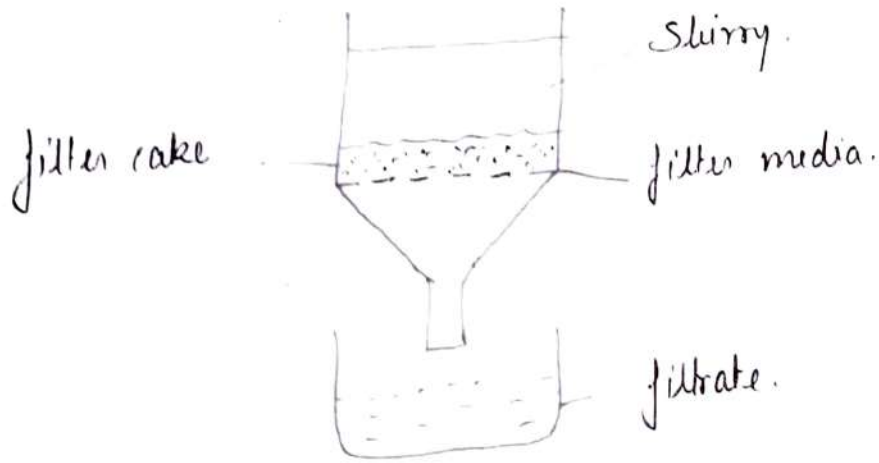
filtration

* filtration is defined as a process of separation of solids from a fluid by passing it through a porous medium that retains the solids, and allow to fluid to pass through
* Slurry - Solution to be filtered.
* filter media - porous medium used to retain the solids
* filter cake - Accumulated solids on filter media.
* filtrate - clear liquid passed through filter media.



* When solids are present in a very low concentration (less than 1% w/v) the process is called clarification

Applications of filtration:-

(1) Production of Sterile products:-

* Air is filtered through HEPA (High Efficiency Particulate air filter)

or laminar air bench to obtain sterile air.

* A solution is passed through a bacteria proof filter in order to obtain sterile solution.

(2) Production of Bulk drugs :-

* Solids of intermediates and final product are separated from the reaction mixture by filtration.
* Impurity can also be removed.

(3) Production of liquid oral formulations :-

* Filtration is an essential step in production of oral liquids to get clear solution.
* It is used in -
  - → Dewaxing of oils.
  - → removal of suspended oils
  - → removal of undesirable solids
  - → Clarification of potable water

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Theories of filtration

* The flow of a liquid through a filter follows basic rules of flow of any liquid.
* The rate of flow may be expressed as -

$$\text{Rate} = \frac{\text{driving force}}{\text{resistance.}}$$

* It is expressed as volume (litres) per unit time
* The resistance to flow is expressed as -

$$\text{Resistance to movement} = \frac{\text{pressure upstream} - \text{pressure down stream}}{\text{length of capillaries}}$$

→ Poiseuille's Equation :-

* Poiseuille considered the filtration is similar to the streamline flow of a liquid under pressure through capillary.

$$V = \frac{\pi \cdot \Delta P \, r^4}{8 L \eta}$$

V = rate of flow (L/s)

ΔP = pressure difference across the filter (Pascals)

r = radius of the capillary in filter bed. (m)

L = Length of filter cake. (m)

η = viscosity of filtrate (Pa.s)

Darcy's Equation :

» The factors influencing the rate of filtration has been incorporated into an equation by Darcy -

$$V = \frac{K A \Delta P}{\eta L}$$

Where K = permeability coefficient of cake

A = Surface area of porous bed.

Kozeny - Carman Equation :-

$$V = \frac{A}{\eta S^2} \times \frac{\Delta P}{K L} \times \frac{\varepsilon^3}{(1-\varepsilon)^2}$$

ε = porosity of the cake

S = Specific surface area of particles comprising the cake.

K = Kozeny constant.

Type of filtration

* Filtration can be classified -

(1) Depth filtration :-

* In this method the removal of suspended material

from the liquid suspension is done by passing liquid through filter bed composed of granular or compressed filter media.

* The filter bed are packed bed of sand, anthracite, or other granular medium
* This method is used in the treatment of surface or potable water supply.

(2) Surface filtration:-

* Surface filtration is involves the removal of solid from a liquid by means of sieving.
* The material that have been used as a filter media include woven wire cloths, cloth fabrics and variety of synthetic materials.

(3) Membrane filtration:-

* Membrane filtration is a separation process that uses a semi-permeable membrane.
* It consists of two steps -
  (a) Permeate the liquid passes through membrane and
  (b) retention of species being separated.

Factors influencing filtration

(1) Surface area of filter media:-

* According to Darcy's equation the rate of filtration is directly proportional to surface area of filter media.

* So rate of filtration can be increased by increasing surface area of filter media

(2) Pressure Drop Across the filter media:-

* The rate of filtration is directly proportional to the overall pressure drop across filter medium and filter cake
* The pressure drop can be achieved by -

Gravity:-

* The pressure developed depends on the density of liquid.
* A head of 10 meters of water creates a pressure difference of 100 kilopascals.

Applying pressure:-

* The most common method of obtaining a pressure difference by applying pressure on the surface of slurry
* It is achieved by pumping slurry into the filter.

Reducing pressure:-

* The pressure below the filter medium may be reduced ~~below~~ below atmospheric pressure
* It is achieved by connecting the filtrate receiver to a vacuum pump

(3) Viscosity of filtrate:-

* Rate of filtration is inversely proportional to the viscosity of the fluid
* Raising the temperature of the liquid lowers the viscosity may increase the rate of filtration
* This is not used when thermolabile materials are involved or filtrate is volatile.

Filter Media

* filter medium act as a mechanical support for the filter cake and is also responsible for the collection of solids

Characteristics and Ideal properties:-

(1) It should have sufficient mechanical strength.
(2) It should be inert
(3) It should not absorb the dissolved material.
(4) It should allow the maximum passage of liquid, while retaining the solids.

Materials:-

(1) Woven materials such as fiber or cloths:-

* Woven materials - wool, cotton, silk, glass, metals or synthetic fibers
* Synthetic fibers have greater chemical resistance than wool or cotton.

② Perforated sheet metal:-

* Stainless steel plates have been used as metal filters

③ Bed of granular solid:-

* In some processes a bed of granules of solids may be formed to reduce the resistance to the flow
* Examples of granular solids are gravel, sand, asbestos, paper pulp and Kieselguhr.

④ Membrane filter media:-

* Cartridge units are economical and available in pore size of 100 μm to even less than 0.2 μm
* These can be used as surface cartridges or depth type

filter Aids

* The filter aids form a surface deposit which screens out the solids and also prevents the plugging of the filter media.
* The object of filter aid is to prevent the blocking of medium and form a clean, porous cake.

Characteristics :-

(a) Should be chemically inert

(b) Low specific gravity.

(c) Insoluble in filtrate

(d) Recoverable.

Examples :-

→ Keiselguhr

→ Talc

→ Charcoal

→ Asbestos.

→ Bentonite

→ Fullers earth.

13.1. FACTORS AFFECTING FILTRATION

The following factors affect filtration:

1. The properties of the liquid, such as density, viscosity and corrosiveness.
2. The properties of the solid present, such as particle size, particle shape, particle size distribution and texure of the solid.
3. The proportion of solids in the slurry.
4. Whether the object is to collect the solid, the liquid or both.
5. Whether the solids have to be washed free from the liquid or the solvent.

ROTARY FILTER

Principle: Rotary filter is continuous in operation. It consists of a system that can remove the filter cake. So, they are suitable for filtering the concentrated slurry. They filter the slurry under vacuum through sieve-like mechanism on a rotating drum surface.

Construction: Rotary drum filter consists of a metal cylinder that is mounted horizontally (Fig. 9.2). The rotary drum is up to 3 meter in diameter and 3.5 meter in length, having an area of 20 meter square. The curved surface is perforated which supports filter cloth of the rotary filter. The drum is radically divided into separated compartments. By an internal pipe each compartment is connected to the centre of the drum through a rotating valve.

Working: During operation drum rotates at low speed. The drum just enters the slurry in the trough. When it (drum) dips in the slurry, due to the applied vacuum, the solid is deposited on the drum surface. The liquid filtered through the cloth and enter in an internal pipe and valve and at last it is collected in collecting tank.

After leaving the slurry section, drum enters the drainage area. Special attachment, like, cake compression rollers, may be included at this section. By this attachment, cake is consolidated by the compression mechanism. This process improves the efficiency of the washing and drying process. From the drainage section, the drum enters the water wash area. In this section water is poured on the cake. In order to suck the water wash and air through the solid cake, a separate vacuum system is

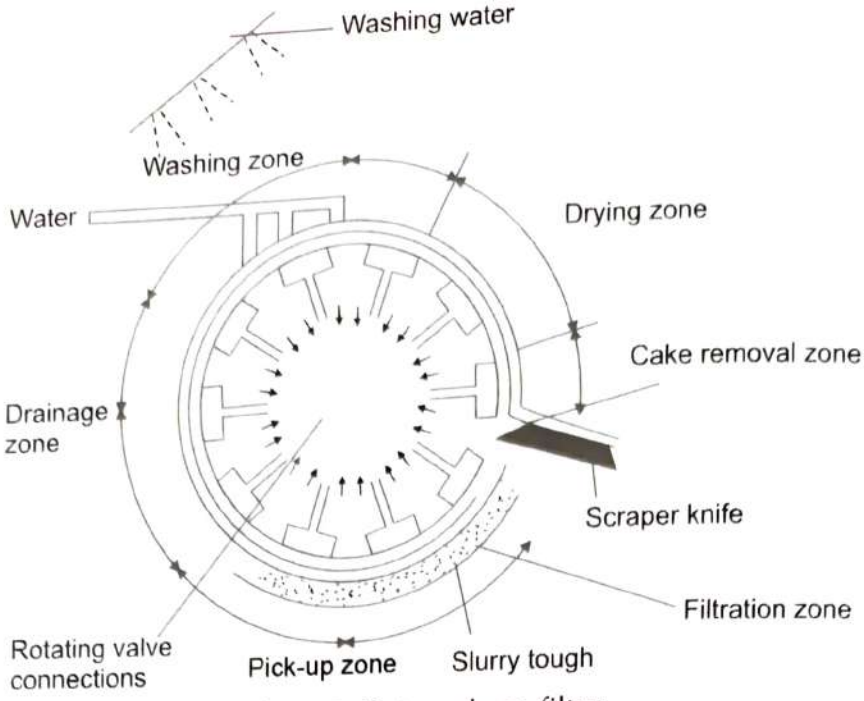


Fig. 9.2: Rotary drum filter

applied. Wash water is filtered into a separate collecting tank. After, leaving washing zone, drum enters into the drying zone where hot air is blown on the cake. Finally, the cake is scraped by knife and then drum is ready to complete another revolution.

Uses

- It is used for large quantities of slurry
- It is suitable for slurry containing considerable amounts of solids in the range of 15–30%.

Advantages

- Filtration area is large
- It is a continuous process and a complete automatic process
- The labour costs are very low
- Thickness of cake on the drum can be controlled by varying the speed of drum.

Disadvantages

- The rotary filter is very expensive process and its functioning is very complex
- Due to the air drawn through by the vacuum system the cake may break
- The pressure difference should not be more than 1 bar.

META FILTER (EDGE FILTER)

Principle: Meta filters are used as a surface filtration unit for coarse particles. It contains metal rings having semicircular projections which are arranged as a nest to form channels on the edge that offers resistance to the flow of solids.

Construction: Meta filter consists of a series of metal rings (Figs 9.3A and B). The thickness of the ring is about 0.8 mm and inner as well as outer diameters are about 15 and 22 mm respectively. These rings are threaded to formed channel on the edges. Each metal rings has various semicircular projections on one side of the surface. These projections are arranged the same way up. The rings are tightened on the drainage rod with nut. Hence also known as edge filters. These filters are mounted in a vessels

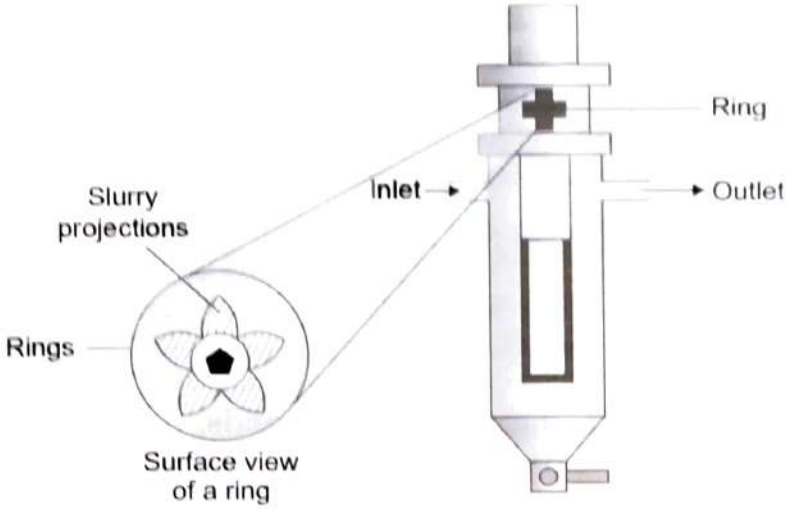


Fig. 9.3A: An edge filter

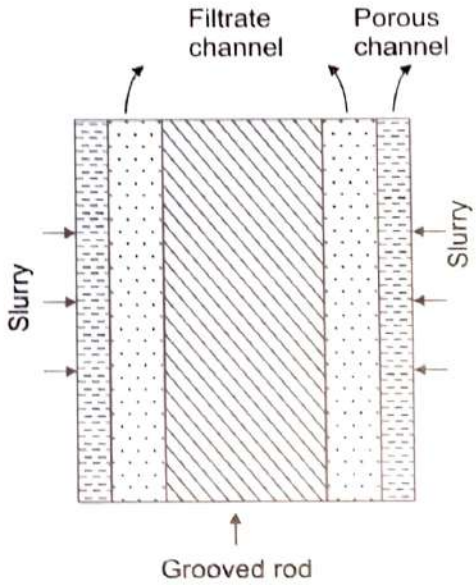


Fig. 9.3B: Mechanism of filtration through metafilters

and may be operated by the application of reduced pressure to the outlet direction or by pumping the slurry under pressure. To separate the fine particles from the slurry, first, a bed of a suitable material (e.g. kieselguhr) is built-up.

Working: In it filters are placed in a vessel and operated by pumping the slurry under pressure or occasionally by the applications of reduced pressure to the outlet side. Then the slurry passes through the channels formed on the edge between the rings and the clear liquid rises up and gets collected from the outlet into the receiver. It works as a strainer. To separate fine particles a bed of kieselguhr is first built-up. The pack of the rings works as a base on which a true filter medium can be supported.

Uses

- It is used for syrups clarification
- It is mainly used for filtering of injections
- It can be used for viscous liquids
- Meta filter are mostly used for insulin liquors.

Advantages

- Edge filter can be used under high pressure
- Running cost is very low and is very economical process
- They can be easily constructed by such metal that can provide excellent resistance against corrosion
- Cake can be easily removed by simple back flushing with water
- Sterile product can be easily filtered.

Disadvantages

The small surface area restricts the collection of solids.

FILTER PRESS

Principle: A **filter press** is used in separation processes, specifically in solid/liquid separation using pressure based principle provided by a slurry pump. It is used in fixed-volume and batch operations, therefore, the operation must be stopped to discharge the filter cake before the next batch can be started. The major components are the skeleton and the filter pack. The skeleton holds the filter pack together while pressure is being developed inside the filtration chamber. The chamber can only hold a specific volume of solids.

Construction: Plate and frame filter is made up of two types of units known as plate and frame. Filter medium usually filter cloth is placed between plate and frames as shown in Fig. 9.4. It may be made by various types of metal to prevent corrosion or metal contamination of the product. Non-metals generally plastic and wood are also used as satisfactory material of construction. There are many types of filter presses. The simplest type is open delivery system. It consists of single conduit for introduction of the slurry and the wash and a single opening in each plate for removal of the liquid. Other is closed delivery system. It consists of separate conduits for introducing the slurry and wash water. Some also have separate conduit for removing filtrate and wash water. The conduit may be at the corner, at the centre or at intermediate location. Plate has a studded or grooved surface to support the filter cloth and an outlet for the filtrate.

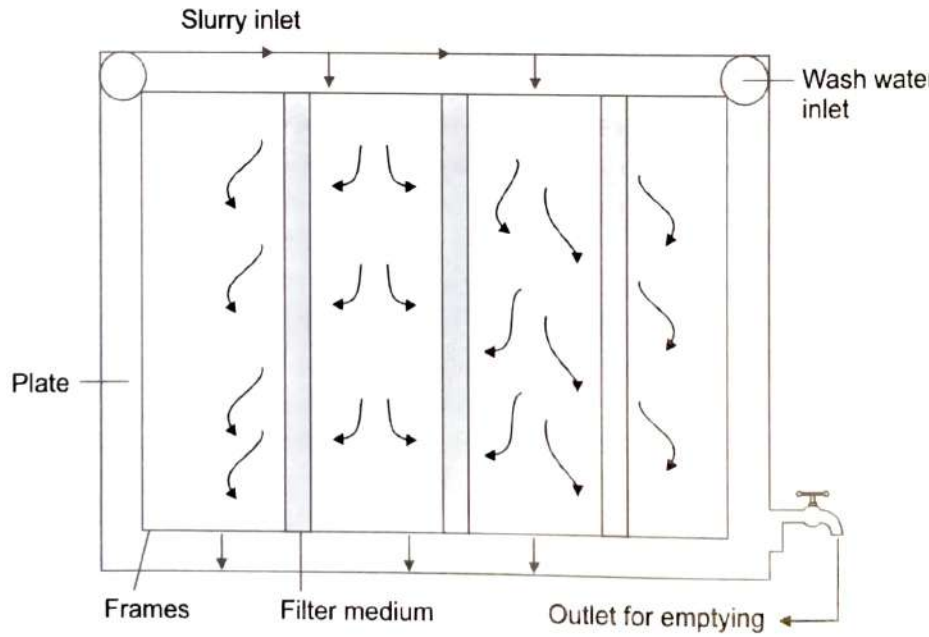


Plate and frame filter press

Centrifugation

Introduction:-

Definition:-

"Centrifugation is a unit operation used for separating the constituents present in a dispersion with the aid of centrifugal force".

* Centrifugal force → is used as driving force ↓ for separation.
* Centrifugation is useful → when separation by ordinary filtration is difficult.
* for example → Separation of highly viscous mixture → Colloidal dispersion (particles less than 5 mm)
* The equipment used are called centrifuges.

Process of Centrifugation:-

* The centrifuge consists of a container in which mixture of solid and liquid or two liquid is placed ↓ rotated at high speed ↓ mixture separated into its constituent parts by action of centrifugal force on their densities.
* A solid or liquid of higher specific gravity is thrown outward with greater force.

Applications :-

(1) Production of bulk drugs :-

* Centrifugation technique is used to separate
  ↓
  crystalline drugs form mother liquor
  (such as aspirin)

(2) Production of biological products :-

* Proteinaceous drugs / macromolecules > present in water as colloidal dispersion
  ↓
  centrifugal force is used to separate them from water.

* Insulin can be obtained in pure form
  ↓
  by selective precipitation of other fraction of proteins
  ↓
  subsequently separating by ultracentrifugation

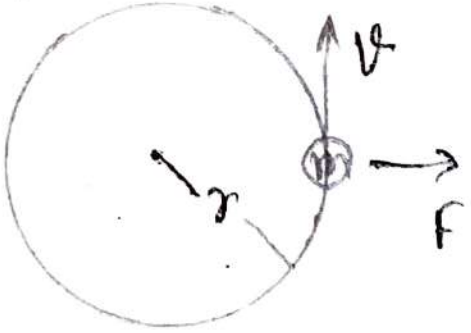
* It is used to separate blood cells from blood.

(3) Biopharmaceutical Analysis of drugs :-

* Drugs present in (blood / tissues fluid / Urine) → Are present in the form of colloidal dispersion
  ↓
  centrifugation is used for separating the drugs

Theory of Centrifugation

Consider a body of mass m kg rotating in circular path of radius r metres at a velocity v metre per minute



The force acting on the body in a radial direction is given by -

$$\text{force acting in radial direction } f = \frac{mv^2}{r} \quad \text{---(1)}$$

where F is centrifugal force.

$$\text{Gravitational force } G = mg \quad \text{---(2)}$$

where g = acceleration due to gravity

The centrifugal effect is expressed as a ratio of centrifugal force to gravitational force.

$$\text{Centrifugal effect } C = \frac{F}{G}$$

$$= \frac{mv^2}{r\,mg}$$

$$= \frac{v^2}{rg} \quad \text{---(3)}$$

But $v = 2\pi r n$ where n = speed of rotation.
(revolution per sec)

$$C = \frac{(2\pi r n)^2}{gr} = \frac{4\pi^2 r^2 n^2}{gr}$$

$$C = \frac{4\pi^2 r n^2}{g}$$

$2r = d$ where d is diameter of rotation

$$C = \frac{2\pi^2 d n^2}{g}$$

As $g = 9.807\ m/s^2$

$\pi = 3.14$

So $C = \dfrac{2 \times (3.14)^2 \times d\, n^2}{9.807}$

$C = 2.013\, n^2 d$