Module-26: Fermentation Product Recovery and Purification-II: *Filtration & Centrifugation*

Filtration

- Filtration is one of the most commonly used and effective processes for separation of solid-liquid with different types of filtration systems.
- The process uses a porous medium that allows the passage of gas or liquids but not the solid material.
- It is amenable to scale up.
- But the following factors influence the choice of the most suitable apparatus for specific requirement with evident cost effectiveness.

1. Viscosity and density of the filtrate
2. Shape, size, distribution and packing characteristics of the solid particles
3. Ratio of solid & liquid
4. Operational Scale
5. Type of material required to be recovered (solid or liquid or both)
6. Mode of operation: continuous or batch
7. Need for additional attachment if necessary for vacuum suction
8. Necessity for low temperature

Theories of Filtration

- A simple filtration device consists of a filtration cloth supported by a porous material.
- When the broth passes from the filter cloth, the solid material is retained on it which forms a cake.
- Due to the continuing deposition of solid on the cloth, the thickness of cake increases that result in a resistance in the flow.
- To make the rate of flow continuous, pressure has to be applied on the cloth.
- The pores of the filtering cloth may be closed sometime due to the blocking or the flow may be clogged due to the compression of the particles.

- In that case pressure cannot be applied for filtration especially when the particles are compressible.

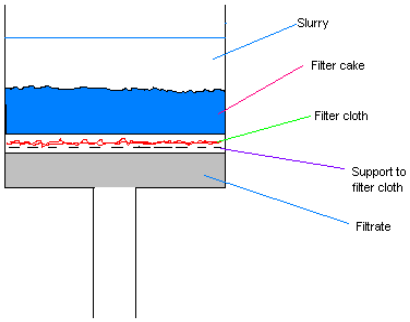

Diagram of a simple filtration

- Flow through a uniform and constant depth porous bed can be presented by the Darcy's equation
- **Rate of flow =** $\frac{dV}{dt} = \frac{KA\Delta P}{\mu L}$

  Where, μ = liquid viscosity

  L = depth of the filter bed

  ΔP = pressure differential across the filter bed

  A = area of the filter exposed to the liquid

  K = constant for the system
- *K* depends on the specific surface area *s* (the surface area / unit volume) of the particles that make up the filtration bed and the voidage (Σ) when the particles are packed together.
- The voidage is defined as the fractional volume of the filter bed that is available for the liquid to pass through.

Use of Filter Aids

- Filter aid is generally used when the filtering material blocks the filter medium (filter cloth) due to bacterial broth or presence of some gelatinous material.
- Diatomaceous earth material (kieselguhr) is the most commonly used filtration aid.

- It has a very high voidage (approximately 0.85) and the porosity of the material increases when it is mixed with the cell suspension.
- On the other hand, the filter aid should be added as less as possible to minimize the absorption of the filter material.
- It may be used as an initial bridging material in the wider pores of a filter medium to stop or reduce the blinding of the pores.
- In order to attain an efficient filtering process using the filter aids
  i) a thin layer of the filter aid is applied on the filter medium to form the precoat before the filtration of the broth,
  ii) before starting the filtration, to build up a satisfactory filter bed an appropriate quantity of filter bed is mixed with the harvested broth and after building up of the bed, the raffinate is returned back to the remaining broth before starting the actual filtration (In case of vacuum drum filter, fitted with an advancing knife, a thick precoat filter is built-up on the drum).
- Generally, filter aids are used in case of intracellular products which facilitates further purification stages.
- Filter aids cannot be used in case of microbial biomass production and for those cells where pretreatment procedures like flocculation or heating are considered.

Different types of filtration systems

Batch filtration

1 Plate and frame filter:

- It is a type of pressure filter.
- It consists of alternately arranged plates and frames where frames are fitted with filter cloth or filter pad.
- The assembly of filters and frames are joined together by a flat frame and forms a sequence of liquid tight components.
- When liquid passes through the assembly, it flows through the grooves and then discharged through an outlet device.
- In industrial scale this is the economic filtration process and required less area.

- The main drawback of this process is the frequent deterioration of the filtering cloth in batch operations due to its frequent disassembling.
- This system finds wide application for clarification in the breweries.
- It is also used for accumulating high value solids for which the use of a continuous filtration apparatus is not suitable.

2 Pressure leaf filter:

- These filters contain metal plates termed as leaves with metal framework of lined plates.
- These are covered with an adequate wire web or sometimes a filter cloth, rarely are pre-coated with a cellulose fiber covering.
- The slurry to be filtered is fed in to the filter by pressure or by suction.
- The system can be steam sterilized.

Types:

1) Vertical metal-leaf filter:

- This filter contains a cylindrical pressure vessel having a horizontal hollow shaft on which various vertical porous metal leaves remain fixed.
- When slurry fed into the filter, solids from the slurry progressively deposited on the leaves while the filtrate is removed via the hollow shaft which sometimes remain gently rotated.
- At the end of a cycle, aggregated solids are removed by propelling air through the shaft and into the filter leaves.

2) Horizontal metal leaf filter:

- This filter contains a cylindrical pressure vessel having a vertical hollow shaft on which metal leaves remain fixed.
- Frequently, only the upper surfaces of the leaves are porous.
- Filtration is continued until the cake blocks the space between the leaves or when the working pressure has become extreme.
- The solid cake can be cleared at the end by liberating the pressure and rotating the shaft with an energy motor.

3) Stacked leaf filter:

- One kind of filter of this type is the Meta filter.

- This is a very robust device and because there is no filter cloth and the bed is easily replaced, labor costs are low.
- It consists of a number of precision made rings which are arranged on a grooved rod. The rings are aggregated on the rods.
- The stacks are located in a pressure vessel, which are usually covered with a thin layer of filter aid like kieselguhr.
- As filtrate passes between the discs, it removed through the channels of the grooved rods, while solids are deposited on the filter covering.
- Filtration is continued until the resistance becomes excessive.
- Solids are removed from the rings by applying back pressure via the grooved rods.
- Meta filters are primarily used for 'polishing' liquids such as beer.

Continuous Filtration

1 Rotary vacuum filter:

- The filter consists of a hollow rotating drum which remains internally segmented.
- A piece of fabric or metal filter forms cover around drum and drum remains dipped in a trough containing the liquid to be filtered.
- Vacuum is given into the drum and the slurry is fed in to the container from outside.
- As vacuum is given, filtrate is drawn into the drum and via the compartments collected in a vessel.
- Air pressure is used to help the release of the filter cake.
- Residual broth is washed out from the cake by water jet.
- In the industries this filter is used for the filtration of huge volume of liquids in a continuous process.
- This process is generally applied for the separation of cells from the broth but at the same time disrupted cell slurry can also be efficiently removed from the liquids.
- A number of rotary vacuum filters are available. These are:
  - String discharge
  - Scraper discharge
  - Scraper discharge with precoating of the drum

1. ***String discharge:***
   - This is used to separate fibrous filter cake produced by fungal mycelia from the drum.
   - Long strings (1.5 cm) are threaded over the drum and round two rollers.
   - When the vacuum pressure is released, the cake is released from the upper part of the drum and passed to the small rollers where it drops free.
2. ***Scraper discharge:***
   - This is used to collect yeast cells on a filter drum by knife blade.
   - The filter cake formed on the drum is detached by precisely situated knife blade.
   - The filter cloth may be blocked by cells (bacteria/ mycelia of actinomycetes).
3. ***Scraper discharge with precoating of the drum:***
   - This filter allows precoating of the drum with a layer of filter-aid (2- 10 cm thick) to avoid problem of blocking of filter cloth.
   - The cake which deposited on the drum is cut by the knife blade.
   - In 1958 Bell and Hutto made a study of precoat drum filtration.

Cross flow Filtration (Tangential flow filtration)

- This filtration system is consist of:
  1. a media storage tank,
  2. a pump and
  3. a system of packs of membrane.
- This filtration is also known as tangential flow fermentation as the flow of medium to be filtered is tangential to the membrane.
- To increase surface area of the membrane, it is usually packed in a spiral cassette or a cartridge of hollow strings.
- The broth is forced across the membrane surface, most of the bulk material sweeps the membrane and returns to the main tank and less than 10% pass through the membrane as permeate.
- As the process continues, the volume of the original feed reduces to 5-10% of their initial volume.
- Two types of membranes can be used depending on the need of the process;

1. microporous membrane with a specific pore size (0.45 μM or 0.22 μM) or
2. an ultrafiltration membrane with a specific molecular weight cut-off.

- There are various benefits of this system like,

1) The process is highly efficient and > 99.9% cell retention can be achieved.
2) This is a closed system which will not form aerosol.
3) The separation process is not dependent on media and cell densities.
4) Addition of filter aid is not required.

- The system can be sterilized with minute modification.
- Increased pressure drop raises the rate of filtration.
- But pressure drop should not be much high otherwise it will lead to the blockage of the membrane.
- Therefore, to achieve the best results higher flow rate is recommended.
- Higher temperature increases the flow rate by lowering the viscosity of the system.
- Membrane may get clogged by the fowl material present in the medium.
- This can be controlled by modification of the constituents of membrane or modifying the media, like, minimizing the addition of antifoam reagent.
- The flow through a bed having a constant and uniform depth can be represented by Darcy's equation.

Centrifugation

- Centrifugation is used for a very high degree of solid separation when separation is not satisfactory by filtration.
- The main principle of centrifugation process is the sedimentation under centrifugation force.
- Centrifuge works using the sedimentation principle, where the centripetal speed causes separation of denser substances along the radial direction.
- According to the Stokes law, for a spherical solid suspended in a liquid of Newtonian viscosity, the rate of sedimentation is proportional to the square of the diameter of the particles.
- Thus the rate of sedimentation of a particle under gravitational force can be expressed as:

$$V_g = \frac{d^2 g (\rho_P - \rho_L)}{18 \mu}$$

Where, V_g = rate of sedimentation

d = particle diameter

g = gravitational constant

ρP = density of the particle

ρL = density of the liquid

μ = viscosity

- The equation can be modified for sedimentation under the centrifugal force as:

$$V_g = \frac{d \omega^2 r (\rho_P - \rho_L)}{18 \mu}$$

Vc = rate of sedimentation under centrifugation force

ω = angular velocity of the rotor

r = radial position of the particle

- Dividing 2nd equation by 1st equation yields,

$$\frac{\omega^2 r}{g}$$

- This equation is a measure of sedimentation power of centrifugal force compared to the gravitational force and is referred as the relative centrifugal force (RCF) and denoted by the symbol *Z*.
- From the above expression it is evident that other factors like viscosity difference between the cell mass and the liquid has little role to play in the rate of sedimentation.
- It is true that with increasing temperature the viscosity of liquid changes, but since the viscosity difference of the particles and the liquid is very less, the effect can be neglected.
- Therefore the diameter of rotor and the angular velocity of the rotor are the main factors to be considered while maximizing sedimentation by centrifugation.
- Generally, in the laboratory batch centrifugation with small volumes can be used, but they are of little practical use in the industry.
- In industries centrifuges are used in continuous or semi-continuous mode.

Different Types of Centrifuges

- For the industrial application different types of centrifuges are available with different dimensions and modes of application.
- They differ mainly in their mode of operation, capacities, speed, mode of loading and discharging.
- The ultimate choice of the type of centrifuge depends on the type of application and effectiveness.

Basket centrifuge (perforated-bowl basket centrifuge)

- Contains perforated bowls with a filter bag of nylon or cotton.
- Liquid is fed in continuous mode and it is operated at not more than 4000 rpm.
- Solid biological mass can be washed before removing the bowls.
- Rate of feeding is 50-300 dm^3/min and have a solid holding capacity of maximum 500 dm^3
- Used for the separation of mycelial mass, moulds and crystalline compounds.

Tubular-bowl centrifuge

- The main component of this system is a cylindrical bowl which may be of variable design depending on their application.
- The system remains suspended by a shaft which is flexible and rotates by a motor fitted overhead.
- The inlet is at the bottom fitted with a nozzle which enters through the bottom bearing.
- The inlet consists of solid and a liquid phase; the liquid generally contains a light and a heavy phase.
- The system is applied for the separation of particles having dimension of 0.1μm to 200 μm in diameter and for a liquid having solid load of only 10% or less.
- It can be applied for

1) light phase/heavy phase liquid separation
2) for the separation of solid/light phase liquid/ heavy liquid phase and
3) only for solid/liquid separation.

- This solid particles sediment on the wall of the rotor and the two liquid phases get separated into two distinct zones.
- At the exit they are kept separated by the adjustable lips of the rings which may be of various sizes.
- System can be operated at a speed of 50,000 rpm.
- Limitations are limited solid holding capacity, loss of efficiency etc.

Solid-bowl scroll centrifuge (Decanter centrifuge)

- The main part of this system is a horizontal rotating solid bowl.
- Slurry is fed to the bowl through the spindle of an Archimedean screw within the bowl.
- Solid is separated on the wall of the bowl and is scraped up to the conical end of the bowl.
- The slope of the bowl is so set that the excessive liquid is drained out from the solid.
- Liquid is discharged at the other end of the bowl.
- The system is used in continuous mode for the separation of solids from the fermentation broth.

- The bigger version can be applied at a speed of maximum 5,000 rpm; smallest versions can be applied sometimes at a speed of 10,000 rpm.
- The system is available for various applications

1) with the facility of cake washing,
2) with vertical bowl decanting facility
3) with the facility of in-situ cleaning, and
4) the facilities for the containment of biohazards.

Multi chamber centrifuge

- This system consists of multiple chambers mounted within the chamber of the rotor.
- The slurry is fed in to the chambers through a system of spindles and travels through the system of chambers through a circuitous route.
- At the outer face of each chamber the solid is collected.
- The system is applied for the separation of particles having dimension of 0.1μm to 200 μm in diameter.
- Though the system is having greater solid handling capacity and there is virtually no loss of efficiency but its mechanical stability limits its larger application and speed maxima (6,500 rpm)

Disc-bowl centrifuge

- This system consists of a central inlet pipe and a system of conical disc, made-up of stainless steel arranged in stacks with a spacer.
- The broth to be separated is injected in to the system through a central pipe and then it flows outward toward the discs.
- There it flows upward and inward in between the disc at an angle 45^{o} with the axis of rotation.
- Within the closed system the solid sediments rapidly, accumulating on the inner wall of the bowl.
- The sediment formed is not slid in this case, forms slurry which flows and can be discharged continuously.

- The system is highly efficient, has capacity of high volume liquid handling, easy removal of solid and *in-situ* cleaning facility.
- Solid can be removed by automatic opening of the solid collection bowl.
- Feed rate is as high as 1800 dm3/min and rotational speed ranges from 5000 rpm to 10000 rpm.

Applications

- Centrifugation is one of the preferred methods of clarification of wine, removal of solids from the fermented broths etc.
- In this, high speed rotation impels suspended material out of the wine.
- Speed of clarification is the primary advantages of centrifugation.
- It is especially useful when early bottling is desired.
- Centrifugation is also particularly valuable with very turbid wines.
- They frequently produce off-odors during spontaneous clarification and typically clog filter units.
- Centrifugation also increases the efficient use of polishing filtration by removing the most remaining suspended material.

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