to 90 ^{0} C – 95 ^{0} C.
- The dissolved gases escapes to the atmosphere.
- The heated water then enters the boiler, in which steam is circulated under pressure through a copper coil.
- The descending steam is condensed in to distilled water., which flows from the lower ends of the tubes.

Steam distillation

Steam distillation is a kind of differential distillation process carried with the aid of steam and used for the separation of high boiling substances from nonvolatile impurities.

Differential Distillation: Distillation based on the differences in volatilities and vapour pressures of the components of the mixture.

Steam distillation is used to separate turpentine oil from a mixture of turpentine oil-water mixture. A mixture of immiscible liquids begins to boil when the sum of vapour pressure is equal to the atmospheric pressure. In case of a mixture of water and turpentine oil, the mixture boils below the boiling point of pure water. But actually, the turpentine oil alone boils at a much higher temperature than that of water. As a result, the high boiling substances may be distilled at a temperature much below its boiling point, when water or steam is used. The turpentine is distilled along with water. These liquids are immiscible and can be separated using a separating funnel. Thus it is possible to separate and purify one liquid from a mixture.

Applications

1. Steam distillation is used for the separation of immiscible liquids like toluene and water

2.This method is used for the extraction of volatile oils such as clove, anise and eucalyptus.

3. It is useful in the purification of liquids with high boiling pint.

4.Camphor is distilled by this method

5. Aromatic waters can be prepared by this method

Advantages

Volatile oils can be separated at a lower temperature in steam distillation, without any decomposition and loss of aroma. If a substance has low volatility, it can be satisfactorily distilled provided its molecular weight is considerably higher than water.

Disadvantages

Steam distillation is not suitable when immiscible liquid and water react with each other

Flash distillation

Defined as a process in which the entire liquid mixture is suddenly vaporized (flash)by passing the feed from a high pressure zone to a low pressure zone

Since the separation is done when the liquid and vapour are in equilibrium, flash distillation is also called as *Equilibrium distillation.*

Principle

When a hot liquid mixture is allowed to enter from a high pressure zone to low pressure zone , the mixture is suddenly vaporized. This process is called flash vaporization. During this process, the chamber gets cooled..The individual vapour phase molecules of high boiling fraction get condensed and low boiling fraction remains as vapour. Therefore the liquid and vapour is kept in intimate contact until equilibrium is achieved. The liquid fraction is collected separately. The vapour is separated from the liquid and further allowed to condense.

Uses

Flash distillation is used for separating components ,which boil at widely different temperatures. It is widely used in petroleum industry for refining crude oil.

Advantages

1. It is a continuous process
2. It is used for obtaining multi-component systems of narrow boiling range, especially in oil industry.Ex:Petroleum and ether.

Disadvantages

1. Not effective in separating components of comparable volatility
2. It is not efficient

Construction

1. It consists of a pump which is connected to a feed reservoir.
2. Pump helps in pumping the feed into the heating chamber which is equipped with suitable heating mechanism.
3. The other end of the pipe is directly introduced in to the liquid-vapour separator through a reducing valve.

The vapour outlet is provided at the top of the separator and liquid out let is provided at the bottom.

Working

1) Feed is pumped through a heater at certain pressure.
2) The liquid gets heated, which enters the vapour-liquid separator through a pressure reducing valve.
3) Due to the drop in pressure, the hot liquid flashes, which enhances the vaporization process.
4) The sudden vaporization enhances cooling.
5) The individual vapour phase molecules of high boiling fraction gets condensed, while low boiling fraction remains as vapour.
6) The mixture is allowed to keep for sufficient time so that vapour and liquid fractions separate and will achieve equilibrium.
7) The vapour is separated through a pipe from above and liquid is collected from the bottom of the separator.
8) It is possible to get continuous distillation by continuous feeding in to the still

Rectification (*Fractional Distillation*)

Fractional distillation is a process in which vaporization of liquid mixture gives rise to a mixture of components from which the desired one is separated in pure form.

Since a part of the vapour is condensed and returned as liquid, this method is also known as rectification.

Used to separate miscible volatile liquids whose boiling points are close by means of a fractionating column

Differences between simple distillation and fractional distillation

Simple Distillation	Fractional distillation
Vapour is directly passed through the condenser.	Vapour must pass through a fractionating column in which partial condensation of vapour is allowed to occur.
Condensate is directly collected into the receiver.	Condensation takes place in the fractionating column so that a part of the condensing vapour returns to the still

Principle

Fractional distillation is a process in which counter current diffusion of the components at each equilibrium stage.

When a liquid mixture is distilled, the partial condensation of the vapour is allowed to occur in a fractionating column. In the column, ascending vapour from the still is allowed to contact with the condensing vapour returning to the still. This results in enrichment of the vapour with more volatile component. By condensing the vapour and re heating the liquid repeatedly, equilibrium between the liquid and vapour is set up at each stage, which ultimately results in the separation of more volatile component.

Applications

It is used in the separation of miscible liquids such as acetone and water, chloroform and benzene

Advantages

- ✓ Flash distillation is a continuous process
- ✓ It is used for obtaining multi-component systems of narrow boiling range, especially in oil refinery. Ex: Petroleum, Ether etc.

Disadvanatges

- ✓ It is not effective in separating components of comparable volatility.
- ✓ It is not suitable for two component systems
- ✓ It is not an efficient distillation when nearly pure components are needed, because the condensed vapour and residual; liquid are far from pure..

Theory

According to the principles of colligative properties, when a substance is dissolved in a liquid, the vapor pressure of the solvent is lowered. When two miscible liquids are mixed, each may be considered as a solution of one in the other. The pressure exerted by each one is known as partial pressure.

According to Dalton's law, the total pressure exerted by a gaseous mixture is the sum of the individual partial pressures of the component gases. If *A* and *B* are two miscible liquids and *PA* and *PB* represent their partial pressures respectively, then Dalton's law may be expressed as,

$$\text{Total pressure} = PA + PB$$

Boiling Point- Composition Curves For Mixtures

Boiling points curves are helpful in predicting whether the separation is possible or not, Easy or difficult and also these are helpful in designing the equipments for fractional distillation

1. Mixtures of liquid *A* and *B* are prepared in different proportions
2. Boiling point of each mixture is determined.
3. Liquid composition of each component is analysed at its boiling point
4. Vapour composition of each component is analysed at its boiling point
5. The boiling points are plotted on y axis against composition of the mixture(x axis)
6. The upper curve represents the vapour phase composition
7. The upper curve represents the liquid phase composition
8. The different areas correspond to the existence of liquid, vapour and liquid plus vapour phases.

Fractional distillation

TYPE I Miscible Liquids for Ideal solutions

Fractional distillation is suitable for a system when the boiling point of the mixture is always intermediate between those of pure components. There is neither a maximum nor a minimum in the composition curves as shown in the figure. These systems are known as Zeotropic mixtures.Ex: Benzene and toluene, carbon tetrachloride and cyclohexane, water and ethanol.

Fractional distillation-Azeotropic Mixtures

Many liquid mixtures cannot be separated completely into pure components by simple distillation, because the volatilities of components are equal. Such mixtures are known as azeotropic mixtures.

(***Azeotrop****e* in Greek means boil unchanged)

Azeotropic mixture is a mixture of solution that distills unchanged at a constant temperature

Such solutions are also known as constant boiling mixtures. Example: 89.4 mol percentage mixture of ethanol and water at atmospheric pressure. If the volatilities of the components of a mixture is equal, the mixture is said to have a relative volatility of 1.0. Hence further purification cannot be obtained by conventional distillation methods. These solutions deviate the Raoult's law to a large extent

Minimum boiling point azeotropic solutions-Type II Solutions (Non ideal solutions)

System that exhibits a minimum in the boiling point construction curve is shown in the figure. Such a system is known as azeotropic mixture with a maximum vapour pressure or minimum boiling point.Ex: Chloroform and acetone, Pyridine and nitric acid, water and nitric acid.

The azeotropic mixture has a lower boiling point than that of component with the least boiling point. At the minimum boiling point temperature, the liquid composition remains constant and is equal to the vapour composition (arising from such a liquid system)

All mixtures of compositions lying between C and T (trough) can be separated by continuous fractional distillation. In this process, pure liquid C is recovered from the still and a mixture with constant composition (as of T) is obtained as a distillate from the condenser. In a similar way, all mixtures of compositions lying between T and D can be separated by continuous fractional distillation. In this process, pure liquid D is recovered from the still and mixture with constant composition (as of T) as condensate.

Since vapour gives constant composition of mixture, liquid curve i.e., liquid present in the still should be considered for the analysis.

Azeotropic and extractive distillation

Azeotropic mixture is a mixture of solution that distills unchanged at a constant temperature

Such solutions are also known as constant boiling mixtures. Example: 89.4 mol percentage mixture of ethanol and water at atmospheric pressure. If the volatilities of the components of a mixture is equal, the mixture is said to have a relative volatility of 1.0. Hence further purification cannot be obtained by conventional distillation methods. These solutions deviate the Raoult's law to a large extent. Hence azeotropic distillation is employed.

Raoult's law states that the partial vapour pressure of each volatile component is equal to the vapour pressure of the pure constituent multiplied by its mole fraction in the solution at a given temperature.

Raoult's law may be mathematically expressed as ,

Partial vapour pressure of a liquid = Vapour pressure of pure liquid x Mole fraction of the liquid

$$PA = P^0A \;\; X A$$

$$PB = P^0B \;\; X B$$

There are two types of azeotropic mixtures.

1. Minimum boiling point azeotropic solution(Type II Solution)

These are azeotropic mixtures with a maximum vapour pressure and minimum boiling point.

Examples: Chloroform and Acetone, Water and nitric acid

2. Maximum boiling point azeotropic solution(Type III solution)

3. These are azeotropic mixtures with a minimum vapour pressure and maximum boiling point.

Examples: Water and ethanol, Benzene and Ethanol

Applications

In the separation of absolute alcohol from fermentation liquor (Which contain 8 to 10 % ethanol)

Advantage: Simple distillation assembly can be used.

Theory

Azeotropic solutions cannot be completely separated by simple, steam, vacuum or fractional distillations because both the vapour and liquid in the still contain a mixture of components. Such a mixture can be separated by azeotropic distillation.

Azeotropic distillation is a distillation method in which an azeotropic mixture is broken by the addition of a third substance, that forms a new azeotropic with one of the components.

The relative volatility of the individual components can be changed by adding a third substance. For example, benzene OR glycerin is added to the azeotropic mixture of water and ethyl alcohol (Rectified spirit).Glycerin breaks the water-alcohol mixture forming a new azeotrope, glycerin and water. Because glycerin and water are more polar than alcohol. Therefore the volatility of ethyl alcohol enhances. When distilling, ethyl alcohol distills freely leaving water and glycerin in the still. This process continues until all alcohol is completely distilled at a temperature of 78.3 degree centigrade. Therefore practically, absolute alcohol can be obtained from azeotropic distillation.

When benzene is added to ethyl -alcohol mixture, the reverse phenomenon is observed. The new azeotrope is benzene-alcohol,

In Extractive distillation, the third substance added to the azeotropic mixture is relatively non volatile liquid compare to the components to be separated.

The third component is withdrawn at the base of the fractionating column.

Example: Separation of toluene from paraffin hydrocarbons of approximately same molecular weights. The separation of toluene and iso octane (Example for hydrocarbon) is difficult. In the presence of phenol, the relative volatility of iso octane increases, therefore separation of toluene is relatively easy. In another example, furfural is added for the separation of butadiene from its mixture containing butane and butane.

Applications

In the preparation of absolute alcohol and in petroleum industries.

Molecular Distillation

Molecular distillation is defined as a distillation process in which each molecule in the vapour phase travels mean free path and gets condensed individually without intermolecular collisions on application of vacuum. known also as evaporative distillation or short path distillation.

Principle

The substances to be distilled have every low vapour pressures. Ex: Oils, greases, waxy materials and high molecular substances. They boil at very high temperatures. In order to reduce the boiling point, vacuum can be applied.

Applications

Molecular distillation is used for the purification and separation of low vapour pressure chemicals.

i. It is used in the purification of chemicals like tri cresyl phosphate, dibutyl and di methyl phthalates.
ii. In the refining of fixed oils
iii. Vitamin A and E from fish liver oil and other vegetable sources
iv. In the distillation of steroids

Theory

- The mean free path of a molecule is defined as the average distance through which a molecule can move without coming into collision with another.
- For material that are regarded as non-volatile under ordinary conditions of temperature and pressure are generally removed by increasing the mean free path.

Mean free path

The mean free path of a molecule can be defined as the average distance through which a molecule can move without coming into collision with each other.

It can be expressed mathematically (λ) as:

$$\lambda = \eta\sqrt{3}/p\rho$$

Where, p = vapour pressure

ρ = density,kg/m3

η = viscosity, Pa.s

λ = mean path length,m

The characteristics of the substance influence the method of distillation include:

i. Liquids having low viscosity and density possess long mean path. Distillation is simple.
ii. Substances having high pressures possess low mean free path.

Based on the method of formation, apparatus may be divided into two types.

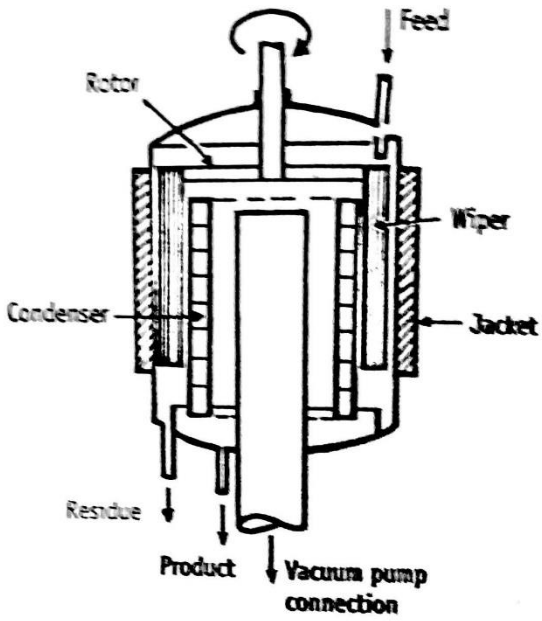
Falling film molecular still or wiped film molecular still

Principle

In this still, vaporization occurs from a film of liquid flowing down a heated surface under high vacuum. The molecules(vapour) travels a short distance and strikes the condenser nearby. The distillate is subsequently collected.

Construction

1. The vessel is having a diameter of 1 meter.
2. The walls of the container are jacketed.
3. Wipers with rotors are fitted adjacent to the vessel wall.
4. Vacuum pump is connected to a large diameter pipe in the center of the vessel.
5. There are outlets for collecting the distillate and providing the feed.



Wiped film molecular still

Working

1. The vessel is heated by suitable means.
2. Vacuum is applied at the center of the vessel and wipers are allowed to rotate.
3. The liquid will form a film by the PTFE (Poly tetra fluro ethylene) wipers.
4. Since the surface is heated, the film evaporates instantly.
5. The vapour travels its mean free path and strikes the condenser.
6. The condensate is collected into a vessel.
7. The residue is collected from the bottom of the vessel and it is recirculated for further distillation.

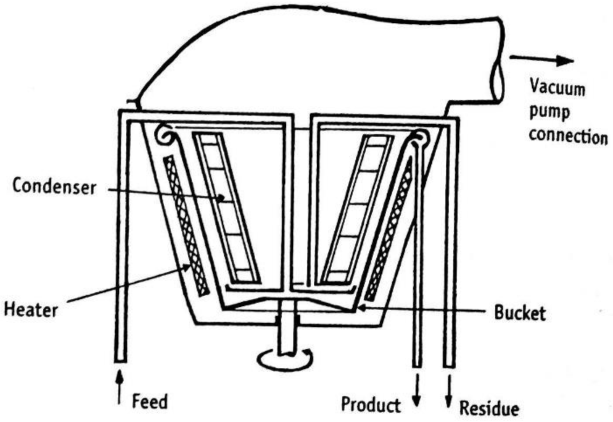
Centrifugal Molecular Still

Principle

In this instrument, liquid feed is introduced into a vessel. Which is rotated at high speed (centrifugal action). Upon heating, vaporistaion occurs from a film of liquid on the sides of the vessel. The vapour molecules travels a short distance and get condensed on the adjacent condenser. Each molecule is condensed individually. The distillate is subsequently collected.

Construction

1. It consists of a bucket shaped vessel having 1 m to 1.5 m diameter.
2. It is rotated at high speed using a motor.
3. Heaters are fixed externally to heat the fluid in the bucket.
4. Condensers are closely arranged to the evaporating surface.
5. Vacuum pump is connected to the entire vessel from the top.
6. There are provisions for collecting the product and recirculation of the residue.



Centrifugal molecular still

Working

1. Vacuum is applied at the center of the vessel
2. The bucket shaped vessel is rotated at high velocity.
3. The feed is introduced from the center of the vessel.
4. The liquid moves outward over the surface of the vessel to form a film, due to centrifugal action.
5. Since the radiant heaters heat the surface, the liquid evaporates directly from the film.
6. The vapour travels its mean free path and strikes the condenser.
7. The residue is collected from the bottom of the vessel and it is recirculated for further distillation.

Disadvantages

Construction and operation are more complicated compared to falling film molecular still.

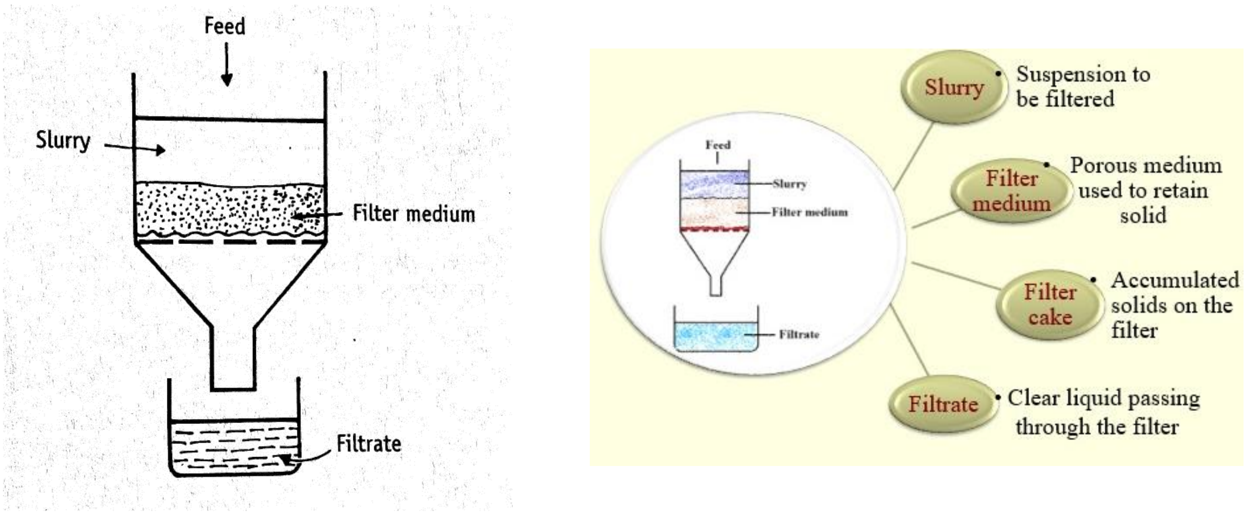
FILTRATION

Filtration can be defined as the process of separation of solids from a fluid by passing the same through a porous medium that remains the solids, but allows the fluid to pass through.

Clarification

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

Process of Filtration



The process of filtration

1. The pores in the filter medium are smaller than the size of the particles to be separated.
2. Filter medium -Filter paper or cloth-is placed on a support (Sieve)
3. When slurry is passed through the falter medium, the fluid flows through the filter medium by virtue of a pressure difference across the filter.
4. Gravity is acting on the liquid column.
5. Solids are trapped on the surface of the filter medium.
6. Once the preliminary layer of particles is deposited, the filter medium serves only as a support. The filter will work only after an initial deposit. After a particular time, the resistance offered by the filter cake is high that virtually filtration is stopped
7. For this reason, a positive pressure is applied on the filter cake(Upstream)or negative pressure(suction) below the filter medium (downstream)

Applications of Filtration in pharmacy

1. In the production of sterile products: Air is filtered through HEPA filters or laminar air flow bench to obtain sterile air.
2. In bacteria proof filters to obtain sterile solutions, particularly when heat sterilization is not suitable on account of thermo labile nature of the contents- Particles as small as 0.2 micrometer should be removed.

3. In areas of antibiotic production, recombinant technologies of biological products and vaccines.
4. In the production of bulk drugs
5. In the production of liquid oral formulations
6. De waxing of oils
7. Removing suspended oil from water-Aromatic waters, syrups, elixirs, eye drops etc.
8. Removal of un desired solids from honey, fruit juices
9. Clarifying the potable water.
10. In affluent and waste water treatment

Mechanisms of filtration

Filtration can happen due to any one or combination of the following process.

1. Straining
2. Impingement
3. Entanglement
4. Attractive forces

Straining

Particles of large size cannot pass through the smaller pore size of the filter medium

Impingement

Solids having the momentum move along the path of streamline flow and strike the filter medium. Thus the solids are retained on the filter medium.

Entanglement

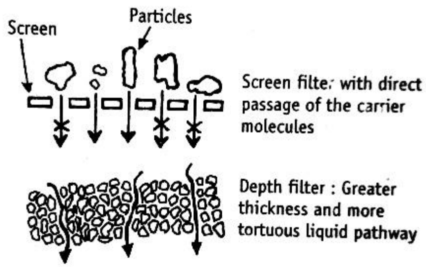
Particle entangles in the mass of fibres of filter medium due to smaller size particles than the pore size.

Attractive forces

Particles retained on the filter medium due to the attractive forces between the particles and filter medium.

Types of filtration

1. ***Surface (Screen) filtration***-By screening action the pores or holes of the medium block the passage of solids. Straining and impingements are responsible for surface filtration.
2. ***Depth filtration*** -Here slurry penetrates to a point where the diameter of the solid particles is greater than that of the channel. Entanglement is the mechanism here. Solids are retained with a gradient density structure or physical restriction or adsorption properties of the medium.



Types of filtration

Differences between surface and depth filtration

Surface filtration	Depth filtration
The size of the particles retained is slightly higher than the mean pore size of the filter medium	The size of the particles retained is much smaller than the mean pore size of the filter medium
Mechanical strength of the filter medium is less	Mechanical strength of the filter medium is high
Low capacity	High capacity
Equipment is expensive since it may need ancillary equipments.	Cheaper without ancillary equipments
Examples: Cellulose Membrane filters	Examples: Ceramic filters, Sintered bed filters

Theories of filtration

In the process of filtration, the fluid passes through the filter medium (upstream and down stream), which offers certain resistance to its passage. Pressure difference across the filter is the driving force for the operation. Therefore,

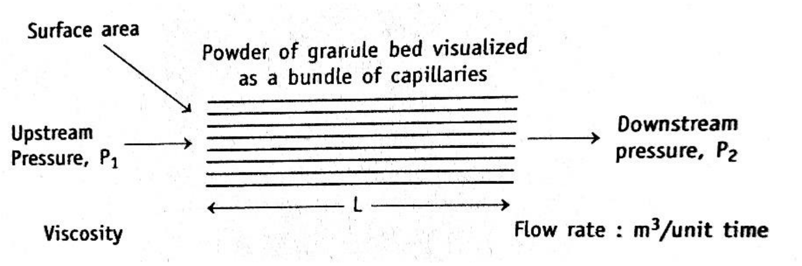
Rate = Driving force/Resistance

Since the resistance increases with the deposition of solids, the filtration process is not a steady one. The rate of flow will be greatest in the beginning since the resistance will be low. Once the filter cake is formed, its surface act as a filter medium and solids continuously deposit, adding to the thickness of the cake. The resistance to flow is related to several factors as mentioned below.

Resistance to movement = Pressure up stream-Pressure downstream

Length of capillaries

Capillary *means* a tube with an internal diameter of hair-like thinness



Poiseuille's equation

Poiseuille considered that filtration is similar to the streamline flow of a liquid under pressure through capillaries. Poiseuilles equation is

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

V = Rate of flow in unit time m^3/s

ΔP= Pressure difference across the filter bed,Pa

r = Radius of the capillary in the filter bed ,m

L = Thickness of the filter cake(capillary length),m

η = Viscosity of the filtrate Pa.s *Pa-pascal*

Darcy's equation

The factors influencing the rate of filtration has been into an equation by Darcy, which is

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

Where K = is the permeability constant of the cake m2 , A= Surface area of the porous bed (filter medium),$m^2$and other items are same as that of the previous equation.

Permeability

May be defined as the flow of rate of a liquid of unit viscosity across a unit area of cake having unit thickens under a pressure gradient.

Kozeny-carman equation

Poiseulli's equation is applicable to porous bed, based on capillary type of structure by includingadditional parameters.

Thus the resultant equation, which is widely used for filtration is Kozeny-Carman equation.

$V= A/\eta S^2 \text{ x } \Delta P/KL \text{ x } \varepsilon^3 /(l-\varepsilon^3)$

e- Porosity of the cake

S- Specific surface area of the particles comprising the cake

K-Kozeny constant

Factors influencing filtration

1. Properties of Liquid- Density, viscosity, corrosiveness

2. Properties of solids- Particle shape, particle size, particle charge, density, particle size distribution, rigidity or compressibility of solids.

3. Proportion solids in the slurry-Rate at which the filter cake is formed, especially in the early stages of filtration.

4. Temperature of suspension to be filtered.

Surface area of filter medium

According to Kozeny-Carman equation, the rate of flow of filtrate through the filter is inversely

proportional to the specific surface of filter bed OR the rate of filtration is directly proportional to the surface area of filter medium. Hence the rate can be increased either using a larger filter or connecting a number of small units in parallel. Pleating the filter paper or using a pleated funnel increases the effective surface area of filtration

Pressure drop across the filter medium

The rate of filtration is directly proportional to the overall pressure drop across the filter medium and filter cake. The pressure drop can be achieved in a number of ways as follows.

1. Gravity

By maintaining a head of slurry above the filter medium, a pressure difference can be achieved.

A head of 10 meters of water can create a pressure difference of 100 kilo Pascal.

2. Applying pressure

By applying a pressure on the surface of the slurry by pumping the slurry on to the filter medium. The pressure difference obtained by this method will be greater than the pressure achieved by reduced pressure. To prevent clogging, the pressure should be less at the starting of filtration process.

3. Reducing pressure

The pressure underneath the filter medium may be reduced below atmospheric pressure by

connecting the filtrate receiver to a vacuum pump. This factor has limited applications only

4. Centrifugal force

Centrifugal force could replace the gravitational force and is used to increase the rate of filtration. (Refer Centrifugation)

Viscosity of filtrate

The rate of filtration is inversely proportional to the viscosity of the fluid. The viscosity of fluid is important than that of the slurry.

Increasing the temperature may decrease viscosity and increase rate of filtration. But for thermo labile and volatile substances, this is not possible.

Surface area and Pressure difference are the two factors which are important in industry

practice. Porosity of medium also can influence the rate of filtration.

Filter media

The filter medium acts as a mechanical support for the filter cake and are also responsible for

the collection of fluids

Ideal characters of filter media

1. It should have Sufficient mechanical strength

2. Must be inert

3. it should not absorb the dissolved material

It should allow maximum passage of liquids, while retaining the solids-That means it should

offer low resistance to flow.

The magnitude of the resistance of the filter medium will change due to the layers of solids deposited earlier, which may block the pores or may form bridges over the entrance of the channels. On continued filtration, the thickness of the cake further builds and hence the rate of filtration decreases. When the rate is un economical, filtration is stopped. The filter cake is removed and filtration is restarted.

Materials used as filter media

1. **Woven materials**

Made up of wool, silk, metal or synthetic fibres (rayon, nylon etc.). These include

a- Wire screening –Ex. stainless steel is durable, resistance to plugging and easily cleaned.
b- Fabrics of cotton, wool, nylon. - Cotton is a common filter, however, Nylon is superior for pharmaceutical use, since it is unaffected by mold, fungus or bacteria and has negligible absorption properties. The choice of fibre depends on chemical

2. **Perforated Sheet metals**

Stainless steel plates have pores which act as channels as in case of meta filters.

3. **Bed of granular solid built up on supporting medium**

In some processes, a bed of graded solids may be formed to reduce resistance of flow.

Ex. Of granular solids are gravel, sand, asbestos, paper pulp and keiselgur.

Choice of solids depends on size of solids in process.

4. **Prefabricated porous solid units**

Used for its convenience and effectiveness.

Sintered glass, sintered metal, earthenware and porous plastics are used for fabrication.

5. **Membrane filter media**

These are cartridge units and are economical and available in pore size of 100 μm to even less than 0.2 μm. Can be either surface cartridges or depth type cartridges.

Surface cartridges

These are corrugated and resin treated papers and used in hydraulic lines. Ex. Ceramic cartridges and porcelain filter candles. Can be reused after cleaning.

Depth type cartridges

Made up of cotton, asbestos or cellulose.

These are disposable items, since cleaning is not feasible

Filter Aids

Filter aids are finely divided solid materials which can increase the rate of filtration when added to the slurry. They are capable of plugging o supporting medium. Various filter aids are used in pharmaceutical field. The most important filter aid widely used is diatomaceous earth or kiesulgar. It consists of siliceous substances of very small marine organisms known as diatoms. Perlite, is an aluminium silicate. Cellulose, Asbestos, charcoal, talc, bentonite, fullers earth etc are examples of filter aids.

Certain degree of resistance is offered by every filter medium during the process of filtration. This resistance increases when the layers of solids build up on the medium. The cake may become impervious (not allowing fluid to pass through) by blocking of the pores in the medium. Flow rate is inversely proportional to the resistance of the solid cake. Filter aids prevent the medium from becoming blocked and they form an open, porous cake, hence reducing the resistance to flow. In this experiment, the effect of filter aid on the filtration of calcium carbonate suspension is studied.

Employment of filter aids

1. A pre coat is formed over the medium. For this purpose, a suspension of the filter aid is filtered to obtain a coating over the medium.
2. A small portion of filter aid is added to the slurry (body mix technique). So the filter cake has a porous structure and filtration can be efficient. Then slurry is re circulated through the filter until a clear filtrate is obtained.

Filtration Equipments

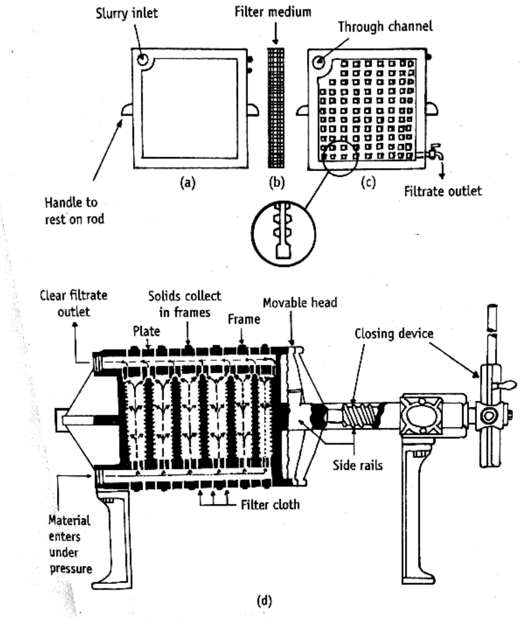
Classification Based On Application of External Forces

1. Pressure Filters-Plate & frame filter press
2. Vacuum filters-filter leaf
3. Centrifugal filters (ref. centrifugation)

Plate and frame filter press

Principle

1. Surface filtration is the basic principle
2. The slurry enters the frame by pressure and flow through the filter medium
3. The filtrate is collected on the plates and sends to the outlet.
4. Number of plates is used so that surface area increases and consequently large volumes of slurry can be processed simultaneously with or without washing.



(a) Frame—Maintains the slurry reservoir, inlet (eye) for slurry.
(b) Filter medium.
(c) Plate along with section—supports the filter medium, receiving the filtrate and outlet (eye).
(d) Assembly of plate and frame filter press.

Filter Press

Construction

1. The filter press is made up of two types of units, Plates and Frames (of different thickness), made of aluminium alloy
2. Frame consists of an open space inside where in the slurry reservoir is maintained for filtration and an inlet to receive the slurry. (2dots in figure)
3. The plate has a studded or grooved surface to support the filter cloth and an outlet. (l dot in figure)
4. The filter medium, usually a cloth is placed between the plate and frame.
5. Plate, filter medium and frame are arranged in the sequence 1.2.1.2.1
6. A number of plates and frames can be employed to increase surface area and thereby rate of filtration.

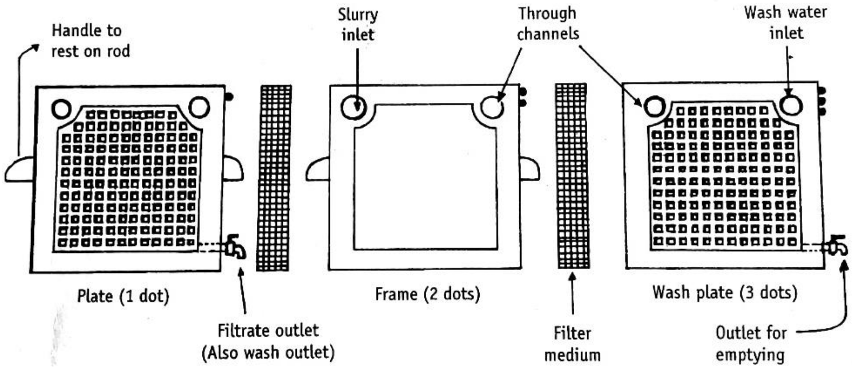
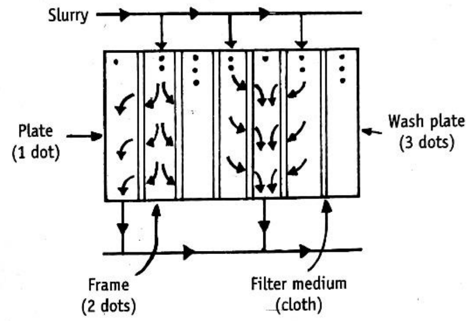
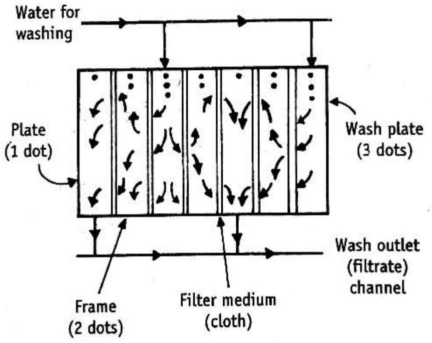


Plate and frame filter press with water wash facility



(a) Principles of filtration operation using the three types of plate and frame.



(b) Principles of cake washing using three types of plate and frame.

Working-Filtration and Washing

1. Slurry enters the frame marked 2 dots from the feed channel and passes through the filter medium on to the surface of the plate marked by1 dot.

2. The solids from the filter cake remain in the frame.

3. Two filter cakes are formed which eventually meets at the centre of the frame.

4. The filtrate drains between the projections on the surface of the plate and escapes from the out let.

5. As filtration proceeds, the rate of filtration decreases due to the resistance of the cake.

6. The process should be stopped at this point and filter press can be emptied and restarted

Washing

1. The sequence of arrangements of plates and frames can be represented by dots as 1.2.3.2.1.2.3.2.1.2.3.2.1 and so on
2. To wash the filter cake, the outlets of washing plates are closed (3 dots)
3. Wash water is pumped into the washing channel
4. Water passes through the filter cloth and enters the frame (2 dots) which contains the cake.Then water washes the cake, passes through the filter cloth and enters the plate (1 dot) down the surface.

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

Uses

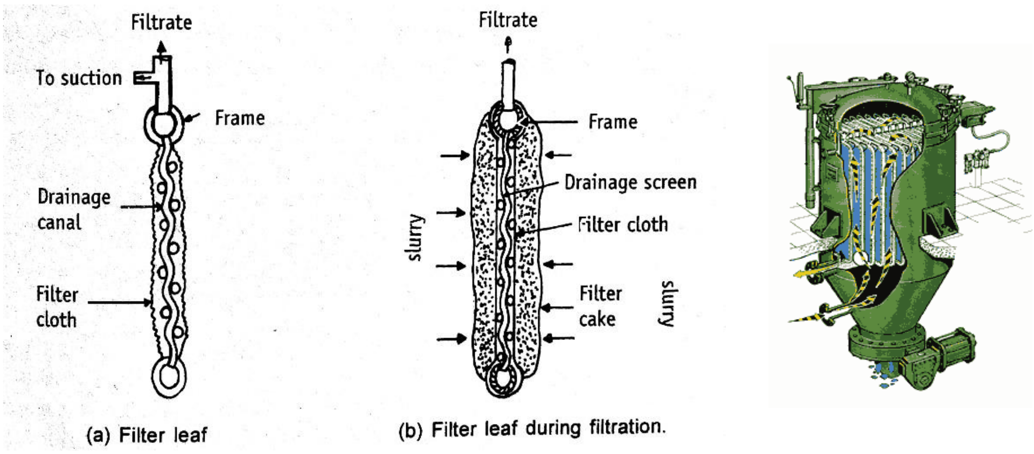
1. In the collection and processing of anti-toxins
2. In the removal of precipitated proteins from insulin.
3. In the removal of cell broth from fermentation medium

Leaf filter

Principle and construction

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

It consists of a narrow frame enclosing a drainage screen or grooved plate. The frame may be of any shape, circular, square or rectangular. The whole unit is covered with filter cloth. The outlet for the filtrate connects to the interior of the frame through suction.



(a) Filter leaf (b) Filter leaf during filtration.

Leaf filter

Working

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

Uses

1. A number of units can be connected in parallel to increase the surface area for filtration.
2. Pressure difference can be obtained using vacuum or using pressure up to 800 kilo Pascal.
3. Labour costs are low
4. Washing efficiency is high
5. Slurry can be filtered from any vessel. Simply immersing the filter in a vessel of water can wash the cake.

Variants of Leaf Filter: *Sweet Land Filter*: Here a number of leaves are connected to a common outlet to provide a large area for filtration

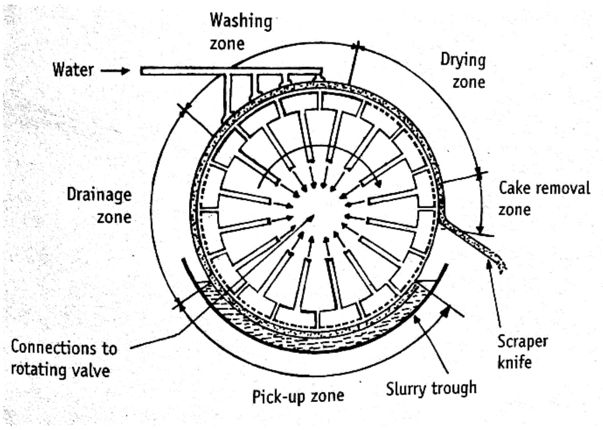
Rotary Drum filter/Drum filter

Principle

Filtering the slurry through sieve like mechanism on a rotating drum surface, under vacuum. Compression, drying and removal of cake is also possible.

Construction

Consists of a metal cylinder mounted horizontally. The drum may be up to 3 meters in diameter 3.5 meters in length with a surface area of 20 m square. The curved surface is a perforated plate on which a filter cloth is attached.



Drum filter

Working

1. The drum is rotated at NLT 1RPM

2. The slurry just enters the trough

3. As it dips, vacuum is applied in this segment so that the solid is build up on the surface.

4. The liquid passes through the filter cloth into an internal pipe and valve.

5. Finally the filtrate reaches the collecting tank

6. As the drum, leaves the slurry zone, it enters the drainage zone.

7. Excess of the liquid is drawn inside.

8. Cakes can be compressed using special cake compressors t this stage, which improve the efficiency of washing and drying process

9. Vacuum is applied to carry the slurry along with drum

10. Drainage zone

11. Water washing arrangement

12. Drying zone-Dry air supply

13. Cake removal zone

14. As the drum enters the drainage zone, it enters the water wash section. Water is sprayed on the cake. A separate system of vacuum is applied on the panel in order to suck the wash liquid and air through the cake of solids and liquid is drawn through the filter into a separate collecting tank.

15. Then the cake enters the drying zone, where hot air is blown on the cake. Finally, the cake is removed using necessary equipment. All these steps are completed in one rotation of the drum.

Uses

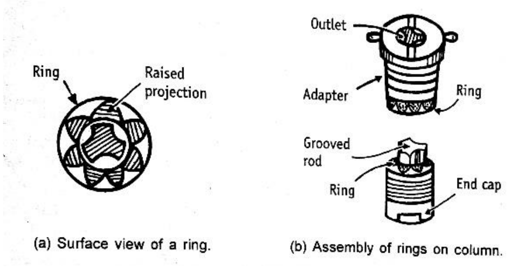
1. Continuous operation and is utilized to filter slurries containing high proportion of solids up to 15 to 30 percent.
2. In the production of penicillin, the extract is separated from broth cells.

In the separation of calcium carbonate, starch and magnesium carbonate

Meta filter

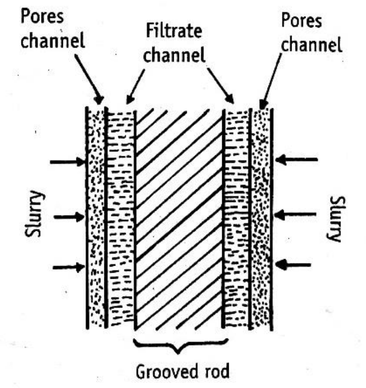
Principle & Construction

1. It is a strainer type of filter (surface filtration)
2. Metal rings containing semi-circular projections which are arranged as a nest to form channels on the edges.
3. This channel offers resistance to the flow of solids
4. The clear liquid is collected into a receiver from the top.



(a) Surface view of a ring. (b) Assembly of rings on column.

Meta filter



Advantages

i. Simple construction
ii. Provides large filtering area in a relatively small floor size.
iii. Pressure up to 200 kilo Pascal can be used
iv. Simple operation

Disadvantages

1. Consumption of time due to batch process
2. Expensive
3. Usually used for slurries containing less than 5% solids.

Working

a. These filters can be placed in a vessel

b. Pressure can be provided to pump the slurry, occasionally by the application of reduced pressure to the outlet side

c. The slurry passes through the channels formed on the edges between the rings.

d. The clear liquid rises up and collected from the outlet into the receiver.

Uses

1. Clarification of syrups
2. Filtration of injection solutions
3. Clarification of insulin liquors
4. Filtration of viscous liquids can be achieved by applying pressure

Advantages

1. Can be used in high pressures
2. Running costs are low
3. Versatile in nature-can be used for the filtration of very fine particles using filter aids
4. Removal of cake is easy
5. Sterile products can be handled.

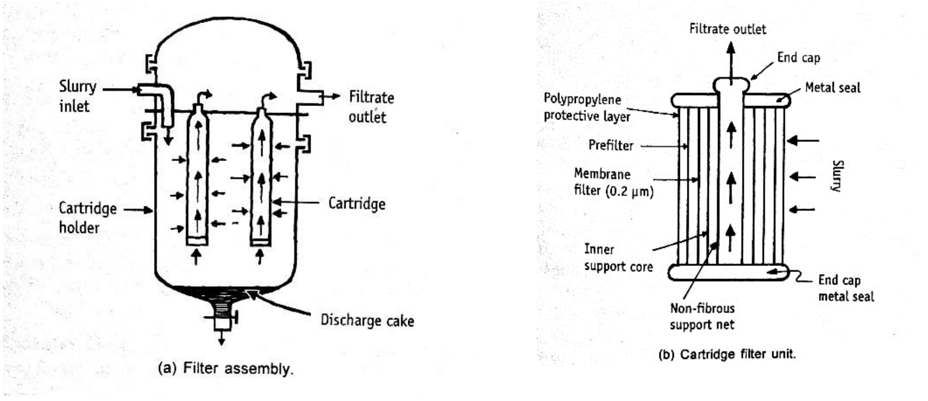
Cartridge filter

Principle

It's a thin porous membrane in which a pre filter and membrane filter are combined into single unit.

Construction

It has a cylindrical configuration made with a disposable or changeable filter media. These are made of plastic and metal. It consists of two sieves like membrane filters made of poly propylene: a prefilter and an actual filter for filtration. A protective layer surrounds them. The cartridges are housed in a holder. A number of cartridges can be placed in the same housing. The housing is closed with a lid. The housing has provisions for slurry inlet and filtrate outlet



(a) Filter assembly.

(b) Cartridge filter unit.

Cartridge filter

Working

The slurry is pumped into the cartridge holder. It passes through cartridge filter unit by the mechanism of straining. The clear liquid passes to the centre and moves up to collect through the outlet

Uses

Cartridge filter is useful in the preparation of particle free solution for parenteral and ophthalmic uses. The filter units can hold up to 1000 to 15000 liters of sterile solution per hour.

Advantages

1. SS construction permits autoclaving for sterile operations
2. Cartridges with self-cleaning devices are available
3. Assembly as well as dismantling is simple

Disadvantages

1. Cost of disposable elements
2. Components made by different suppliers are not inter changeable

Membrane filters

A membrane is a thin layer of semi-permeable material that separates substances when a driving force is applied across the membrane. Membrane processes are increasingly used for removal of bacteria, microorganisms, particulates, and natural organic material, which can impart colour, tastes, and odors to water and react with disinfectants to form disinfection by-products. As advancements are made in membrane production and module design, capital and operating costs continue to decline. The membrane processes discussed here are microfiltration (MF), ultrafiltration (UF), Nano filtration (NF), and reverse osmosis (RO).

So a membrane most commonly is a thin film of material with selective permeability. Membrane-based separation processes make use of this selective permeability. Membrane filters are supported by a perforated metal, plastic or coarse sintered glass.

The first commercial membranes were made of cellulose acetate. Temperatures had to be maintained below 30C and the pH range was 3–6. These constraints limited their use, as they could not be disinfected by heat or cleaned with acid or alkali detergents.

considerable number of different membrane processes have found industrial applications.

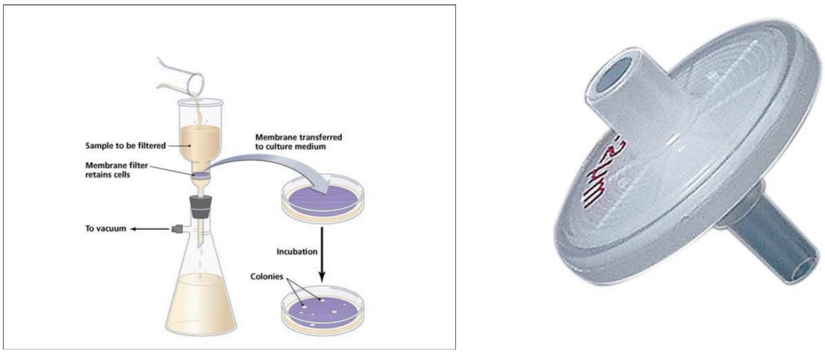
We shall discuss about four main types of membrane filtration

microfiltration (MF), ultra-filtration (UF), Nano filtration (NF) and reverse osmosis (RO).

The driving force for material transport through the membrane in those four processes is a pressure difference. These processes are therefore called pressure-driven membrane processes.

Some other Types of Membrane filtration: Electro dialysis, in which the driving force is an electric field. Pervaporation, a separation technique based on vaporization through perm-selective membranes used in distillation.

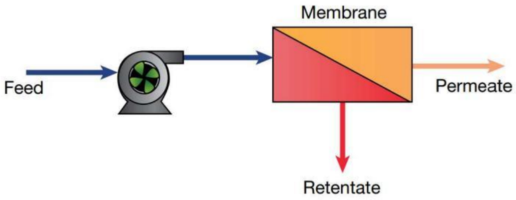
Several grade membrane filters are available with pore size ranging from 0.010 plus or minus 0.002 microns to 5.0 plus or minus 1.2 microns. Millipore filter corporation assigned the codes VF and SM for these two extreme ranges.



Membrane filters

Working

1. In membrane filtration, a portion of fluid known as permeate (filtrate) passes through the membrane, while other constituents are rejected by the membrane and retained in the retentate (concentrate) steam



Uses

1. In the filtration of sterile preparations like injections, eye, ear drops etc.

Advantages

1.They are usually continuous

2.Comparatively low energy utilisation

3. No phase change of contaminants

4. Small temperature change

6 Minimum of moving parts, disposable, negligible adsorption

7.Physical separation of contaminants

8.The whole unit can be sterilized.

Disadvantages

1. High cost.
2. Clogging may happen

Seitz Filter

These filters are originally developed in Germany. It consists of perforated discs and asbestos sheet which is made up of asbestos fibres but may also contain cellulose and alkaline earth metals. Due to fibrous nature of asbestos pads it may shed fiber into filtrate. Hence a few ml of filtrate should always be rejected. Negative, positive pad pressure filters and large scale fibrous pad filters are available.

Seitz filter

Working

1. Seitz pads are used for filtration, which are made of highly shredded asbestos.
2. A few pads must be used for each filtration to avoid contamination from previous filtration residues.
3. They are fragile and hence to be used on a perforated support.
4. They work on the basis of surface charging and electrostatic attraction.

Uses

1. In air filtration
2. Efficient in removing bacteria from fluids

Advantages

1. Pads are inexpensive and filtration is rapid
2. Better than ceramic and sintered glass filters for viscous solutions

Disadvantages

1. Removal of bacteria depends upon the pads taking up water and swelling thus reduces interstitial spaces in the filter. Hence fibrous pads are not suitable for sterilizing some of the products like alcohol or oil.

CENTRIFUGATION

It is a unit operation employed for separating the constituents present in dispersion with the help of centrifugal force. Centrifugal force replaces gravitational force in sedimentation.

USES

1. When ordinary filtration is difficult- Ex: Viscous mixtures and colloidal dispersions
2. Separation of two immiscible liquids or a solid from a liquid
3. The equipment used for centrifugation is called as *Centrifuge*
4. The speed of centrifuge is commonly expressed in terms of *Revolutions Per Minute(RPM)*

Pharmaceutical Applications

i. Production of bulk drugs –Separation of crystalline drugs such as aspirin from mother liquor

ii. Production of biological products: Insulin can be obtained by selectively precipitating other fractions of proteins during its manufacture. Centrifugation is employed for separating the blood cells from blood

III. Biopharmaceutical analysis of drugs: Drugs present in blood, tissue fluids and urine etc.are separated using centrifugation

IV. Evaluation of suspensions and Emulsions- A stable emulsion should not show any signs of separation even after centrifuging at 2000-3000 RPM at room temperature.

V. In the determination of mol.wt of colloids

Theory Of Centrifugation

In colloidal dispersion, the dispersed phase may be either solid or liquid. Particles having a size above 5 micrometer sediment at the bottom due to gravitational force. In such cases, separation of solids can be done by simple filtration. If particles are of the order of 5 micro meter or less, they undergo Brownian motion. Hence they do not sediment under gravity. Therefore, centrifugal force can be applied.

Centrifugal Effect Or Relative Centrifugal Force (Rcf)

Consider a body of mass m kg rotating in a circular path of radius r meter at a velocity of v meter per minute. The force acting on the body in a radial direction is given by,

Force acting in radial direction, $F = mv2/r$

Where F is equal to centrifugal force N

The same body is experiencing gravitational force (Newton).It can be expressed as

Gravitation force G = mg, where g = Acceleration due to gravity, m/s2

- The centrifugal effect is expressed as a ratio of centrifugal force to gravitational force.
- Separating power of a centrifuge is expressed as ratio of centrifugal force to gravitation acceleration.

The centrifugal effect can be expressed as:

Centrifugal effect = Force acting in radial direction/Gravitational force

= ***F/G***

Classification of centrifuges

1. Sedimentation Centrifuge
2.Ultra Centrifugation
3.Filtration centrifuge

1.Sedimentation Centrifuge

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

2. Ultra Centrifugation

When extremely fine solid matter is to be separated from liquid for example in colloidal research, ultra centrifuge is used -100000RPM

3.Filtration centrifuge

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

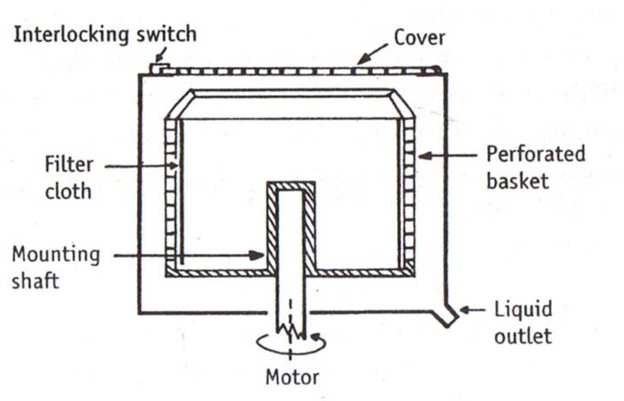
Equipments

i. Perforated basket centrifuge
ii. Non-Perforated basket centrifuge
iii. Super Centrifuge
iv. Conical Disc Centrifuge

Perforated Basket Centrifuge

Principle

It is a filtration centrifuge. A basket is mounted above a driving shaft. The separation is through a perforated wall based on the difference in densities of solid and liquid phases. The bowl contains a perforated side wall. During centrifugation the liquid phase passes through the perforations while solid are retained in the bowl.



Perforated basket centrifuge

During centrifugation, the liquid passes through the perforated wall, while the solid phase retains in the basket. The liquid leaves the basket and collected in the outlet. The cake is then spun to dry as much as possible. After a definite period of time, the power is turned off and the solids are collected.

Uses

In the

- Separation of crystalline drugs like ASPIRIN during its manufacture
- Separation of precipitated proteins from **INSULIN**
- Separation of sugar crystals

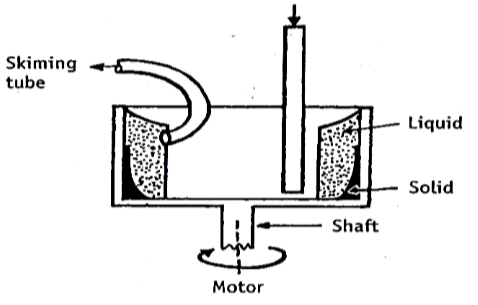
Non-perforated basket centrifuge

Principle and construction

This is a sedimentation centrifuge

The separation is based on the difference in the densities of solid and liquid phases

During centrifugation, solid phase is retained on the side of the basket; while liquid remain at the top which is removed by a skimming tube.



Construction of non-perforated basket cen

Pharma Vertical Bottom Discharge Centrifuge

Working

The suspension is fed continuously into the basket. During centrifugation, solid phase is retained on the sides of the basket while liquid remains at the top. The liquid is removed using a skimming tube. When sufficient depth of solids has been deposited on the walls of the basket, the operation is stopped. The solids are collected by a suitable method.

USES

It is used when the deposited solid contents offer high resistance to the flow of liquid

Super centrifuge

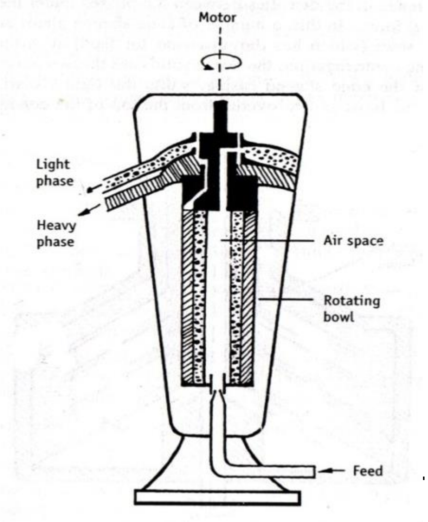
Principle and construction

It is a continuous centrifuge used for the separation of two immiscible liquid phases. It's also a sedimentation type centrifuge. The separation is based upon the densities of two immiscible liquids

During centrifugation the heavier liquid is thrown against the wall of the bowl while the lighter liquid remains as an inner layer. They are separated by modified weirs.

Working

The centrifuge is allowed to rotate on its longitudinal axis at a high frequency usually about 2000 rpm. The feed is introduced from the bottom of the centrifuge using a pressure system. During centrifugation, two liquid phases will be separated, based on the difference in densities.



Construction of supercentrifuge

Super centrifuge

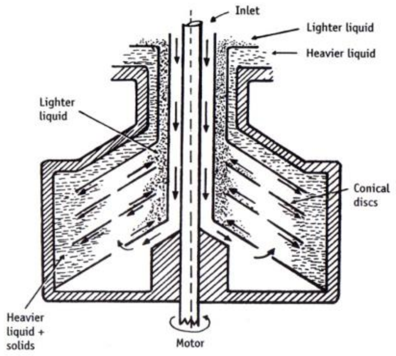
Uses

Separation of liquid phases in emulsions in food and pharmaceuticals

Conical disc centrifuge / de lavel clarifier

Principle and construction

- It is a sedimentation centrifuge. The separation is based on the difference in densities between the phases under the influence of centrifugal force.
- A number of cone shaped plates are attached to a central shaft at different elevations.
- During centrifugation, the solids are thrown outwards to the underside of the cone shaped casing, while the lighter clarified liquid passes over the bowl and recovered from the top of the cone.



Construction of the conical disc centrifuge

Working

Centrifuge is allowed to rotate. The feed is introduced through the concentric tube surrounding the shaft. The feed flows down and enters into the spaces between the discs. During centrifugation, the liquid flows into the channels (holes at the centre of the disc) and upward past the disc. These substances move along the surfaces of the discs to the limits of the inner and outer layers. The solids and heavy liquids are thrown outward and move underside of the disc.

Then the solids deposit at the bottom of the cone, which is removed intermittently. The lighter liquid moves to the upper side of the discs. The liquid raises to the top and escapes from the outlet.

Uses

1. In the manufacture of antibiotics
2. Suspensions containing low concentration of solids can be separated quickly
3. In the manufacture of insulin to remove precipitated proteins
4. In the separation of cream from milk
5. In rubber, oil, ink and beverage industries

Materials of Construction

1.Ferrous Metals & Alloys

Cast Iron

Stainless steel, Alloys

2.Non-Ferrous Metals

Nickel

Aluminium

Copper

Lead, Titanium, Tantalum, Alloys

3.Non Metals

1. Carbon
2. Graphite
3. Porcelain
4. Wood
5. Brick
6. Rubber
7. Elastomers
8. Glass
9. Plastics

Stainless steel

IRON + 12% to 30% Cr

0 to 2% Ni

Minor amounts of C, Columbium, Cu, Mb, Se, Tantalum and Titanium

There are about 70 types of SS.

Advantages

- ✓ Ease of fabrication
- ✓ Heat resistance
- ✓ Corrosion Resistance
- ✓ Ease of cleaning and sterilization
- ✓ Good tensile strength

Alloy

A metal made by combining two or more metallic elements, especially to give greater strength or resistance to corrosion.

Alloys of Stainless Steel

Composition	Advantages	Disadvantages	Uses
MARTENSITIC (Type 410) Chromium: 12-20% Carbon: 0.2 -0.4% Nickel : 2.0%	Resistant to mild corrosion, atmospheric and organic exposures	Poor ductility	Sinks,bench tops,storage tanks,buckets, mixing elements
FERRITIC (Type 430) Chromium: 15-30% Carbon: 0.1% Nickel : 0%	Better corrosion resistant, easy machine, resistant to oxidation and temperature (Up to 800degree)	Not good against reducing agents, hydrochloric acid	Tower linings,baffles, seperator,tower, heatexchanger, tubings, condensers,furnace parts, pump shafts, valve parts.

AUSTENTIC Chromium: 13-20% Carbon: 6 -22% Nickel : 0.1% - <0.25%	Highly corrosion resistant,readily cleaned, sterilizable,easy to weld, non magnetic	Not easy machine	Fermentors,Storage vessels,Evaorators, Extraction vessels, small apparutus (Funnels, buckets etc.)

Non-Ferrous Metals

Aluminium

- Aluminium is cheap
- Light weight
- Enough mechanical strength
- Easy fabrication
- Maintenance and cleaning easy

Advantages

1. High resistance to atmospheric conditions, industrial fumes, vapour and fresh and salt waters.
2. Aluminium can be used with conc. nitric acid and acetic acid
3. Thermal conductivity of aluminium is 60% more than that of pure copper

Disadvantages

1. Many mineral acids attack aluminium
2. Oxide and hydro oxide films are formed rapidly, when its surface is exposed.
3. Aluminium cannot be used with strong caustic solutions

Uses

In Food and Pharmaceutical industries

Heat transfer applications .Ex: Heat Exchangers

Storage containers

#A Number of aluminium alloys are available with improved qualities and mechanical properties

Non-metals-in organic

1. Glass

Glass is a Super cooled liquid, though it appear as a solid. The constituents are present in amorphous state.

Composition

Sand: Silicon di oxide- *Base Material*

Soda Ash: Sodium carbonate-*Improve the properties*

Calcium carbonate: -*Improve the properties*

Cullet: Broken Glass-*Fusion Agent*

Advantages

- Superior protective qualities, attractiveness and low cost
- Chemically inert
- Available in a variety of sizes, shapes and colours
- They are excellent barriers to almost everything except ***light***

Disadvantages

Fragility and Weight

Pharmaceutical glass containers have to comply with the official test for hydrolytic resistance as per I.P.The degree of attack by water on the container is determined by the amount of alkali released from the glass under specified conditions.

2.Plastics

They are synthetic materials made from a wide range of organic polymers such as poly- ehylene, PVC, Nylon etc

Advantages

- Completely resistant to most of the chemicals in common use
- Tough to withstand factory handling
- Metallic contamination can be avoided
- Excellent resistance to weak mineral acids

Disadvantage

- Poor resistance to high temperatures
- Use is limited to 450 ^{o} F

Classification of Plastics

- **Thermosetting Resins-** Permanently shaped into a rigid structure during manufacture

  Ex: Phenol plastics filled with asbestos, carbon, graphite etc.
- **Thermoplastic Resins-** Soften under the influence of heat but regain their rigidity when cooled Ex: Modern plastics

Thermoplastic resins

- Acryic resins Ex: Perspex
- Poly Vinyl Chloride(PVC)
- Polystyrene
- Polythene and poly propylene(PP)
- Nylons
- Polyester resins

- Epoxide resins
- Poly Tetra Fluro Ethylene (PTFE)- Known as **Teflon,Fluon .**This is stable even at 850 degree centigrade.

3.Rubber-Natural & Synthetic

- Natural Rubber (Soft Rubber)-Polymer of monomeric Isoprene.C_5H_8
- Hard Rubber- Soft Rubber+ Sulphur+Warm .This process is known as Vulcanisation

Synthetic Rubber

- Neoprine(Poly chloroprene)
- Nitrile rubber
- Butyl Rubber
- Silicon Rubber (Poly siloxanes)
- Poly Isoprene

CORROSION

Corrosion of metals is defined as the spontaneous destruction of metals in the course of their chemical, electrochemical or biochemical interactions with the environment. Thus, it is exactly the reverse of extraction of metals from ores.

Example: Rusting of iron A layer of reddish scale and powder of oxide (Fe_3O_4) is formed on the surface of A green film of basic carbonate [$CuCO_3 + Cu(OH)_2$] is formed on the iron metal. Surface of copper, when it is exposed to moist-air containing carbon dioxide.

Consequences (effects) of corrosion

The economic and social consequences of corrosion include,

i) Due to formation of corrosion product over the machinery, the efficiency of the machine gets failure leads to plant shut down.

ii) The products contamination or loss of products due to corrosion.

iii) The corroded equipment must be replaced

iv) Preventive maintenance like metallic coating or organic coating is required.

v) Corrosion releases the toxic products.

vi) Health (eg. from pollution due to a corrosion product or due to the escaping chemical from a corroded equipment) Corrosion-Oxidation.

Causes of corrosion

In nature, metals occur in two different forms.

1) Native State(2) Combined State

Native State: The metals exist as such in the earth crust then the metals are present in a native state. Native state means free or uncombined state. These metals are non-reactive in nature. They are noble metals which have very good corrosion resistance. Example: Au, Pt, Ag, etc.

Combined State: Except noble metals, all other metals are highly reactive in nature which

undergoes reaction with their environment to form stable compounds called ores and minerals. This is the combined state of metals. Example: Fe2O3, ZnO, PbS, CaCO3, etc.

Metallic Corrosion: The metals are extracted from their metallic compounds (ores). During the extraction, ores are reduced to their metallic states by applying energy in the form of various processes. In the pure metallic state, the metals are unstable as they are considered in excited state (higher energy state). Therefore as soon as the metals are extracted from their ores, the reverse process begins and form metallic compounds, which are thermodynamically stable (lower energy state). Hence, when metals are used in various forms, they are exposed to environment, the exposed metal surface begin to decay (conversion to more stable compound). This is the basic reason for metallic corrosion.

Although corroded metal is thermodynamically more stable than pure metal but due to corrosion, useful properties of a metal like malleability, ductility, hardness, luster and electrical conductivity are lost.

Classification or theories of corrosion

Based on the environment, corrosion is classified into

(i) Dry or Chemical Corrosion (ii) Wet or Electrochemical Corrosion

Dry or chemical corrosion

This type of corrosion is due to the direct chemical attack of metal surfaces by the atmospheric gases such as oxygen, halogen, hydrogen sulphide, sulphur dioxide, nitrogen or anhydrous inorganic liquid, etc. The chemical corrosion is defined as the direct chemical attack of metals by the atmospheric gases present in the environment.

Example: (i) Silver materials undergo chemical corrosion by Atmospheric H_2S gas.
(ii) Iron metal undergo chemical corrosion by HCl gas.

Types of dry or chemical corrosion

1.Corrosion by Oxygen or Oxidation corrosion
2.Corrosion by Hydrogen
3.Liquid Metal Corrosion

Corrosion by oxygen or oxidation corrosion

Oxidation Corrosion is brought about by the direct attack of oxygen at low or high temperature on metal surfaces in the absence of moisture. Alkali metals (Li, Na, K etc.,) and alkaline earth metals (Mg, Ca, Sn, etc.,) are rapidly oxidized at low temperature. At high temperature, almost all metals (except Ag, Au and Pt) are oxidized. The reactions of oxidation corrosion are as follows:

Mechanism

1)Oxidation takes place at the surface of the metal forming metal ions M2+

2)Oxygen is converted to oxide ion (O2-) due to the transfer of electrons from metal.

3)The overall reaction is of oxide ion reacts with the metal ions to form metal oxide film.

The Nature of the Oxide formed plays an important part in oxidation corrosion process.

Metal + Oxygen ^ Metal oxide (corrosion product)

When oxidation starts, a thin layer of oxide is formed on the metal surface and the nature of this film decides the further action. If the film

Wet or electrochemical corrosion

Electrochemical corrosion involves:

i) The formation of anodic and cathodic areas or parts in contact with each other

ii) Presence of a conducting medium

iii) Corrosion of anodic areas only and

iv) Formation of corrosion product somewhere between anodic and cathodic areas.This involves flow of electron-current between the anodic and cathodic areas.

At anodic area oxidation reaction takes place (liberation of free electron), so anodic metal is destroyed by either dissolving or assuming combined state (such as oxide, etc.). Hence corrosion always occurs at anodic areas.

At cathodic area, reduction reaction takes place (gain of electrons), usually cathode reactions do not affect the cathode, since most metals cannot be further reduced. So at cathodic part, dissolved constituents in the conducting medium accepts the electrons to form some ions like OH- and O_2.

Cathodic reaction consumes electrons with either by

(a) Evolution of hydrogen or

(b) Absorption of oxygen, depending on the nature of the corrosive environment

Difference between (dry) chemical and (wet) electrochemical corrosion:

S.No.	Chemical Corrosion	Electrochemical Corrosion
1.	It occurs in dry condition.	It occurs in the presence of moisture or electrolyte.
2.	It is due to the direct chemical attack of the metal by the environment.	It is due to the formation of a large number of anodic and cathodic areas.
3.	Even a homogeneous metal surface gets corroded.	Heterogeneous (bimetallic) surface alone gets corroded.
4.	Corrosion products accumulate at the place of corrosion	Corrosion occurs at the anode while the products are formed elsewhere.
5.	It is a self-controlled process.	It is a continuous process.
6.	It adopts adsorption mechanism.	It follows electrochemical reaction.
7.	Formation of mild scale on iron surface is an example.	Rusting of iron in moist atmosphere is an example.

Types of electrochemical corrosion

The electrochemical corrosion is classified into the following two types:

I. Galvanic (or Bimetallic) Corrosion
II. Differential aeration or concentration cell corrosion.

Galvanic Corrosion:

When two dissimilar metals (eg., zinc and copper) are electrically connected and exposed to an electrolyte, the metal higher in electrochemical series undergoes corrosion. In this process, the more active metal (with more negative electrode potential) acts as a anode while the less active metal (with less negative electrode potential) acts as cathode.

In the above example, zinc (higher in electrochemical series) forms the anode and is attacked and gets dissolved; whereas copper (lower in electrochemical series or more noble) acts as cathode.

Mechanism: In acidic solution, the corrosion occurs by the hydrogen evolution process; while in neutral or slightly alkaline solution, oxygen absorption occurs. The electron-current flows from the anode metal, zinc to the cathode metal, copper.

Zn_________ ^ Zn ^{2}+ + 2e" (Oxidation)

Thus it is evident that the corrosion occurs at the anode metal; while the cathodic part is protected from the attack.

Example: (i) Steel screws in a brass marine hardware (ii)Lead-antimony solder around copper wise; (iii) a steel propeller shaft in bronze bearing (iv Steel pipe connected to copper plumbing.

Factors influencing corrosion

There are two factors that influence the rate of corrosion. Hence a knowledge of these factors and the mechanism with which they affect the corrosion rate is essential because the rate of corrosion is different in different atmosphere.

1. Nature of the metal
2. Nature of the corroding environment

Nature of the metal

a) **Physical state:** The rate of corrosion is influenced by physical state of the metal (such as grain size, orientation of crystals, stress, etc). The smaller the grain size of the metal or alloy, the greater will be its solubility and hence greater will be its corrosion. Moreover, areas under stress, even in a pure metal, tend to be anodic and corrosion takes place at these areas.

b) **Purity of metal:** Impurities in a metal cause heterogeneity and form minute/tiny electrochemical cells (at the exposed parts), and the anodic parts get corroded. The cent percent pure metal will not undergo any type of corrosion. For example, the rate of corrosion of aluminium in hydrochloric acid with increase in the percentage impurity is noted.

c) **Over voltage:** The over voltage of a metal in a corrosive environment is inversely proportional to corrosion rate. For example, the over voltage of hydrogen is 0.7 v when zinc metal is placed in 1 M sulphuric acid and the rate of corrosion is low. When we add small amount of copper sulphate to dilute sulphuric acid, the hydrogen over voltage is reduced to 0.33 V. This results in the increased rate of corrosion of zinc metal.

d) **Nature of surface film:** In aerated atmosphere, practically all metals get covered with a thin surface film (thickness=a few angstroms) of metal oxide. The ratio of the volumes of the metal oxide to the metal is known as a specific volume ratio. Greater the specific volume ratio, lesser is the oxidation corrosion rate. The specific volume ratios of Ni, Cr and W are 1.6, 2.0 and 3.6 respectively. Consequently the rate of oxidation of tungsten is least, even at elevated temperatures..

e) **Relative areas of the anodic and cathodic parts:** When two dissimilar metals or alloys are in contact, the corrosion of the anodic part is directly proportional to the ratio of areas of the cathodic part and the anodic part.

Corrosion is more rapid and severe, and highly localized, if the anodic area is small (eg., a small steel pipe fitted in a large copper tank), because the current density at a smaller anodic area is much greater and the demand for electrons can be met by smaller anodic areas only by undergoing corrosion more brisky.

f) **Position in galvanic series**
g) **Passive character of metal**
h) **Solubility of corrosion products**
i) **Volatility of corrosion products**

<u>Nature of the Corroding Environment</u>

a) Temperature: The rate of corrosion is directly proportional to temperature ie., rise in temperature increases the rate of corrosion. This is because the rate of diffusion of ions increases with rise in temperature.
b) Humidity of air: The rate of corrosion will be more when the relative humidity of the environment is high. The moisture acts as a solvent for oxygen, carbon dioxide, sulphur dioxide etc. in the air to produce the electrolyte which is required for setting up a corrosion cell.
c) Presence of impurities in atmosphere: Atmosphere in industrial areas contains corrosive gases like CO_2, H_2S, SO_2 and fumes of HCl, H_2SO_4 etc. In presence of these gases, the acidity of the liquid adjacent to the metal surfaces increases and its electrical conductivity also increases, thereby the rate of corrosion increases.
d) Presence of suspended particles in atmosphere: In case of atmospheric corrosion: (i) if the suspended particles are chemically active in nature (like NaCl, Ammonium sulphate), they absorb moisture and act as strong electrolytes, thereby causing enhanced corrosion; (ii) if the suspended particles are chemically inactive in nature (eg., charcoal), they absorb both sulphur gases and moisture and slowly enhance corrosion rate.
e) Influence of pH: Generally acidic media (ie., pH<7) are more corrosive than alkaline and neutral media. However, amphoteric metals (like Al, Zd, Pb, etc.) dissolve in alkaline solutions as complex ions. The corrosion rate of iron in oxygen-free water is slow, until the pH is below 5. The corresponding corrosion rate in presence of oxygen is much higher. Consequently corrosion of metals, readily attacked by acid, can be reduced by increasing the pH of the attacking environment, eg., Zn (which is rapidly corroded, even in weakly acidic solutions such as carbonic acid suffers minimum corrosion at pH=11.
f) Nature of ions present:

g) Conductance of the corroding medium:
h) Formation of oxygen concentration cell:
i) Flow velocity of process stream:
j) Polarization of electrodes:

Corrosion control (protection against corrosion)

As the corrosion process is very harmful and losses incurred are tremendous, it becomes necessary to minimize or control corrosion of metals. Corrosion can be stopped completely only under ideal conditions. But the attainment of ideal conditions is not possible. However, it is possible only to minimize corrosion considerably. Since the types of corrosion are so numerous and the conditions under which corrosion occurs are so different, diverse methods are used to control corrosion. As the corrosion is a reaction between the metal or alloy and the environment, any method of corrosion control must be aimed at either modifying the metal or the environment.

a. Choice of metals and alloys:

1. The first choice is to use noble metals such as gold and platinum. They are most resistant to corrosion. As they are precious, they cannot be used for general purposes.
2. The next choice is to use purest possible metal. But in many cases, it is not possible to produce a metal of high chemical purity. Hence, even a trace amount of impurity leads to corrosion.
3. Thus, the next choice is the use of corrosion resistant alloys. Several corrosion resistant alloys have been developed for specific purposes and environment. For example, a) Stainless steel containing chromium produce an exceptionally coherent oxide film which protects the steel from further attack. (b) Cupro-nickel (70% Cu + 30%Ni) alloys are now used for condenser tubes and for bubble trays used in fractionating column in oil refineries. (c) Highly stressed Nimonic alloys (Ni-Cr-Mo alloys) used in gas turbines are very resistant to hot gases.

b. Proper Designing:

Proper geometrical design plays a vital role in the control of corrosion of equipments and structures. The general guidelines of the design of materials and components to control corrosion are the following:

a. Use always simple design and structure
b. The design must avoid more complicated shapes having more angles, edges, corners etc.
c. Avoid the contact of dissimilar metals as they may lead to galvanic type corrosion. To overcome this, insulation can be used.
d. When two dissimilar metals are to be in contact, the anodic area must be as large as possible and the cathodic area should be as small as possible.
e. As far as possible, crevices (gap or crack) should be avoided between adjacent parts of a structure.
f. Bolts and rivets should be replaced by proper welding

g. Metal washers should be replaced by rubber or plastic washers as they do not adsorb water. They also act as insulation.
h. Corrosion in pipelines can be prevented by using smooth bends.
i. Heat treatment like annealing minimizes the stress corrosion.

j. A good design of water storage container is the one from which water can be drained and cleaned easily. Such a design avoids accumulation of dirt etc.

Cathodic protection

The reduction or prevention of corrosion by making metallic structure as cathode in the electrolytic cell is called cathodic protection. Since there will not be any anodic area on the metal, corrosion does not occur. There are two methods of applying cathodic protection to metallic structures.

a) Sacrificial anodic protection (galvanic protection)
b) Impressed current cathodic protection

Sacrificial anodic protection method

In this method, the metallic structure to be protected is made cathode by connection it with more active metal (anodic metal). Hence, all the corrosion will concentrate only on the active metal. The parent structure is thus protected. The more active metal so employed is called sacrificial anode. The corroded sacrificial anode block is replaced by a fresh one. Metals commonly employed as sacrificial anodes are magnesium, zinc, aluminium and their alloys. Magnesium has the most negative potential and can provide highest current output and hence is widely used in high resistivity electrolytes like soil.

Applications

1. Protection as buried pipelines, underground cables from soil corrosion.
2. Protection from marine corrosion of cables, ship hulls, piers etc.
3. Insertion of magnesium sheets into the domestic water boilers to prevent the formation of rust.
4. Calcium metal is employed to minimize engine corrosion.

Advantages:

1. Low installation and operating cost.
2. Capacity to protect complex structures.
3. Applied to wide range of severe corrodents.

Limitations

1. High starting current is required.
2. Uncoated parts cannot be protected.
3. Limited driving potential, hence, not applicable for large objects.

Impressed Current Cathodic Protection Method

In this method, an impressed current is applied in opposite direction to nullify the corrosion current and convert the corroding metal from anode to cathode.

Usually the impressed current is derived from a direct current sources (like battery or rectifier on AC line) with an insoluble, inert anode (like graphite, scrap iron, stainless steel, platinum or high silica iron).

A sufficient DC current is applied to an inert anode, buried in the soil (or immersed in the corroding medium) and connected to the metallic structure to be protected. The anode is, usually, a back fill, composed of coke breeze or gypsum, so as to increase the electrical contact with the surrounding soil.

Impressed current cathodic protection has been applied to open water box coolers, water tanks, buried oil or water pipes, condensers, transmission line towers, marine piers, laid up ships etc.

This kind of protection technique is particularly useful for large structures for long term operations.

Modifying the environment-corrosion control

Environment plays a major role in the corrosion of metals. Hence, we can prevent corrosion to a great extent by modifying the environment. Some of the methods are,

i) Deaeration:

Fresh water contains dissolved oxygen. The presence of increased amount of oxygen is harmful and increases the corrosion rate. Deaeration involves the removal of dissolved oxygen by increase of temperature together with mechanical agitation. It also removes dissolved carbon dioxide in water

ii) By using inhibitors:

Inhibitors are organic or inorganic substances which decrease the rate of corrosion. Usually the inhibitors are added in small quantities to the corrosive medium. Inhibitors are classified into

1) Anodic inhibitors (chemical passivators)
2) Cathodic inhibitors (adsorption inhibitors)
3) Vapour phase inhibitors (volatile corrosion inhibitors)

Anodic Inhibitors

Inhibitors which retard the corrosion of metals by forming a sparingly soluble compound with a newly produced metal cations. This compound will then adsorb on the corroding metal surface forming a passive film or barrier. Anodic inhibitors are used to repair

a) the crack of the oxide film over the metal surface
b) the pitting corrosion
c) the porous oxide film formed on the metal surface.

Examples: Chromate, phosphate, tungstate, nitrate, molybdate etc.

Cathodic Inhibitors:

Depending on the nature of the cathodic reaction in an electrochemical corrosion, cathodic inhibitors are classified into,

a. **In an acidic solution:** the main cathodic reaction is the liberation of hydrogen gas, the corrosion can be controlled by slowing down the diffusion of H+ ions through the cathode. Eg., Amines, Mercaptans, Thiourea etc.

1. 2 H+ + 2 e$^-$ _ ^ H$_2$ **I**

b. **In a neutral solution:** in a neutral solution, the cathodic reaction is the adsorption of oxygen or formation of hydroxyl ions.

The corrosion is therefore controlled either by eliminating oxygen from the corroding medium or by retarding its diffusion to the cathodic area.

The dissolved oxygen can be eliminated by adding reducing agents like Na_2SO_3.

The diffusion of oxygen can be controlled by adding inhibitors like Mg, Zn or Ni salts. Eg., Na_2SO_3, N_2H_4, Salts of Mg, Zn or Ni.

$$\frac{1}{2} O_2 + H_2O + 2e^- \text{-----}\blacktriangleright 2OH^-$$

Vapour phase inhibitors:

These are organic inhibitors which are readily vapourised and form a protective layer on the metal surface.

These are conveniently used to prevent corrosion in closed spaces, storage containers, packing materials, sophisticated equipments etc.

Examples are Dicyclohexylammonium nitrate, dicyclohexyl ammonium chromate, benzotriazole, phenylthiourea etc.

Anodic protection

This is an electrochemical method of corrosion control in which an external potential control system, called potentiostat, is used to produce and maintain a thin non corroding, passive film on a metal or an alloy. The use of potentiostat is to shift corrosion potential into passive potential so that the corrosion of the metal is stopped.

The potential of the object (say acid storage tank) to be protected is controlled by potential controller (potentiostat) so that under certain potential range, the object becomes passive and prevents further corrosion. This potential range depends upon the relationship between the metal and the environment.

Applications:

1. Used in acid coolers in dilute sulphuric acid plants
2. used in storage tanks for sulphuric acid
3. used in chromium in contact with hydrofluoric acid

Limitations

1. This method cannot be applied in the case of corrosive medium containing aggressive chloride.
2. This cannot be applied if protection breaks down at any point, it is difficult to reestablish.

Protective coatings

Introduction

In order to protect metals from corrosion, it is necessary to cover the surface by means of protective coatings. These coatings act as a physical barrier between the coated metal surface and the environment. They afford decorative appeal and impart special properties like hardness, oxidation resistance and thermal insulation.

Classification

Protective coatings can be broadly classified into two types. They are

1. Inorganic coatings **2) Organic coatings**

Inorganic coatings are further classified into two types. They are

i) **Metallic coating:**
   1. Hot dipping- Galvanising, Tinning
   2. Metal cladding
   3. Cementation-Sherardising, Chromising, Calorising
   4. Electroplating.

ii) **Non-metallic coating:**
   1. Surface coating or chemical conversion coating - Chromate coating, Phosphate coating and Oxide coating.
   2. Anodising
   3. Enamel coating or Vitreous or Porcelain coating.

Organic coatings consists of

Paints, Varnishes, Lacquers and Enamels.

Paints

Paint is a viscous, opaque (not clear), mechanical dispersion mixture of one or more pigments (dye) in a vehicle (drying oil).

Requisites of a good paint:

A good paint should the following properties, it should

1) Have a high hiding power
2) Form a good and uniform film on the metal surface
3) The film should not crack on drying
4) Give a glossy film
5) The film produced should be washable
6) Give astable and decent colour on the metal surface
7) Have good resistance to the atmospheric conditions
8) Be fluid enough to spread easily over the surface
9) Possess high adhesion capacity to the material over which it is intended to be used
10) Dry quickly or in a reasonable duration.

11) The colour of the paint should not fade.

Constituents of paint

a. Pigment
b. Vehicle or medium or drying oil
c. Thinner
d. Driers
e. Fillers or Extenders
f. Plasticizers
g. Antiskinning agents

a) Pigment

It is a solid substance which imparts colour to the paint. It is an essential constituent of a paint. Its functions are to

I. Give opacity(cloudiness) and colour to the film
II. Provide strength to the paint
III. Provide an aesthetical appeal
IV. Give protection to the paint film by reflecting UV light.
V. Increase weather resistance of the film
VI. Provide resistance to paint film against abrasion.

The most commonly used pigments in paints and the compounds required as as follows:

White pigments - White lead, ZnO, $BaSO_4$, TiO_2, ZrO_2
Blue pigments - Prussian blue, ultramarine blue
Black pigments - Graphite, carbon black, lamp black
Red pigments - Red lead, Fe_3O_4, carmine
Green pigments - Chromium oxide, chrome green
Brown pigments - Burnt umber, ochre
Yellow pigments - Chrome yellow, lead chromate

b) Vehicle or drying oil or medium:

Vehicle is a liquid substance and film forming material. It holds all the ingredients of a paint in liquid suspension. Eg., linseed oil, tung oil.

Functions

i) To hold the pigment on the metal surface
ii) to form the protective film by evaporation or by other means.
iii) to impart water repellency, durability and toughness to the film
iv) to improve the adhesion of the film

a) Thinners

Thinners are volatile substances which evaporate easily after application of the paint. They are added to the paints for reducing the viscosity of the paints so that they can be easily applied to the metal surface. Eg., Dipentine, turpentine, toluol, xylol.

Functions

i) To reduce the viscosity of the paint
ii) To dissolve vehicle and the additives in the vehicle
iii) To suspend the pigments
iv) To increase the penetration power of the vehicle
v) To increase the elasticity of the paint film
vi) To help the drying of the paint film.

b) Driers:

These are the substances used to accelerate the process of drying. They are oxygen carrier catalysts. Eg., Naphthenates, linoleates, borates, resonates and tungstates of heavy metals (Pb, Zn, Co, Mn).

Functions:

i) To accelerate the drying of the oil film through oxidation, polymerization and condensation
ii) To improve the drying quality of the oil film.

c) Extenders or Fillers:

These are the inert materials which improve the properties of the paint. Eg., Gypsum, chalk, silica, talc, clay, $CaCO_3$, $CaSO_4$.

Functions:

i) To fill the voids (empty space or any curved area) in the film
ii) To act as a carrier for the pigment color.
iii) To reduce the cost of the paint
iv) To increase the durability of the paint
v) To reduce the cracking of dry paint
vi) To increase random arrangement of pigment particles.

d) Plasticisers:

These are added to the paint to provide elasticity to the film and to minimize its crack. Eg., Triphenyl phosphate, dibutyl tartarate, tributyl phthalate, tricresyl phosphate, diamyl phthalate.

e) **Antiskinning agents:**

These are sometimes added to some paints to prevent gelling and skinning of the finished product. Eg., Polyhydroxy phenols.

Metallic coatings

Corrosion of metals can be prevented or controlled by using methods like galvanization, tinning, metal cladding, electroplating, cementation, anodizing, phosphate coating, enamelling, electroless plating. Some of the methods are

1) **Hot dipping:**

It is used for producing a coating of low-melting metals such as Zn (m.p.=419 deg C), Sn (m.p.=232 deg C), Pb, Al etc., on iron, steel and copper which have relatively higher melting points. The process in immersing the base metal in a bath of the molten coating-metal, covered by a molten flux layer (usually zinc chloride).

2) **Galvanizing:**

It is the process of coating iron or steel sheets with a thin coat of zinc to prevent them from rusting. The process is iron or steel article is first cleaned with dil. Sulphuric acid and washed with distilled water and dried. The dried metal is dipped in bath of molten zinc, now the thin layer of zinc is coated on the iron or steel article.

3) **Metal cladding:**

It is the process by which a dense, homogeneous layer of coating metal is bonded firmly and permanently to the base metal on one or both sides. Corrosion resistant metals like nickel, copper, lead, silver, platinum and alloys like SS, nickel alloys, copper alloys, lead alloys can be used as cladding materials.

4) **Tinning:**

It is a method of coating tin over the iron or steel articles. The process is first treating steel sheet in dilute sulphuric acid and it is passed through a flux ($ZnCl_2$), next steel passes through a tank of molten tin and finally through a series of rollers from underneath (bottom of) the surface of a layer of palm oil.

Electroplating or electrodeposition

Electroplating is a coating technique. It is the most important and most frequently applied industrial method of producing metallic coating.

Electroplating is the process by which the coating metal is deposited on the base metal by passing a direct current through an electrolytic solution containing the soluble salt of the coating metal.

The base metal to be plated is made cathode whereas the anode is either made of the coating metal itself or an inert material of good electrical conductivity (like graphite).

Objectives:

Electroplating is carried out for

1. Decoration or better appearance
2. Increasing the resistance to corrosion of the coated metal.

3. Improving the hardness of the metal
4. Increasing the resistance to chemical attack
5. Electro refining.

Procedure

The article is to be plated first treated with organic solvent like carbon tetrachloride, acetone, tetrachloro ethylene to remove oils, greases etc. Then it is made free from surface scale, oxides, etc. by treating with dil. HCl or H_2SO_4 (acid pickling). The cleaned article is then made as the cathode of the electrolytic cell. The anode is either the coating metal itself or an inert material of good electrical conductivity. The electrolyte is a solution of soluble salt of the coating metal.

When direct current is passed, coating metal ions migrate to the cathode and get deposited there. Thus, a thin layer of coating metal is obtained on the article made as the cathode.

In order to get strong, adherent and smooth deposit, certain types of additives (glue, gelatin, boric acid) are added to the electrolytic bath.

In order to improve the brightness of the deposit, brightening agents are added in the electrolytic bath.

The favourable conditions for a good electrodeposit are,

i) Optimum temperature ii) Optimum current density iii) Low metal ion concentrations.

Gold Electroplating:

Anode: Gold Cathode: Metal article Electrolyte: Gold + KCN temperature: 60 deg C.
Current density (mA cm-2): 1-10

Use

i) This is used for electrical and electronic applications.
ii) It is used for high quality decorations and high oxidation resistant coatings
iii) Usually for ornamental jewellery, a very thin gold coating (about 1x10-4 cm) is given.

Electroless plating

Principle

Electroless plating is a newer technique of depositing a noble metal from its salt solution on a catalytically active surface of the metal to be protected by using a suitable reducing agent without using electrical energy.

The reducing agent reduces the metal ions into metal which gets plated over the catalytically activated surface giving a uniform and thin coating.

Metal ions + reducing agents ________ Metal (deposited) + Oxidized product

Electroless nickel plating

Pretreatment and activation of the surface

The surface to be plated is first degreased by using organic solvents or alkali, followed by acid treatment.

- i) The surface of the stainless steel is activated by dipping in hot solution of 50 % dilute sulphuric acid.
- ii) The surface of magnesium alloy is activated by thin coating of zinc or copper over it.
- iii) Metals (Al, Cu, Fe) and alloys like brass can be directly nickel plated without activation.
- iv) Non metallic articles like plastics, glass are activated by dipping them in the solution containing $SnCl_2$ +HCl, followed by dipping in palladium chloride solution. On drying, a thin layer of palladium is formed on the surface.

Applications

1) It is used extensively in electronic appliances.
2) It is used in domestic as well as automotive fields (eg., jewellery, tops of perfume bottles).
3) Its polymers are used in decorative and functional works.
4) Its plastic cabinets are used in digital as well as electronic instruments.

MATERIAL HANDLING SYSTEMS

CONVEYING

Transportation of solids is commonly referred as Conveying

OBJECTIVES

1. Decreased product cost and increased manufacturing capacity.
2. Decreased cost of raw material
3. Decreased processing time and conservation of energy
4. Compliance with cGMP
5. Minimum contamination & Dust formation
6. Increased employee safety and reduced labour costs

Classification

a) **Belt Conveyors**
b) **Screw Conveyors**
c) **Pneumatic Conveyors**
d) Chain Conveyors*
e) Bucket Conveyors *****
f) ***** Not widely used in pharmaceutical industries

Belt Conveyor

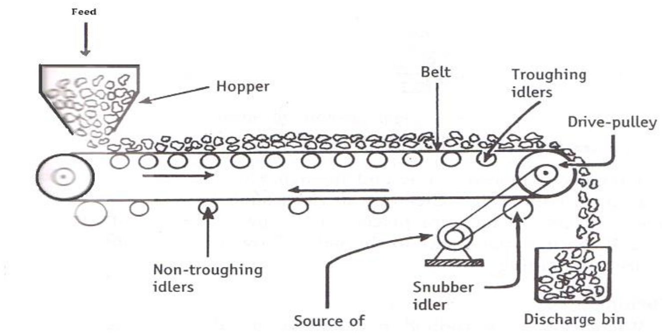
Parts

1. Belt & Belt tightening systems
2. Belt drive & Power supply
3. Roller supports
4. Feeding system
5. Discharge System

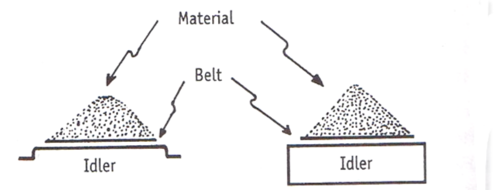
Belt conveyor

Construction

1. A skeleton of several piles of cotton duck, each layer is bonded with rubber
2. Both end of the belt is connected together so that the belt can revolve freely and continuously.
3. The belt ay run horizontally or little inclined from feed point
4. A belt taut (Stretch)
5. Idlers (Pulleys)-Toughing and Non- Troughing (a long, narrow open container)
6. Tighteners
7. Feed arrangement
8. Drive pulley
9. Snubber idler (shock absorber)
10. Discharge bin

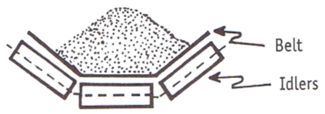


Construction of belt conveyor



(a) Flat belt on continuous plate (non-troughing)

(b) Flat belt on flat belt idlers (or non-troughing)



Support Idlers

Working

- ✓ When power is applied the belt start rotating
- ✓ The material to be transported is loaded on the centre of the belt with the help of a hopper.
- ✓ The belt move on troughing idlers
- ✓ Once material is unloaded, the belt returns on non troughing idlers to the point of feeding

Pharmaceutical Applications

- ✓ Transport of containers for filling, caping,sealing,labelling,pasting,visual inspection etc.in the production of injectables, liquid orals, ointments and jellies
- ✓ In the strip and blister packing of tablets, capsules and packing of them in cartons
- ✓ In the manufacture of Lozenges

Advantages

1. Belts can be made of asbestos fibers, neoprene, teflon and vinyl polymer. They can be designed for covering lengthy distances
2. Routine maintenance is easy
3. Economical in terms of cost per unit tonne that it can handle

Disadvantage

Belt conveyor is expensive

Screw conveyors

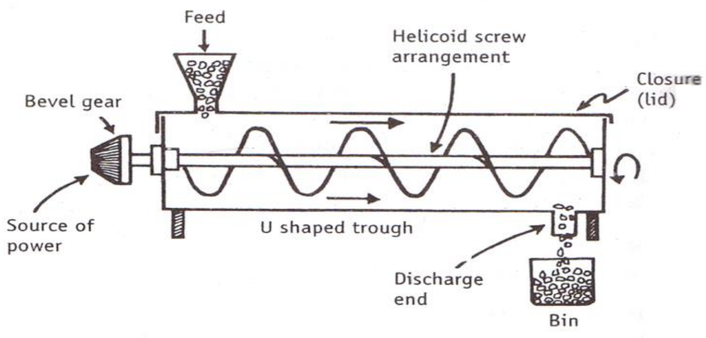
Parts

1. Trough System
2. Flights & Power supply
3. Feeding arrangements
4. Discharge arrangements

Construction

1. Trough is a "**U**" shaped vessel in which the material to be transported is enclosed
2. Screw consist of a spiral blade called as a ***flight*** usually helicoid in nature which is welded to a central shaft
3. ***Helicoid***- An object having a three-dimensional shape like that of a wire wound uniformly in a single layer around a cylinder or cone
4. Hangers are also used for suspending the flight

5. The discharge ends are called as ***box-ends.***
6. The shaft is rotated around the axis
7. Feed is introduced by ***spouts*** (a pipe, trough, or chute for conveying liquid, grain, etc.)
8. At the discharge end, containers are placed to collect the material



Screw conveyor

Working

✓ When power is applied, the drive rotates the shaft through the bevel gears.

Bevel-a surface or edge which slopes away from a horizontal or vertical surface

✓ Material will be trapped between the gaps of the spiral blades

✓ As the flight moves, the material also will move forward along the path of the sections

Applications

✓ In the transportation of finely divided solids or pasty solids

✓ Different density materials can be handled

✓ Special flights and accessories are available for operations such as mixing, de-watering, heating and cooling

✓ High capacity conveyors

Advantages

1. Materials can be conveyed horizontally, vertically or inclined
2. Easy operation\ Less space consumption
3. Variety of materials can be handled
4. High and low temperatures can be maintained by insulating the casing

5. Can be made dust free

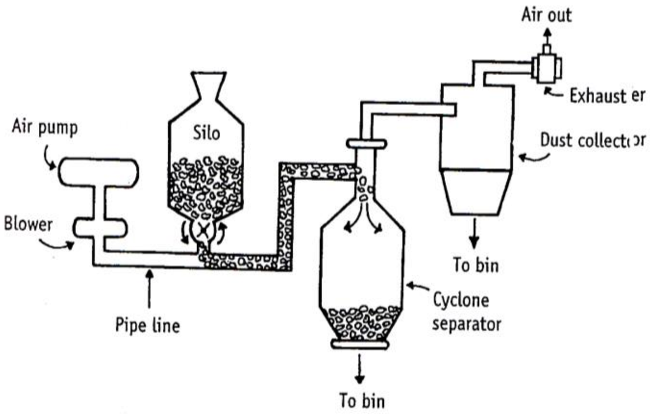
Disadvantages

1. Low speed
2. Power consumption is high

Pneumatic conveyors

Parts

1. Air supply or vacuum system
2. Air slide & pipeline
3. Feed Arrangements
4. Discharge arrangements (Air & Feed Arrangements)



Pneumatic conveyor

Principle

1. When high velocity of air is passed through a bed of solids, individual particles are dragged by the air and suspended in the air.
2. Such a system is known as ***fluidized bed***
3. The suspension behaves as a fluid and can be transported from one point to another point and at the discharge end, the gas is separated and the solids are collected.

Construction

- Fans /cycloidal blowers are connected to the conveying system
- An air slide consists of a chute with a porous base through which air passes
- Material to be transported is connected to the air slide through a rotary feeder valve
- The other end of the pipeline is connected to a cyclone separator

Slide: Move along a smooth surface, especially downwards, while maintaining continuous contact with it.

Chute: A sloping channel or slide for conveying things to a lower level.

Working

1. The cycloidal blowers /fans produce air at about 7.0 kps
2. This air is passed through the air slide at the rate of 1.5 meter per minute
3. The rotary feeder valve rotates and allows the feed to enter the pipeline
4. These solids are suspended into a stream of air in a fluidized state until it reaches the receiving end.
5. The material is then passed through a cyclone separator for sorting out different size particles.

Advantages

1. The gas can be recycled.
2. In the mfg.of powdered insufflations
3. Light and bulky materials can be transported
4. Handling of unpleasant and injurious toxic materials, because it is a closed system

Advantages

1. Economical
2. Friction losses are negligible

Disadvantages

1. Utilizes more power
2. Erosion of internal surface of vessels can happen
3. Uneven contact of gas and solids is observed

Storage of solids

Bunkers (large container or compartment for storing fuel)

Used for pulverized solid materials

Bin

Cylindrical rectangular vessel of concrete or metal. They are wide & short

Silo

Tall and relatively small in diameter

Hopper

Small vessel with a sloping bottom. It is used for temporary storage

References

1. Unit operations of Chemical Engineering McCabe, Smith, Harriot Sixth Edition Mc GrawHill

2. Pharmaceutical Engineering –Principles and practices by C.V. S Subramanyam, J. Thimma Setty, V. Kusumdevi and Sarasija Suresh - Vallabh Prakashan, 2007 edition

3. Introduction to Pharmaceutical Engineering by Anant Paradkar, Nirali Prakashan eleventh edition (2009)

4.Pharmaceutical Engineering by K,Sambamurthy , New Age International Private Limited Publishers First edition (2005)

Kerala University of Health Sciences

Syllabus (2017)

PHARMACEUTICAL ENGINEERING (Theory)

45 Hours

Scope: This course is designed to impart a fundamental knowledge on the art and science of various unit operations used in pharmaceutical industry.

Objectives: Upon completion of the course student shall be able:

1. To know various unit operations used in Pharmaceutical industries.
2. To understand the material handling techniques.
3. To perform various processes involved in pharmaceutical manufacturing process.
4. To carry out various test to prevent environmental pollution.
5. To appreciate and comprehend significance of plant lay out design for optimum use of resources.
6. To appreciate the various preventive methods used for corrosion control in Pharmaceutical industries.

Course content

UNIT-I 10 Hours

- Flow of fluids: Types of manometers, Reynolds number and its significance, Bernoulli's theorem and its applications, Energy losses, Orifice meter, Venturimeter, Pitot tube and Rotameter.

- Size Reduction: Objectives, Mechanisms & Laws governing size reduction, factors affecting size reduction, principles, construction, working, uses, merits and demerits of Hammer mill, ball mill, fluid energy mill, Edge runner mill & end runner mill.

- Size Separation: Objectives, applications & mechanism of size separation, official standards of powders, sieves, size separation Principles, construction, working, uses, merits and demerits of Sieve shaker, cyclone separator, Air separator, Bag filter & elutriation tank.

- Mixing: Objectives, applications & factors affecting mixing, Difference between solid and liquid mixing, mechanism of solid mixing, liquids mixing and semisolids mixing. Principles, Construction, Working, uses, Merits and Demerits of Double cone blender, twin shell blender, ribbon blender, Sigma blade mixer, planetary mixers, Propellers, Turbines, Paddles & Silverson Emulsifier,

UNIT-II 10 Hours

- Evaporation: Objectives, applications and factors influencing evaporation, differences between evaporation and other heat process. principles, construction, working, uses, merits and demerits of Steam jacketed kettle, horizontal tube evaporator, climbing film evaporator, forced circulation evaporator, multiple effect evaporator& Economy of multiple effect evaporator.
- Heat Transfer: Objectives, applications & Heat transfer mechanisms. Fourier's law, Heat transfer by conduction, convection & radiation. Heat interchangers & heat exchangers.

UNIT- III 10 Hours

- Drying: Objectives, applications & mechanism of drying process, measurements & applications of Equilibrium Moisture content, rate of drying curve. principles, construction, working, uses, merits and demerits of Tray dryer, drum dryer spray dryer, fluidized bed dryer, vacuum dryer, freeze dryer.
- Distillation: Objectives, applications & types of distillation. principles, construction, working, uses, merits and demerits of (lab scale and industrial scale) Simple distillation, preparation of purified water and water for injection BP by distillation, flash distillation, fractional distillation, distillation under reduced pressure, steam distillation & molecular distillation

UNIT-IV 08 Hours

- Filtration: Objectives, applications, Theories & Factors influencing filtration, filter aids, filter media. Principle, Construction, Working, Uses, Merits and demerits of plate & frame filter, filter leaf, rotary drum filter, Meta filter & Cartridge filter, membrane filters and Seitz filter.

- Centrifugation: Objectives, principle & applications of Centrifugation, principles, construction, working, uses, merits and demerits of Perforated basket centrifuge, Nonperforated basket centrifuge, semi continuous centrifuge & super centrifuge.

UNIT- V 07 Hours

- Materials of pharmaceutical plant construction, Corrosion and its prevention: Factors affecting during materials selected for Pharmaceutical plant construction, Theories of corrosion, types of corrosion and their prevention. Ferrous and nonferrous metals, inorganic and organic non metals.

- Material handling systems: Objectives & applications of Material handling systems, different types of conveyors such as belt, screw and pneumatic conveyors.

Recommended Books: (Latest Editions)

1. Introduction to chemical engineering – Walter L Badger & Julius Banchero, Latest edition.

2. Solid phase extraction, Principles, techniques and applications by Nigel J.K. Simpson- Latest edition.

3. Unit operation of chemical engineering – McCabe Smith, Latest edition.

4. Pharmaceutical engineering principles and practices – C.V.S Subrahmanyam et al., Latest

edition.

5. Remington practice of pharmacy- Martin, Latest edition.

6. Theory and practice of industrial pharmacy by Lachmann., Latest edition.

7. Physical pharmaceutics- C.V.S Subrahmanyam et al., Latest edition.

8. Cooper and Gunn’s Tutorial pharmacy, S.J. Carter, Latest edition.