

# FILTRATION

## Definition <sup>[1]</sup>

**Filtration:** It may be define as a process of separation of solids from a fluid by passing the same through a porous medium that retains the solids but allows the fluid to pass through.

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

## Terms used in Filtration

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

## Process of filtration

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

## Applications of filtration <sup>[1]</sup>

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

## Mechanism of filtration <sup>[1, 4]</sup>

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

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

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

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

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

### Types of filtration <sup>[1]</sup>

#### Surface/ screen filtration –

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

#### Depth filtration -

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

### Theories of filtration <sup>[1]</sup>

- The flow of liquid through a filter follows the basic rules that govern the flow of any liquid through the medium offering resistance.
- The rate of flow may be expressed as- Rate = driving force / resistance
- The rate of filtration may be expressed as volume (litres) per unit time (dv/dt).
- Driving force = pressure upstream – pressure downstream
- Resistance is not constant.
- It increases with an increase in the deposition of solids on the filter medium.
- Therefore filtration is not a steady state.
- The rate of flow will be greatest at the beginning of filtration process, since the resistance is minimum.
- After forming of filter cake, its surface acts as filter medium and solids continuously deposit adding to thickness of the cake.

**Resistance to movement**= {pressure upstream- pressure downstream}/ length of capillaries

#### 1. Poiseuille's Equation

- Poiseuille considered that filtration is similar to the streamline flow of liquid under pressure through capillaries.
- Poiseuille's Equation is-  $V = \frac{\pi \Delta P r^4}{8 L \eta}$

Where,  $V$  = rate of flow,  $m^3/s$  (l/s)

$\Delta P$  = Pressure difference across the filter, Pa

$r$  = radius of capillary in the filter bed, m

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

$\eta$  = viscosity of filtrate, Pa.s

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

## 2. Darcy's Equation

- Poiseuille's law assumes that the capillaries found in the filter are highly irregular and non-uniform.
- Therefore, if the length of capillary is taken as the thickness of bed, a correction factor for radius is applied so that the rate is closely approximated and simplified.
- The factors influencing the rate of filtration has been incorporated into an equation by Darcy, which is:  
$$V = \frac{KA\Delta P}{\eta L}$$

Where,  $K$  = permeability coefficient of cake,  $m^2$

$A$  = surface area of porous bed (filter medium),  $m^2$

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

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

## 3. Kozeny-Carman (K-C) equation

- Kozeny-Carman equation is widely used for filtration.

$$V = \frac{A}{\eta S^2} \frac{\Delta P}{KL} \frac{\epsilon^3}{(1-\epsilon)^2}$$

Where,

$\epsilon$  = porosity of cake (bed)

$S$  = specific surface area of particles comprising the cake  $m^2 / m^3$

$K$  = Kozeny constant (usually taken as 5)

### **Limitations:**

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

### **Factors influencing filtration <sup>[1,4]</sup>**

#### **1. Surface area of filter medium –**

- Rate of filtration is Inversely proportional to specific surface of filter bed (According to K-C equation)
- Directly proportional to surface area of filter medium (According to Darcy's equation)
- Rate can be increased either using large filter or connecting a number of small units in parallel. Filter press works on principle of connecting units in parallel.

#### **2. Pressure drop across the filter medium**

- According to K-C equation the rate of filtration is proportional to the overall pressure drop across both the filter medium and filter cake.
- The pressure drop can be achieved in a number of ways:
  - Gravity- A pressure difference could be obtained by maintaining a head of slurry above the filter medium. The pressure developed will depend on the density of the slurry
  - Vacuum (Reducing pressure) - The pressure below the filter medium may be reduced below atmospheric pressure by connecting the filtrate receiver to a vacuum pump and creating a pressure difference across the filter.
  - Pressure - The simplest method being to pump the slurry into the filter under pressure
  - Centrifugal force - The gravitational force could be replaced by centrifugal force in particle separation

#### **3. Viscosity of filtrate**

- According to K-C equation rate of filtration is inversely proportional to the viscosity of the fluid.
- This is an increase in the viscosity of the filtrate will increase the resistance of flow.
- This problem can be overcome by two methods:
  - The rate of filtration may be increased by raising the temperature of the liquid, which lowers its viscosity. However, it is not practicable if thermolabile materials are involved or if the filtrate is volatile.
  - Dilution is another alternative but the rate must be doubled.

### **Filter Media <sup>[1,2]</sup>**

- The surface upon which solids are deposited in a filter is called the "Filter medium"

- Properties of ideal filter medium are as follows
- It should
  - Be capable of delivering a clear filtrate at a suitable production rate.
  - Have sufficient mechanical strength.
  - Be inert.
  - Retain the solids without plugging at the start of filtration.
  - Not absorb dissolve material.
  - Sterile filtration imposes a special requirement since the pore size must not exceed the dimension of bacteria or spores.

### **Material used as filter media**

- **Woven material**
  - Made up of wool, silk, metal or synthetic fibres (rayon, nylon etc.).
  - These include a- wire screening and b- fabrics of cotton, wool, nylon.
  - Wire screening e.g. stainless steel is durable, resistance to plugging and easily cleaned.
  - 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 reactivity with the slurry
- **Perforated sheet metal**
  - Stainless steel plates have pores which act as channels as in case of meta filters.
- **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.
- **Membrane filter media** •
  - These are cartridge units and are economical and available in pore size of 100  $\mu\text{m}$  to even less than 0.2  $\mu\text{m}$ . •
  - Can be either surface cartridges or depth type cartridges. •
  - **Surface cartridges** –
  - These are corrugated and resin treated papers and used in hydraulic lines.
  - Ceramic cartridges and porcelain filter candles are examples.
  - Can be reuse after cleaning.
  - **Depth type cartridges:**
  - Made up of cotton, asbestos or cellulose.
  - These are disposable items, since cleaning is not feasible.

### **Filter Aids**

- The objective of filter aid is to prevent the medium from becoming blocked and to form an open, porous cake, hence, reducing the resistance to flow of the filtrate.

- Filter aid forms a surface deposit which screens out the solids and also prevents the plugging of supporting filter medium.

#### **Characteristics of filter aids:**

- Chemically inert and free from impurities.
- Low specific gravity, so remain suspended in liquids.
- Porous rather than dense, so that pervious cake can be formed.
- Recoverable.

#### **Disadvantages:**

- Remove the coloured substances by absorbing them.
- Sometimes active principles such as alkaloids are absorbed on filter aid.
- Rarely, filters are source of contamination such as soluble iron salts, which can provoke degradation of sensitive ingredient

#### **Handling of filter aids**

- Filter aids may be used in either or both two ways:
  - **Pre- coating technique:** by forming a pre-coat over the filter medium by filtering a suspension of the filter aid.
  - **Body- mix technique:** A small proportion of the filter aid (0.1- 0.5 %) is added to the slurry to be filtered. This slurry is recirculated through the filter until a clear filtrate is obtained, filtration then proceeds to completion.
- Different flow rates can be achieved depending on grade of aid
  - **Low flow rate:** fine grade filter aids- mainly used for clarity
  - **Fast flow rate:** coarse grade filter aids- acceptable filtrate.
- **Examples of filter aids**
  - Diatomite (Keisलगुर) , obtained from natural siliceous deposits.
  - Perlite , it is an aluminium silicate.
  - Cellulose, Asbestos, charcoal, talc, bentonite , fullers earth etc.

#### **Classification of filtration equipments**

##### **Based on application of external force:**

- Pressure filter – Plate and frame filter press, metafilter
- Vacuum filters – Filter leaf
- Centrifugal filters

##### **Based on operation of filtration**

- Continuous filtration - Discharge and filtrate are separated steadily and uninterrupted
- Discontinuous filtration - Discharge of filtered solid is intermittent. Filtrate is removed continuously. Operation should be stopped to collect solid.

## Based on nature of filtration

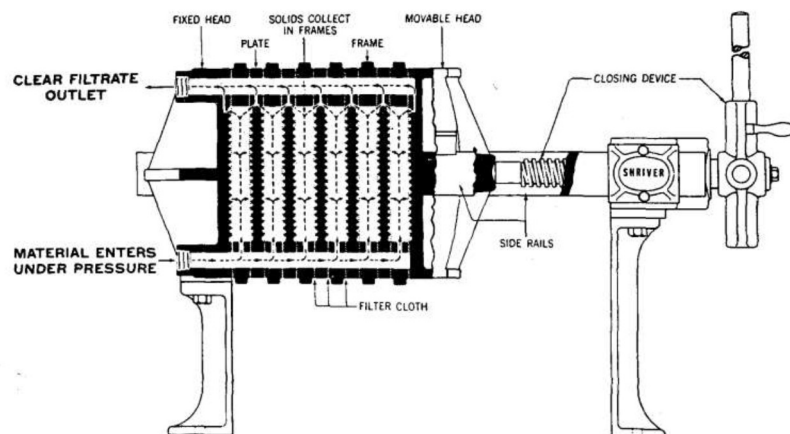
- Cake filters - Remove large amount of solids
- Clarifying filters - Remove small amounts of solids
- Cross-flow filters - Feed of suspension flows under pressure at fairly high velocity across the filter medium

## EQUIPMENTS <sup>[1, 2]</sup>

### Plate and frame filter press

#### Principle:

- Mechanism is surface filtration.
- The slurry enters the frame by pressure and flows through filter medium.
- The filtrate is collected on the plates and send to outlet.
- A number of frames and plates are used so that surface area increases and consequently large volumes of slurry can be processed simultaneously with or without washing.



**Fig 1- Filter press**

#### Construction

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

#### Frame-

- It contains a open space inside wherein the slurry reservoir is maintained for filtration and an inlet to receive the slurry.
- It is indicated by two dots in description.
- Frames of different thickness are available.

- It is selected based on the thickness of cake formed during filtration.
- Optimum thickness of frame should be chosen.

### **Plate**

- The plate has a studded or grooved surface to support the filter cloth and an outlet.
- It is indicated by one dot in description.
- Plate supports the filter medium, receiving the filtrate and outlet.
- The filter medium usually cloth is interposed between plate and frame.
- Plate, filter medium, frame, filter medium and plate are arranged in sequence and clamed to a supporting structure.
- It is normally described by dots as 1.2.1.2.1 so on.
- A number of plates and frames are employed so that the filtration area is as large as necessary.
- Number of filtration units are operated in parallel.
- Channels for slurry inlet and filtrate outlet can be arranged by fitting eyes to the plates and frames, these join together to form a channel.
- In some types only one inlet channel is formed, while each platev is having individual outlets controlled by valves.

### **Working**

Working can be divided into two steps

#### **1. Filtration operation**

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

#### **2. Washing of cake (if desirable)**

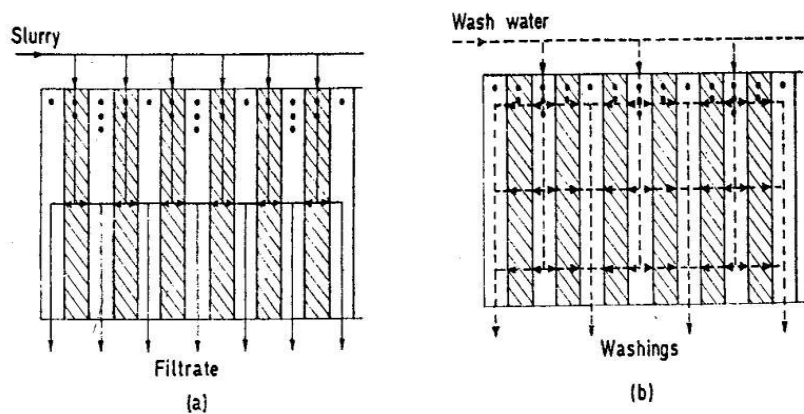
- When washing of cake is also required modified plate and frame filter is used.
- For this purpose an additional channel is included called as washing plate and are identified by 3 dots.



- In the half of the washing plate, there is a connection from wash water channel to the surface of plate.
- The sequence of arrangement of plates and frames can be represented by dots as 1.2.3.2.1.2.3.2.1 so on (between 1 and 1, 2.3.2 must be arranged).

### Procedure for washing the press

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



**Fig 2 – Diagrammatic working procedure**

### Special provisions:

- Any possible contamination can be observed by passing the filtrate through a glass tube or sight glass from the outlet on each plate.
- This permits the inspection of quality of filtrate. The filtrate goes through the control valves to an outlet channel.
- The filtration process from each plate can be seen. In the event of broken cloth, the faulty plate can be isolated and filtration can be continued with one plate less.

### Uses:

- Sterile filtrate can be obtained by using asbestos and cellulose filter sheet (for this, whole filter press and filter medium have been sterilized previously).
- Filtration of viscous liquid can also be done by incorporating heating/cooling coils in the press.

### Advantages

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

### Disadvantages

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

### Filter leaf

#### Principle:

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

#### Construction:

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

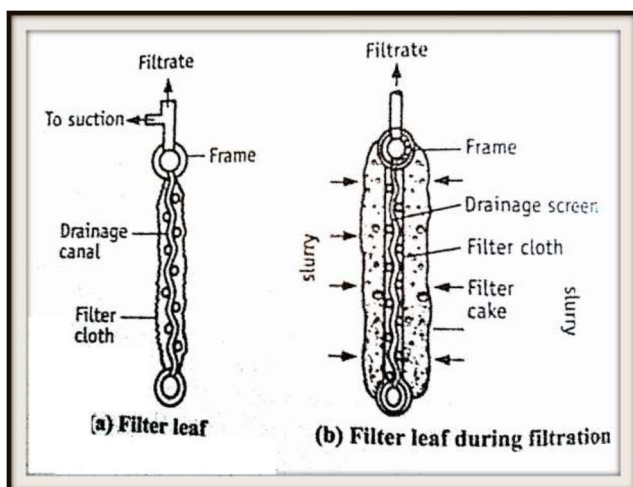


Fig 3 – Filter leaf

## Working

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

## Use:

- Use for the filtration of slurry which do not contain high solid content, about 5%, i.e. dilute suspensions.

## Advantages

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

## Sweetland filter (variant of filter leaf)

- An alternative method is to enclose the filter leaf in a special vessel into which the slurry is pumped under pressure.
- A number of leaves are connected to a common outlet, to provide a large area for filtration.

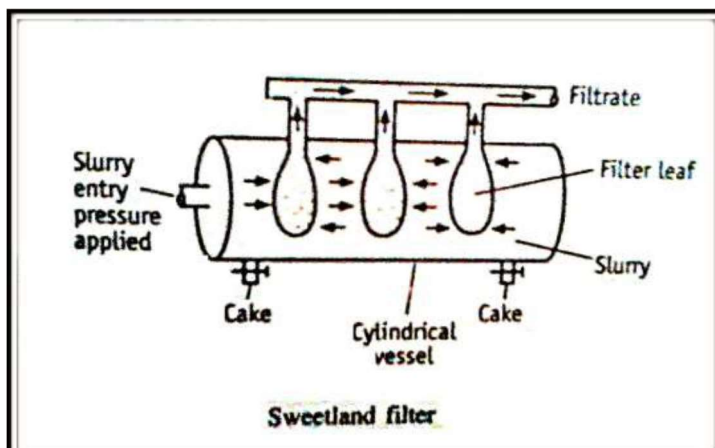


Fig 4 – Sweetland filter

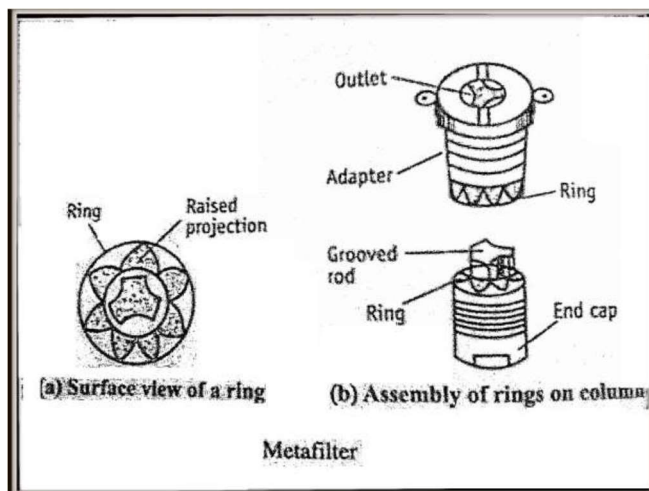
## Metafilter

### Principle:

- Mechanism is surface filtration.
- In this, metal rings contain semicircular projections, which are arranged as a nest to form channels on the edges.
- This channel offers resistance (strainer) to the flow of solids (coarse particles).
- The clear liquid is collected into receiver from the top.

## Construction

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



**Fig 5 - Metafilter**

## Working

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

## Uses

Metafilters can be used for-

- Clarification of syrups
- Filtration of injection solutions
- Clarification of insulin liquors
- Filtration of viscous liquids can be achieved by applying pressure.

## Advantages

- Can be used under high pressures, without any danger of bursting the filter medium.
- Running cost is low, as separate filter medium is not used.

- Can be constructed from a material that can provide excellent resistance to corrosion and avoid contamination of sensitive products.
- It is extremely versatile filter because fine as well as large both type of particles can be separated.
- Removal of cake can be carried out by simply back- flushing with water.
- Change over from one batch to another or one product to another is easy.
- Sterile products can be handled.

### Cartridge filter

#### Principle:

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

#### Construction:

- It has cylindrical configuration made with disposable or changeable filter media.
- Made up of either plastic or metal.
- Consist of two membrane filters (sieve like) made of polypropylene: prefilter and actual filter for filtration.
- A protective layer surrounds them.
- The cartridge is housed in a holder and a number of cartridges can be placed in a same housing.
- The housing is closed with the lid.
- Housing has provisions for slurry inlet and outlets.

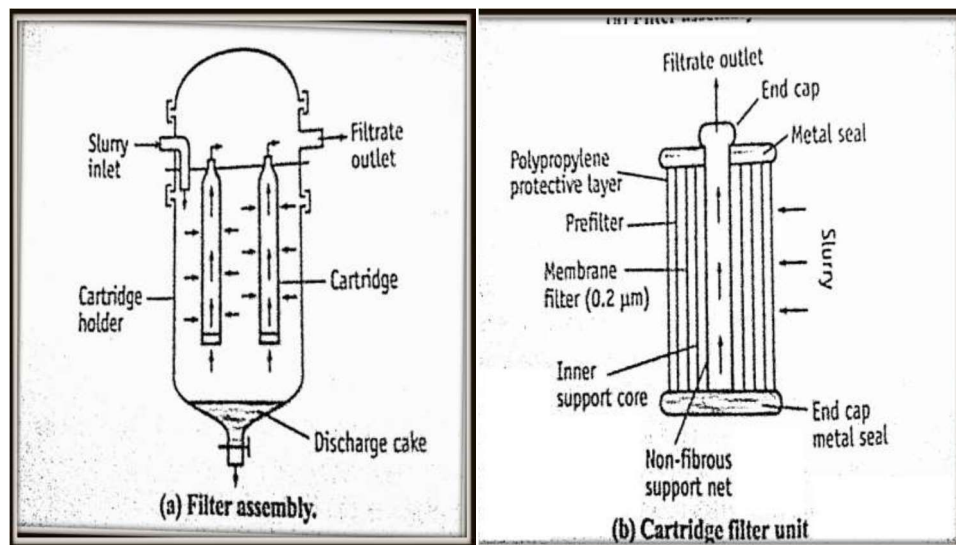


Fig 6 – Cartridge filter

#### Working:

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

#### Uses:

- Particularly useful for preparation of particulate free solutions for parenterals and ophthalmic uses.
- This filter holder will process 1000 – 15000 litres of sterile solution per hour.

**Advantages:**

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

**Disadvantages:**

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

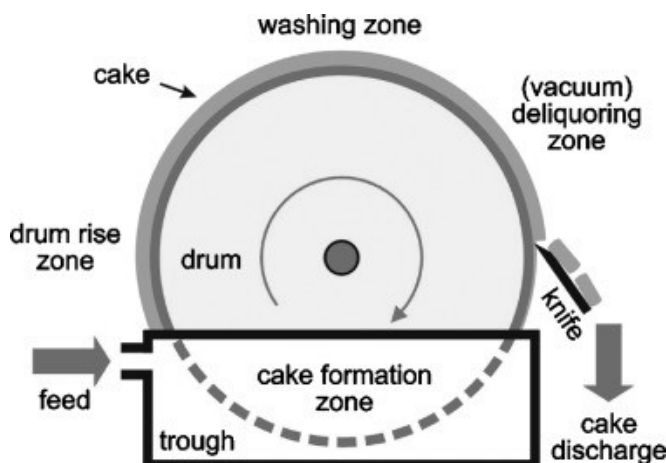
**Rotary drum filter<sup>2</sup>**

**Principle:**

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

**Construction:**

- It consists of a metal cylinder mounted horizontally.
- The drum may be up to 3 meters in diameter and 3.5 meters in length and gives surface area of 20 meter square.
- The curved surface being a perforated plate, supporting a filter cloth.
- Internally, it is divided into several sectors and a separate connection is made between each sector and a special rotary valve.



**Fig 7 – Rotary drum Filter**

**Working**

- The drum is dipped into the slurry and vacuum applied to the outlet, which is connected to the filtrate receiver.
- When the cake has formed, the cake drained or partially dried by vacuum.
- The drum is sprayed with water to wash the cake.

- Retaining the vacuum connection drains the cake and produces partial dryness then, removed by a doctor knife.
- When the solids of the slurry are too much that the filter cloth becomes blocked with the particles, a pre-coat filter may be used.
- A pre-coat of filter aid is deposited on the drum prior to the filtration process.

#### **Uses**

- The rotary filter for continuous operation on large quantities of slurry.
- Suitable for slurry contains considerable amounts of solids in the range 15-30%.
- Examples of pharmaceutical application includes the collection of calcium carbonate, magnesium carbonate, and starch.
- The separation of the mycelium from the fermentation liquor in the manufacture of antibiotics.

#### **Advantages**

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

#### **Disadvantages**

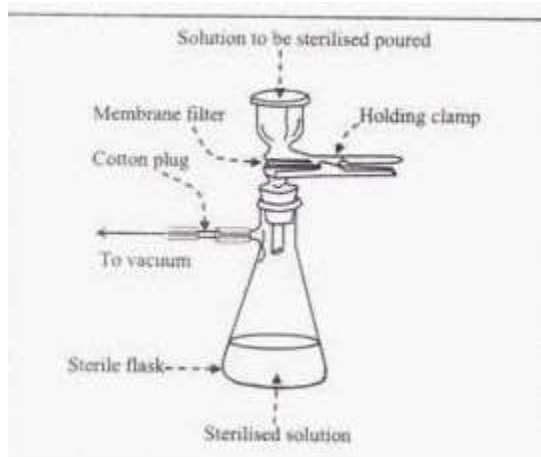
- The rotary filter is a complex piece of equipment, with many moving parts and is very expensive.
- In addition to the filter itself, some accessories are connected, e.g., a vacuum pump, vacuum receivers, slurry pumps and agitators are required.
- The cake tends to crack due to the air drawn through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

#### **Membrane filters <sup>[2]</sup>**

Membrane filters act as a barrier to separate contaminants from water, or they remove the particles contaminating the water. Reverse osmosis, ultrafiltration, and nanofiltration all use a membrane in their different filtration processes.

#### **Construction**

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



**Fig 8- Membrane filter**

### Working

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

### Uses

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

### SEITZ FILTER <sup>[3]</sup>

#### Principle

- It is based on filtration of asbestos pad filter disc

#### Construction

- It consists of two parts. Lower part fitted with a perforated plate over which compressed asbestos pad is placed.
- Upper part has a valve through which pressure can be applied.
- Both parts joined together by winged nuts.
- A valve is present on the upper part through which vacuum is applied
- The asbestos pads may yield alkali and cause precipitation of alkaloids
- It may shed fibres into the filtrate and absorb drug from solution.





**Fig 9- Seitz Filter**

**Advantages:**

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

**Disadvantages:**

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

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## CENTRIFUGATION

### Definition<sup>[1,3]</sup>

- Centrifugation is a process which involves the use of the centrifugal force for the sedimentation of heterogeneous mixtures with a centrifuge, used in industry and in laboratory settings.
- This process is used to separate two immiscible liquids.
- More-dense components of the mixture migrate away from the axis of the centrifuge, while less dense components of the mixture migrate towards the axis.
- Centrifuge is a device for separating particles from a solution according to their size, shape, density, viscosity of the medium<sup>1</sup>.

### Applications of centrifugation<sup>[1,3]</sup>

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

### Theory of centrifugation<sup>[2]</sup>

- The centrifuge works on sedimentation. Spinning sample at a high speed. The component of a mixture is subjected to centrifugal force. Dense particles migrate away from axis of rotation and lighter ones towards it.
- **Relative Centrifugal Force (RCF)**
- RCF is the ratio of the centrifugal acceleration at a specified radius and the speed to the standard acceleration of gravity.
- Relative Centrifugal force is defined as  $f = M\omega^2 r$

Where,

F= intensity of centrifugal force

M= mass of particle

$\omega$ = angular velocity of rotation

R= distance of migrating particles from central axis of rotation.

The RCF value for any centrifuge may be calculated from the following equation:

$$\text{RCF} = 1.18 \times r \times (\text{rpm})^2 \times 10^{-5}$$

Where,

$1.18 \times 10^{-5}$  = An empirical factor

r is the radius in cm from the centre of the centrifuge shaft to the external tip of centrifuge tube.

rpm is the number of revolution per minute of the centrifuge rotor

- The time required to separate particles depends on the rotor speed, radius of rotor and effective path length travelled by sedimented particles.
- The following formula useful for the calculation of the speed required for a rotor with a radius that differ from the radius with which a prescribed,
- $R.P.M. = 1000 \times \sqrt{RCF/1.18 \times r}$
- The length of time of centrifugation also can be calculated so, that running with an alternative rotor of a different size is equivalent to running with a original rotor.

## **CLASSIFICATION OF CENTRIFUGES [2]**

### **Sedimentation centrifuge**

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

### **Filtration centrifuge**

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

## **EQUIPMENT [1, 3]**

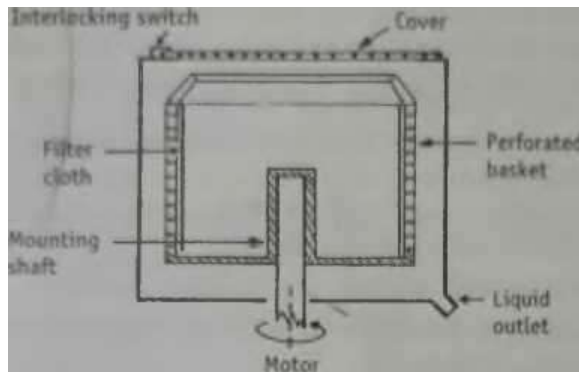
### **PERFORATED BASKET CENTRIFUGE**

#### **Principle:**

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

#### **Construction:**

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



**Fig 1- PERFORATED BASKET CENTRIFUGE**

**Working:**

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

**Uses:**

- Performed basket centrifuge is extensively used for separating crystalline drugs (such as aspirin) from the mother liquor. Free flowing product can be obtained because mother liquor is removed completely.
- It is also used for removing unwanted solids from a liquid. For example, precipitated proteins are removed from insulin.

**Advantages:**

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

**Disadvantages:**

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

## NON-PERFORATED BASKET CENTRIFUGE

### Principle:

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

### Construction:

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

### Working:

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

### Uses:

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

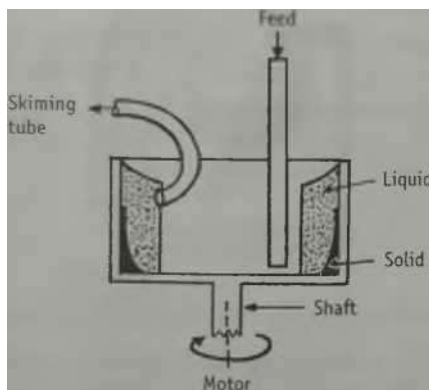


FIG 2-NON-PERFORATED BASKET CENTRIFUGE

## SEMI-CONTINUOUS CENTRIFUGE OR SHORT CYCLE AUTOMATIC BATCH CENTRIFUGE

### Principle:

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

### Construction:

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

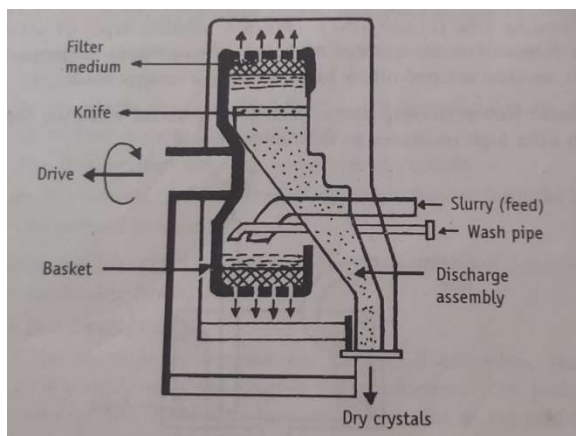


Fig 3 - SEMI-CONTINUOUS CENTRIFUGE

### Working:

- The perforated basket is allowed to rotate and slurry is introduced from the side pipe.
- During centrifugation, the slurry passes through the perforated wall. The solids are retained in the basket, while filtrate leaves the basket, which is collected at outlet.

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

**Advantages:**

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

**Disadvantage:**

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

**SUPERCENTRIFUGE**

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

**Principle:**

- It is a sedimentation centrifuge.
- The separation is based on the difference in the densities between two immiscible liquids. Centrifugation is done in the bowl of small centrifuge.
- During centrifugation, the heavier liquid is thrown against the wall, while the lighter liquid remains as an inner layer.
- The two layers are simultaneously separated using modified weirs.

**Construction:**

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