

Filtration

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Contents

- Introduction
- Objectives
- Applications,
- Theories of filtration
- Factors influencing filtration,
- Filter aids, filter medias.
- Principle, construction, working, uses, merits and
- Demerits of plate & frame filter,
- Filter leaf,
- Rotary drum filter,
- Meta filter
- Cartridge filter, membrane filters and seidtz filter.

Introduction

 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.







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- Clarification: When solid are present in very low concentration, i.e., not exceeding 1.0% w/v, the process of its separation from liquid is called clarification.
- Used when contaminating material is finely subdivided, amorphous or colloidal in nature.

Process of filtration



- Once the primary layer is formed further filtration is brought about wherein the filter medium serves only as a support.
- In industrial scale large quantities of suspensions are mechanically handled.
- After a particular point, resistance offered by filter cake is so high that virtually filtration is stopped.
- So positive pressure is applied on the filter cake (upstream) or negative pressure (suction) is applied below the filter medium (downstream).

Applications

- Production of sterile products:
 HEPA filters or laminar air bench
 Membrane filters.
- Production of bulk drugs
- Production of liquid dosage
- Effluents and waste water treatment

Mechanism of filtration

Mechanisms of Filtration



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



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.



Topological entanglement



Cohesional entanglement

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

Types of filtration

Surface/ screen filtration

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



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.



Difference between surface and depth filtration

Surface filtration

- slightly higher than the mean pore size of medium.
- Mechanical strength of filter medium is less, unless it is made of stainless steel
- It has low capacity.
- The size of particles retained is more predictable.
- Equipment is expensive because ancillary equipment such as edge clamps is required.
- Ex. Cellulose membrane filter.

Depth filtration

- The size of particles retained is The size of particles retained is much smaller than the mean pore size of medium.
 - Mechanical strength of filter medium is high.
 - It has high capacity.
 - The size of particles retained is less predictable.
 - Equipment is cheaper because 100 ancillary equipment is not required.
 - Ex. Ceramic filters and sintered filters

Cake Filtration

Cake filtration consists of passing a solid suspension (slurry) through a porous medium or septum (e.g., a woven wire). The solids in the slurry are retained on the surface of the medium where they build up, forming an increasing thicker cake.

Cake Filtration



- Special case of depth filter is the sheet filtration.
- Series of pads are mounted endwise to the flow of fluids.
- Pads consist of mixture of asbestos fibers embedded in cellulose.

Theories of filtration

- 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.



Poiseullie's Equation

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

$$V = \frac{\pi \Delta P r'}{8L\eta}$$

- Where, V = rate of flow, m³/s (l/s)
 - ΔP = Pressure difference across the filter, Pa
 - r = radius of capillary in the filter bed, m
 - L = thickness of filter cake (capillary length), m
 - η = viscosity of filtrate, Pa.s
- If the cake is composed of bulky mass of particles and the liquid flows through the interstice, then flow of liquids through these may be expressed by this equation.

Darcy's Equation

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

$$V = \frac{KA\Delta P}{\eta L}$$

- Where, K = permeability coefficient of cake, m²
 - A = surface area of porous bed (filter medium), m^2

Other terms are same as previous equation

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.

Kozeny-Carman (K-C) equation

Kozeny-Carman equation is widely used for filtration.

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

Where,

 $\boldsymbol{\varepsilon} = \text{porosity of cake (bed)}$

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

K = Kozeny constant (usually taken as 5)

Other terms are same as previous equations

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 through out the bed, but it is sinuous or tortuous.

Factors affecting filtration



Surface area of filter medium

Rate of filtration

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.



Viscosity of filtrate

- According to K-C equation rate of filtration is inversely proportional to the viscosity of the fluid.
- Reason behind 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

- The surface upon which solids are deposited in a filter is called the "Filter medium"
- Properties of ideal filter medium:
- It should-
- be capable of delivering a clear filtrate at a suitable production rate.
- 2) have sufficient mechanical strength.
- 3) be inert.
- 4) retain the solids without plugging at the start of filtration.
- 5) 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

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.

Prefabricated porous solid units

- Used for its convenience and effectiveness.
- Sintered glass, sintered metal, earthenware and porous plastics are used for fabrication.

Membrane filter media

- These are cartridge units and are economical and available in pore size of 100 μm to even less than 0.2 $\mu m.$
- 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.
- <u>Body- mix technique</u>: 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-
- 1. Low flow rate: fine grade filter aids- mainly used for clarity
- 2. Fast flow rate: coarse grade filter aids- acceptable filtrate.

Examples of filter aids

- Diatomite (Keiselgur), obtained from natural siliceous deposits.
- Perlite , it is an aluminium silicate. Cellulose, Asbestos, charcoal, talc, bentonite , fullers earth etc.



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Based on operation of filtration





Selection of filters depends upon below listed factors

Material related

Properties of fluid- viscosity

Nature of solids- size, shape, distribution and packing characteristics of particles

Conc. Of solid in suspension

Quantity of material to be handle

Whether it is necessary to wash the solid

Whether any form of pretreatment will be helpful.

Equipment and process related

Flow rate

Should be absolute in sense, limit to size of particles passing through the filter should be known.

Should be sterilisable by heat, radiation or gas

Should be economical.

28
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.



Assembly of plate and frame filter



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 plate is having individual outlets controlled by valves.



Working

- Working can be divided into two steps-
- 1. Filtration operation
- 2. Washing of cake (if desirable)
- Filtration operation

Frame- marked by 2 dots Plate – marked by 1 dot



Plate and frame filter press, principle of filtration operation

•46





Washing operation

- 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 cannel to the surface of plate.
- The sequence of arrangement of plates and frames can be represented by dots as 1.2.3.2.1.2.3.2.1 so on (between 1 and 1, 2.3.2 must be arranged.



Procedure for washing the press



Things to be noted

- Water wash is efficient only if the frames are full with filter cake.
- If the solid do not fill the frame completely, the wash water causes the cake to break (on the washing plate side of the frame), then washing will be less effective.
- Hence it is essential to allow the frames become completely filled with washing cake.
- This helps not only in emptying the frames but also helps in washing the cake correctly.

Special provisions and Uses

Special provisions:

- 1. 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. 2.
- 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 by obtain 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. 41

Advantages

- Construction of filter press is very simple and a variety of materials can be used.
- Cast iron for handling common substances.
- Bronze for smaller units.
- III. Stainless steel contamination can be avoided.
- IV. Hard rubber and plastic- used where metals must be avoided.
- V. Wood- for lightness though it must be kept wet.
- 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 produce 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 % 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.

Filter Leaf



•56

Working and uses



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.



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.

Diagram



•62



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 are 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: pre filter and actual filter for filtration.
- A protective layer surrounds them.
- The cartridge are 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.

Diagram



Working and uses

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 and Disadvantages

Advantages:

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

Disadvantages:

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

Rotary drum filter

Principle:

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

Construction:

- It consist 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.

Diagram


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 include
- 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 Filter

• Principal:-

It act as a sieve and particulate matter is retained on the surface of the membrane.

Construction:-

- -Made up of cellulose acetate, cellulose nitrate or mixed cellulose esters.
- -These are supported on rigid base of perforated metal, plastic or coarse sintered glass.





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- -These have got high retention characteristics due to pore size, high dielectric constant and surface sensitivity.
- -These are available with different pore sizes.
- If the solution to be filtered contains a considerable quantity of suspended solids, preliminary filtration through a suitable depth filter avoids clogging of the membranes.

• Use:-

- Disc membranes with nutrient broth are used for studying the growth of micro-organisms and colony counts.
- Membrane filters can be autoclaved and used for sterile filtration.

- Advantages:-
- 1. Rapid filtration.
- 2. Negligible adsorption.
- 3. Not prone to fibre contamination and does not impart alkali to the filtrate.
- 4. Available as disposable medium and hence contamination is prevented.
- 5. No bacterial growth on the membrane.
- 6. Membranes are packed in holders or between thick filter pads to prevent curling.
- 7. Pre-sterilised hermetically sealed packs are available.

Disadvantages:-

- 1. Expensive (most refined grades).
- 2. Brittle when dry and can be stored indefinitely in this state. But are tough when wet.
- 3. Filters may get clogged.
- 4. Ordinary types are less resistant to solvents such as chloroforms, polyethylene glycol, certain alcohols, dimethylformamide. esters. ketoses. etc.

Seitz Filter

- Consists of a pad of compressed asbestos as a filtering medium.
- Typical Seitz Filter pads are about 2 mm thick and offered in a range of size.
- Finest pore size gives almost perfect filtration and retains small viruses.
- Filtration occurs by depth filtration.
- <u>Use:-</u>
- 1. For sterile preparation.
- 2 Filtration of viscous soltions.
- 3 Air filtration
- 4 <u>Removes particles due to surface charges and</u> <u>electrostatic attractions.</u>

- Advantages:-
- 1. Rapid filtration with fewer tendency to clog.
- 2. Better than ceramic and sintered glass filters for viscous solutions.
- Disadvantage:-
- Pliable and fragile when wet and should be supported on a perforated metal, plastic, glass disc.
- 2. New pad must be used each time to avoid residue of previous filtration.
- 3. Not suitable for sterilizing products containing alcohol or oil.