SUBJECT NAME & CODE: PHARMACEUTICAL ENGINEERING(BP304T)

UNIT: IV **(FILTRATION)**

CLASS:30

TOPIC: FILTER AIDS, FILTER MEDIAS. PRINCIPAL, CONSTRUCTION, WORKING, USES, MERITS AND DEMERITS OF PLATE AND FRAME FILTER

FILTER MEDIA AND FILTER AIDS

Filter Media

The filter medium acts as a mechanical support for the filter cake and is also responsible for the collection of solids.

Characteristics: Filter medium should have the following characteristics.

1. It should have sufficient mechanical strength.
2. It must be inert, for example, it should not show chemical or physical interaction.
3. It should not absorb dissolved material.
4. It should allow the maximum passage of liquid, while retaining the solids. It means that it must offer low resistance to flow. The resistance offered by the filter medium is not significant in large scale operations and can be neglected.

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 entrances of the channels. Therefore, the pressure should be kept low at the beginning to avoid the plugging of the pores. The usual procedure is to filter at constant rate by increasing the pressure as necessary. When normal working pressure is reached, it is maintained. On continued filtration, the thickness of the cake further builds up and hence the rate of filtration decreases. When the rate is uneconomical, filtration is stopped. The filter cake is removed and filtration is restarted.

Materials: The following materials are used as filter media.

(1*) Woven materials such as felt or cloth*: Woven material is made of wool, cotton, silk, glass, metal or synthetic fibres (rayon. Nylon etc.). Synthetic fibres have greater chemical resistance than wool or cotton, which are affected by alkali and acid, respectively. The choice of the fibre depends on the chemical reactivity with the slurry.

(2) *Perforated sheet metal*: For example, stainless steel plates have pores which act as channels as in case of meta filter (edge filter).

(3) *Bed of granular solid built up on a supporting medium*: In some processes, a bed of graded solids may be formed to reduce the resistance to the flow. Typical examples of granular solids are Gravel, sand, asbestos, paper pulp and kieselguhr. The choice of solids depends on the size of the solids in the process.

(4) *Prefabricated porous solid unit:* porous solids prefabricated into a single unit are being increasingly used for its convenience and effectiveness. Sintered glass, sintered metal, earthenware and porous plastics are some of the materials used for the fabrication.

(5) *Membrane filter media*: Cartridge units are economical and avail- able in pore size of 100 µm to even less than 0.2 µm. These can be used either as surface cartridges or depth type cartridges.

a) *Surface type cartridges*: These are corrugated and resin treated papers. These are used in hydraulic lines. Ceramic cartridges are advantageous in cleaning for reuse by back flushing or firing. Porcelain filter candles are used for sterile filtration.
b) *Depth type cartridges*: These are made of cotton, asbestos or cellulose. These are disposable items, since cleaning is not feasible.

Filter Aids

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

Justification: The objective of the 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.

(a) Usually, low resistance is offered by the filter medium itself. But as the layers of solid built up, the resistance will be increased. The cake may become impervious by blocking of the pore in the medium. Flow rate is inversely proportional to the resistance of the solid cake.
(b) Slimy or gelatinous material and highly compressible substances form impermeable cakes. The filter medium gets plugged and the flow of filtrate stops.

Characteristics: The important characteristics of the filter aids are:

(a) Chemically inert to the liquid being filtered and free from impurities.
(b) Low specific gravity, so that filter aids remain suspended in liquid.
(c) Porous rather than dense, so that pervious cake can be formed.
(d) Recoverable.

Disadvantages: The filter aids remove the coloured substances by absorbing them. Sometimes active principles such as alkaloids are adsorbed on the filter aid. Rarely, filter aids are a source of contaminants such as soluble iron salts, which can provoke degradation of sensitive ingredients. Liquid retained in the pores of the filter cake is lost in the manufacturing process.

Examples of filter aids are:

Kieselguhr Paper pulp Talc Charcoal

Fullers earth Asbestos Bentonite

Activated charcoal is used for removal of organic and inorganic impurities. Kieselguhr is a successful filter aid and as little as 0.1 percent can be added to the slurry. The rate of filtration

is increased by 5 times or more, at the above concentration, though the slurry contains 20% solids.

Handling of filter aids: Filter aids are mostly used for clarification processes, i.e., where solids are discarded. Different flow rates can be achieved depending on the grade of the aids.

Low flow rate (fine solids)-Fine grade filter aids-mainly intended for clarity.

Fast flow rate (coarse solids) coarse grade filter aids-acceptable filtrate.

The filter aid can be employed in either one or both ways.

1. Firstly, a pre-coat is formed over the medium. For this purpose, A suspension of the filter aid is filtered to give a coating up to 0.5 kg per metre square.
2. Secondly, a small proportion of filter aid (0.1 to 0.5% of total batch weight) is purposely added to the slurry. So, the filter cake has a porous structure and filtration can be efficient. The filter aid of 1 to 2 parts per each part of contaminant is mixed in the feed tank. This slurry is recirculated through the filter until a clear filtrate is obtained. Filtration then proceeds to completion. The body mix method minimizes equipment requirements and cross contamination potentials.

CLASSIFICATION OF FILTRATION EQUIPMENT

Equipment are classified based on the *application of external force*.

(a) *Pressure Filters*-Plate and frame filter press and meta filter.
(b) *Vacuum filters*-Filter leaf.
(c) Centrifugal filters

Classification based on the *operation of the filtration*.

(a) Continuous filtration-Discharge and filtrate are separated steadily and uninterrupted.
(b) Discontinuous filtration-Discharge of filtered solids is intermit- tent. Filtrate is removed continuously. The operation should be stopped to collect the solids.

Classification based on the *nature of filtration*.

(a) Cake filters-Remove large amounts of solids (sludge or crystals).
(b) Clarifying filters-Remove small amounts of solids.
(c) Cross-flow filters-Feed of suspension flows under pressure at a fairly high velocity across the filter medium.

These classifications methods help in selecting filtration equipment

INDUSTRIAL EQUIPMENT

In the bulk drug industry, solid is the desired product. Its size, physical properties and purity are important. These factors should be considered, while selecting the equipment and operating conditions. Some of them are:

Materials related:

(a) Properties of the fluid-viscosity.
(b) Nature of the solids - particle size, shape, size distribution and packing characteristics.
(c) Concentration of solids in suspension.
(d) Quantity of material to be handled.
(e) Whether it is necessary to wash the filtered solids
(f) Whether any form of pre-treatment will be helpful

Equipment and process related:

(a) Flow rate.
(b) It should be absolute in the sense, the limit to size of particles passing through the filter should be known.
(c) It should be sterilisable by heat, radiation or gas (examples are ethylene oxide, formaldehyde etc.).
(d) Independently checking the efficiency of filter. It should be economical.

Plate and Frame Filter Press

Principle: The mechanism is surface filtration. The slurry enters the frame by pressure and flows through the filter medium. The filtrate is collected on the plates and sent to the 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.

Construction: The construction of a plate and frame filter press is shown in Figure. The filter press is made of two types of units, plates and frames. These are usually made of aluminium alloy. Some- times, these are also lacquered for protection against corrosive chemicals and made suitable for steam sterilisation.

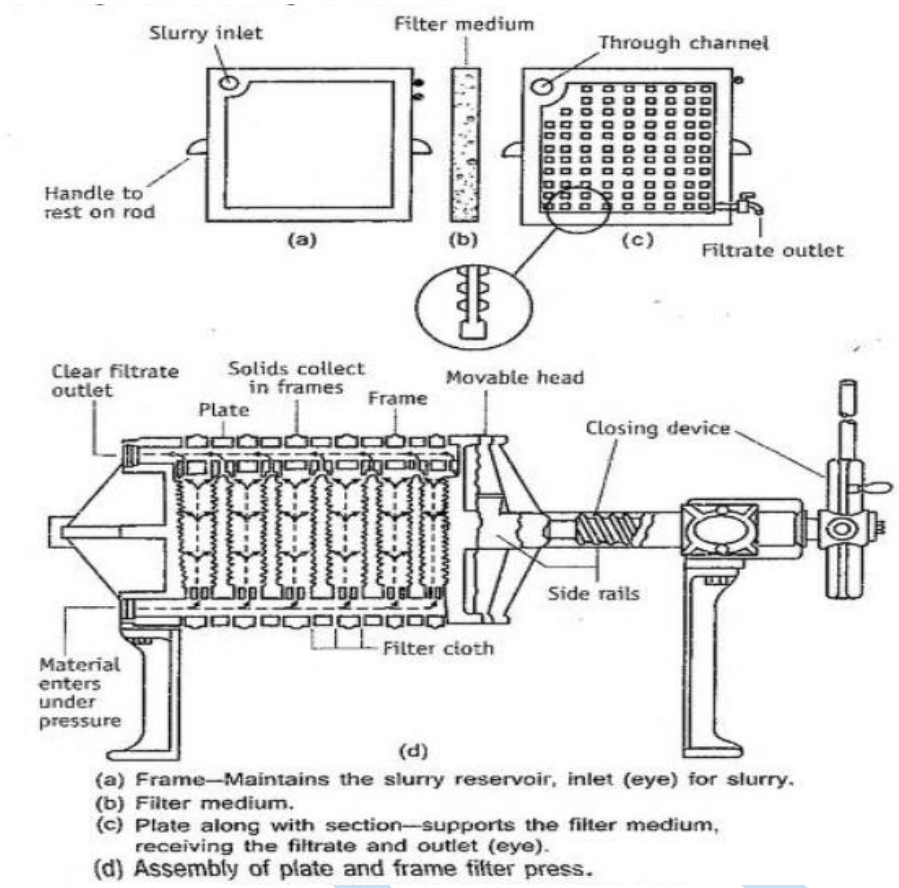


Plate and frame filter press

Frame contains an 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 the description (Figure). The plate has a studded or grooved surface to support the filter cloth and an outlet. It is indicated by one dot in the description (Figure). The filter medium (usually cloth) is interposed between plate and frame.

Frames of different thicknesses are available. It is selected based on the thickness of the cake formed during filtration. Optimum thickness of the frame should be chosen. Plate, filter medium, frame, filter medium and plate are arranged in the sequence and clamped 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 filtration area is as large as necessary. In other words, a number of filtration units are operated in parallel. Channels for the 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: The working of the frame and plate process can be described in two steps, namely filtration and washing of the cake (if desirable).

Filtration operation: The working of a plate and frame press is shown in Figure. Slurry enters the frame (marked by 2 dots) from the feed channel and passes through the filter medium on

to the surface of the plate (marked by 1 dot). The solids form a filter cake and remain in the frame. The thickness of the cake is half of the frame thickness. Because on each side of the frame filtration occur. Thus, two filter cakes are formed, which meet eventually in the centre of the frame. In general, there will be an optimum thickness of filter cake for any slurry, depending on the solid content in the slurry and the resistance of the filter cake.

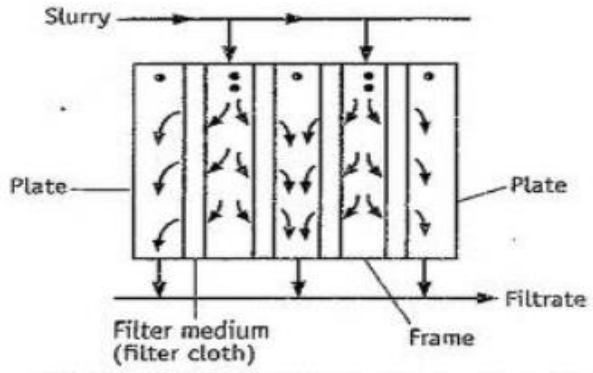


Plate and frame filter press, principle of operation (filtering).

The filtrate drains between the projections on the surface of the plate and escapes from the outlet. As filtration proceeds, the resistance of the cake increases and the filtration rate decreases. At a certain point, it is preferable to stop the process rather than continuing at very low flow rates. The press is emptied and the cycle is restarted.

Washing operation: If it is necessary to wash the filter cake, the ordinary plate and frame press is unsatisfactory. Two cakes are built up in the frame meeting eventually in the middle. This means that flow is brought virtually to a stand still. Hence, water wash using the same channels of the filtrate is very inefficient, if not impossible. A modification of the plate and frame press is used. For this purpose, an additional channel is included (Figure). These wash plates are identified by three dots. In half the wash plate, there is a connection from the wash water channel to the surface of the plate.

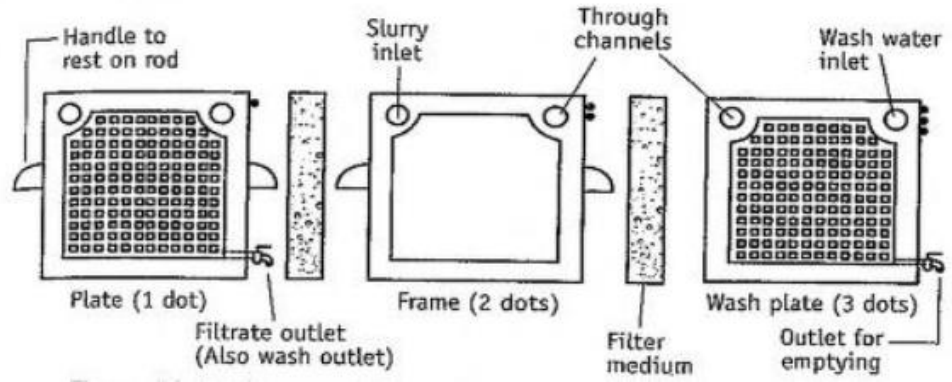


Plate and frame filter press with water wash facility.

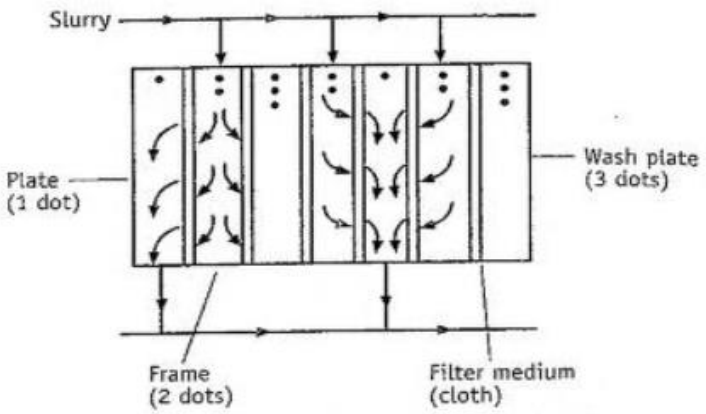
The sequence of arrangement of plates and frames can be represented by dots as 1.2.3.2.1.2.3.2.1.2.3.2.1 so on (between 1 and 1, 2.3.2 must be arranged). Such an

arrangement is shown in Figure (a) and (b) for the operations of filtration and water washing, respectively.

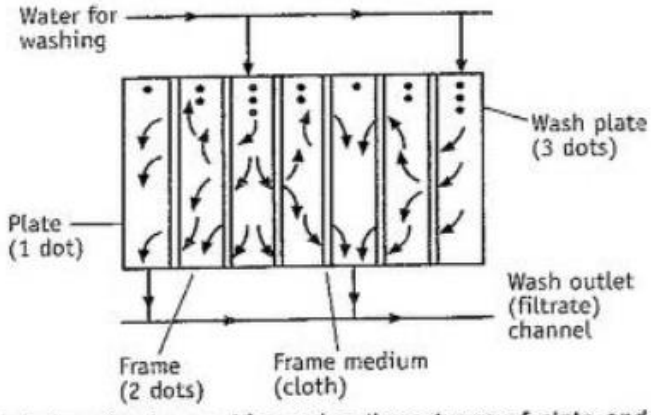
The procedure for washing the press is shown in Figure. The steps are as follows.

1. Filtration proceeds in the ordinary way until the frames are filled with cake.
2. To wash the filter cake, the outlets of the washing plates (three dots) are closed.
3. Wash water is pumped into the washing channel. The water enters through the inlets on to the surface of the washing (three dots) plates.
4. Water passes through the filter cloth and enters frame (two dots) which contains the cake. Then water washes the cake, passes through the filter cloth and enters the plate (one dot) down the surface.
5. Finally washed water escapes through the outlet of that plate.

Thus, with the help of special washing plates, it is possible for the wash-water to flow over the entire surface of washing (three dots) plate, so that the flow resistance of the cake is equal to all points. Hence, the entire cake is washed with equal efficiency.



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



Principles of cake washing using three types of plate and frame.

Plate and frame filter press

Principles of filtration and washing.

It should be noted that water-wash is efficient only if the frames are full with filter cake. If the solids 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 the cake. This helps not only in emptying the frames but also helps in washing the cake correctly.

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 the filtrate. The filtrate goes through the control valve to an outlet channel.
2. The filtration process from each plate can be seen. In the event of a broken cloth, the faulty plate can be isolated and filtration can be continued with one plate less.

Uses: Filter sheets composed of asbestos and cellulose are capable of retaining bacteria, so that sterile filtrate can be obtained, provided that the whole filter press and filter medium have been previously sterilized. Usually, steam is passed through the assembled unit for sterilization.

Examples include collection of precipitated antitoxin, removal of precipitated proteins from insulin liquors and removal of cell broth from the fermentation medium.

Heating/cooling coils are incorporated in the press so as to make it suitable for the filtration of viscous liquids.

Advantages:

1. 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.
    - Stainless steel is used thereby contamination can be avoided.
    - Hard rubber or plastics where metal must be avoided.
    - Wood for lightness though it must be kept wet.
2. It provides a large filtering area in a relatively small floor space. It is versatile, the capacity being variable according to the thick- ness of frames and the number used. Surface area can be increased by employing chambers up to 60.
3. The sturdy construction permits the use of considerable pressure difference. About 2000 kilopascals can be normally used.
4. Efficient washing of the cake is possible.
5. Operation and maintenance is straight forward, because there are no moving parts, filter cloths are easily renewable. Since all joints are external, a plate can be disconnected if any leaks are visible. Thus, contamination of the filtrate can be avoided.
6. It produces dry cake in the form of slab.

Disadvantages:

1. It is a batch filter, so there is a good deal of ‘down-time’, which is non-productive.

2. The filter press is an expensive filter. The emptying time, the labour involved and the wear and tear of the cloth resulting in high costs.
3. Operation is critical, as the frames should be full, otherwise washing is inefficient and the cake is difficult to remove.
4. The filter press is used for slurries containing less than 5% solids. So high costs make it imperative that this filter press is used for expensive materials. Examples include the collection of precipitated antitoxin and removal of precipitated proteins from insulin liquors.

MCQ Each carry 1 mark

1. Which of the following is not a filter aid
    A. Diatomaceous earth
    B. Perlite
    C. Cellulose
    D. Cotton
2. Filter aids are added to the liquid
    A. To increase the porosity
    B. To increase cake permeability
    C. Both A & B
    D. None of above
3. The fluid flows through the filter medium by virtue of
    A. The potential difference across the filter
    B. Pressure difference across the filter
    C. Temperature difference across the filter
    D. Volume difference across the filter
4. Filter aids are mainly used when
    A. The liquid is required as a product
    B. Filter medium is not available
    C. Solid & liquid are required as products
    D. Solids are required as product
5. Which of the following acts as a mechanical support for the filter cake and is also responsible for the collection of solids?
    A. Feed
    B. Filter aid
    C. Filter medium
    D. Funnel
6. The efficiency of filtration increases if,
    A. The compressibility of solids is high
    B. Filter aid is added to the slurry
    C. Filter medium is used
    D. Size distribution of solids is wide in slurry

Each carry 2 marks

1. List the functions of filter aids
2. List the properties of filter aids.
3. Distinguish filtration and clarification.
4. What are filter aids? Give two examples.

5. Explain the mechanism by which filter aids improve the rate of filtration

Each carry 5 marks

1. What are filter aids? Name the filter aids commonly used in pharmacy practice
2. Write about filter aids

Each carry 10 marks

1. With a neat diagram, describe the construction and working of plate and frame filter press

MCQ Each carry 1 mark

1. Filtration is a unit operation that is commonly used for collecting
   - **a) Filtrate**
   - b) Particulate matter
   - c) Precipitate
   - d) Slurry
2. In the clarification process, which factor is more important
   - a) Depth of the media
   - **b) Pore size of the filter media**
   - c) Surface area of the filter
   - d) Volume of slurry
3. Who has proposed that the filtration process is like the streamlined flow of liquid under pressure through capillaries?
   - a) Carman
   - b) Darcy
   - c) Kozeny
   - d) **Poiseuille**
4. Which one of the following is not a mechanism of filtration?
   - a) Entanglement
   - **b) Impact**
   - c) Impingement
   - d) Straining
5. The process of separation of insoluble particles from suspension or slurry is called
   - **a. Filtration**
   - b. Sieving
   - c. Distillation
   - d. Drying
6. The separation process in which the amount of solid in a liquid is not more than 1% w/v is called
   - **a. Clarification**
   - b. Filtration
   - c. Centrifugation
   - d. Evaporation
7. Which of the following does not influence filtration?
   - a. Temperature
   - b. Density
   - c. Viscosity

d. pH

8. The rate of flow of the filtrate through the filter cake is……to the thickness of the cake
   - **a. Inversely proportional**
   - b. Directly proportional
   - c. Remain constant
   - d. None of above
9. Which of the following theory not describe the rate of filtration?
   - a. Darcy Law
   - b. Poiseuille's equation.
   - c. Kozeny Carman equation
   - **d. Noyes Whitney equation**
10. The slurry is
   - a. **A suspension to be filtered**
   - b. A porous membrane used to retain the solids
   - c. The solids which are present on the filter
   - d. A clear liquid passing through the filter.
11. The correct definition of filtration is:
   - **a. When solids are present in a very low concentration, i.e., not exceeding 1.0% w/v, the process of its separation from liquids**
   - b. When solids are present in a very low concentration, i.e., not exceeding 5.0% w/v, the process of its separation from liquids
   - c. When solids are present in a very low concentration, i.e., not exceeding 2.0% w/v, the process of its separation from liquids
   - d. When solids are present in a very low concentration, i.e., not exceeding 7.0% w/v, the process of its separation from liquid
12. In the equation of Poiseulle's & Konzeny's Theory, "V" stands for
   - a. Velocity
   - b. Viscosity
   - **c. Rate of filtration/flow**
   - d. None