PHARMACEUTICAL ENGINEERING

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CENTRIFUGATION

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❖ Centrifugation is the process in which separation of particles take place by using centrifugal force

❖ It is used for separating either two immiscible liquids or a solids from a Liquid.

❖ It is widely used or helpful when filtration process is not applicable

❖ The equipment used for separation is known as centrifuge

PRINCIPLE :

➢ The centrifugation involves the principle of sedimentation.

➢ We apply a centrifugal force to achieve separation by a difference in density.

➢ The particles have a size more than 5µm are separated by simple filtration process. while the particles having size 5µm or less do not sediment under gravity, the centrifugal force is used to separate them.

➢ Centrifugal force generated due to rotation which acts radially outwards

APPLICATIONS OF CENTRIFUGATION

- Centrifugation is used for separation of blood cells from blood.
- Used for Separating drug present in blood, urine and tissue fluids.
- It is widely used in bulk drug industry to separate Crystalline material from suspension
- It is used to obtain pure form of insulin by selecting precipitating other fraction of protein and separating them by ultracentrifugation.
- Ultracentrifugation is used for separation of virus particles which are utilized in industrial applications

EQUIPMENT FOR CENTRIFUGATION

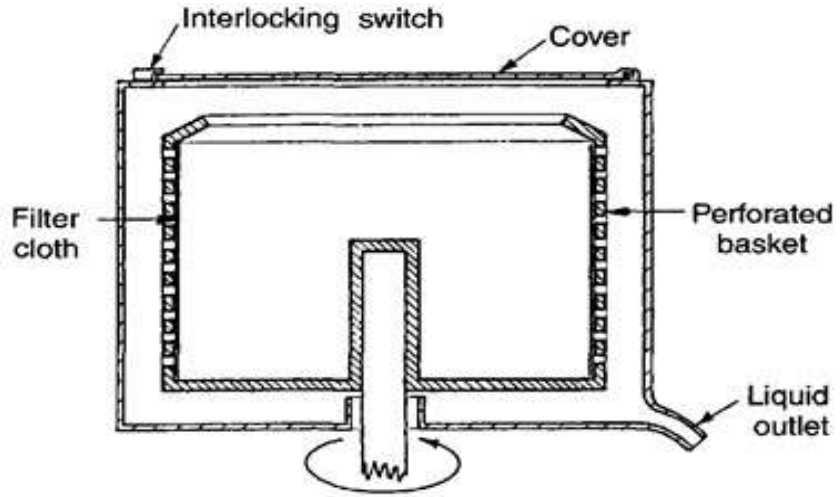
1. Perforated Basket Centrifuge
2. Non-perforated Basket Centrifuge
3. Semi continuous Centrifuge
4. Super Centrifuge

PERFORATED BASKET CENTRIFUGE

PRINCIPLE

Separation occurs through perforated Wall depend on the difference in densities of solids and liquid phases.

CONSTRUCTION



- It consists of a basket made up of steel or any other suitable metal
- It is perforated with filter cloth.
- The basket is suspended on vertical shaft and Driven by motor.
- The basket is Surrounded by Casing which collect the filtrate and discharge through the outlet.
- The diameter of the basket is 0.9 miter.
- The diameter of the perforation depends on the crystal size
- The basket operated at a speed of 1000 rpm.

WORKING

- The material is loaded into the basket when it is in stationary
- Power is applied to rotate 5kw then power reduced to 2 kw (speed 1000RPM)
- The Liquid passes through a perforated wall while Solid retains in the basket.
- Liquid is collected through casing, centrifuge is stopped by applying brake
- Now Unload the solid.

USES

1. Used to separate crystalline drugs (aspirin etc..) from mother Liquor.
2. Used to separate precipitated protein from insulin.

ADVANTAGES

1. It occupies little space.
2. Rapid process
3. Used when solid concentration in slurry is high.

DISADVANTAGES

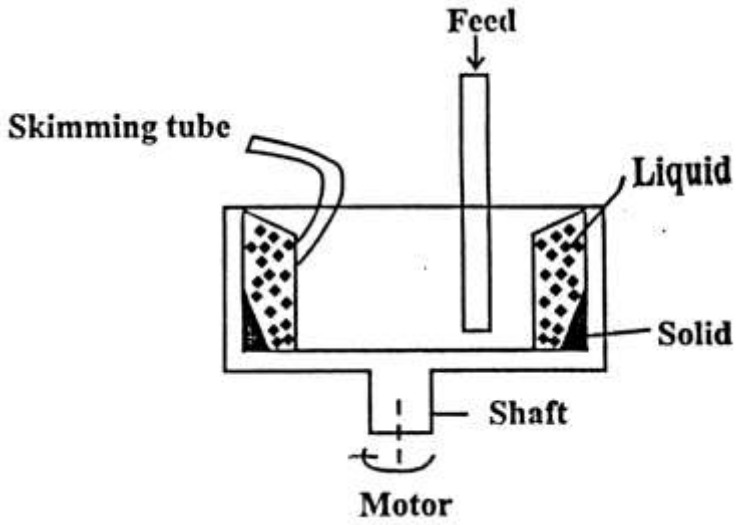
1. Labor cost is high
2. Batch process

NON-PERFORATED BASKET CENTRIFUGE

PRINCIPLE

The separation of solids and liquids phase depends on difference in densities of both phases without a porous barrier [sedimentation]

CONSTRUCTION



- It consists of a non-perforated basket made of steel.
- The material is loaded into basket through feed tube
- The basket is mounted on rotated by motor.
- The Liquid is removed with the help of skimming tube.

WORKING

- The Suspension is fed into the basket continuously through feed tube
- During centrifugation solid deposited at side of basket while Liquid remaining at top Skimming tube
- When sufficient amount of solids get deposited at the site of basket then it is removed intermittently by hand and continuously by Scraper blade

USES

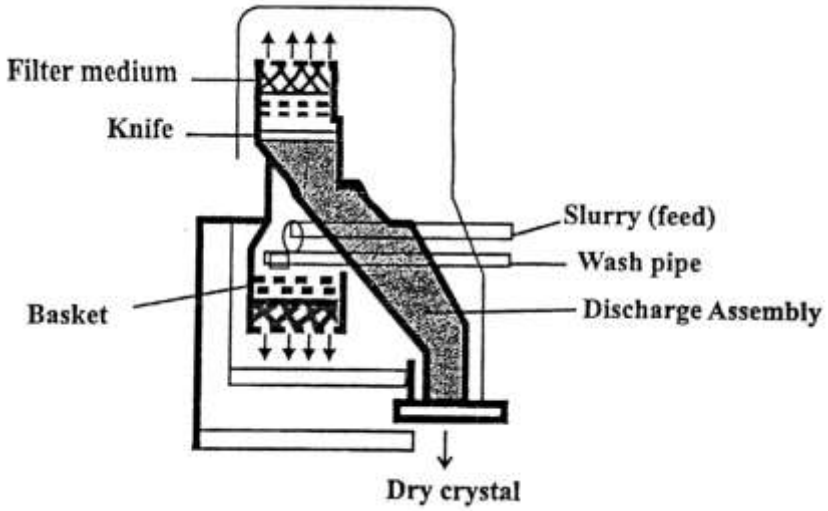
Non perforated basket centrifuge is useful when the deposited solids offer high resistance to the flow of Liquids.

SEMI CONTINUOUS CENTRIFUGE

PRINCIPLE

Separation occurs through perforated wall depends on the difference in the density Solid and liquid phases.

CONSTRUCTION



- It consist of perforated basket mounted on horizontal shaft and run Continuously by motor.
- Feed introduced through feed pipe and wash the crystal through wash pipe.
- The layer of Cake is removed by a knife through discharge assembly.

WORKING

- The basket is rotated horizontally with motor.
- Suspension (slurry) introduced through feed pipe
- The Liquid (filtrate) is eliminated passed through perforated site of basket and solid residue (cake) remain inside the basket.
- When the height of cake is approx. 2-3 inch, the slurry inlet is stopped by "feeler diaphragm valve assembly"
- Then Cake washed with water.

After all that , hydraulic apparatus raises knife to cut the Cake which collected through outlet

ADVANTAGE

Used to separate crystal from mother liquor. Used to clarify Liquid removing unwanted Solids from oils.

DISADVANTAGES

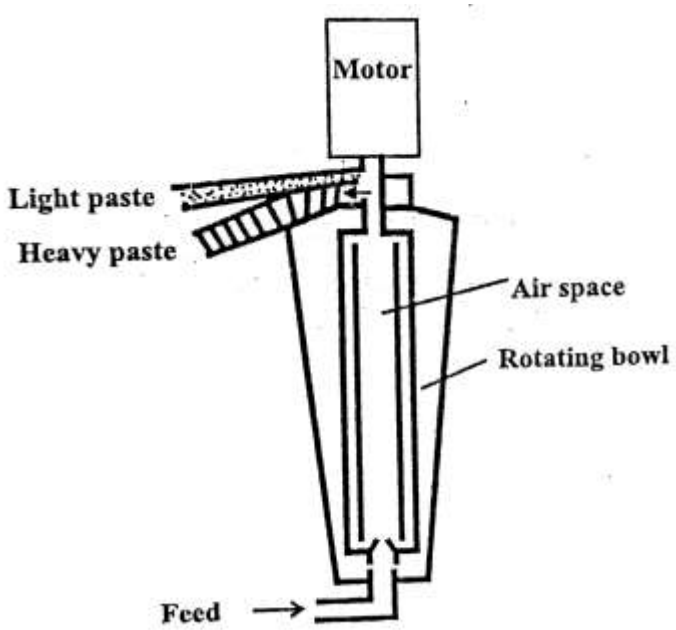
1. Complicated process.
2. High power consumption.

SUPER CENTRIFUGE

PRINCIPLE

It is based on the principle of sedimentation and it is used to separate two immiscible liquid phases.

CONSTRUCTION



- It consists of a long, hollow, cylindrical bowl of Small diameter and rotate on vertical axis.
- Feed is introduced through the bottom through a nozzle with pressure
- Two Liquid outlet are provided at different height
- It rotate approx 2000 rpm

WORKING

- It rotates at 2000 rpm on its axis and then feed introduced through bottom nozzle with pressure
- Two Liquid phases were separated acc. to their density , the heavier liquid moves toward the periphery and Lighter Liquid forms an inner layer.
- Both liquids ascend to the upper part of bowl. these removed separately from different height through modified outlets.

USE

Used for separating liquid phases of emulsion in food, bio chemical and pharmaceutical industries

THANK YOU

DRYING

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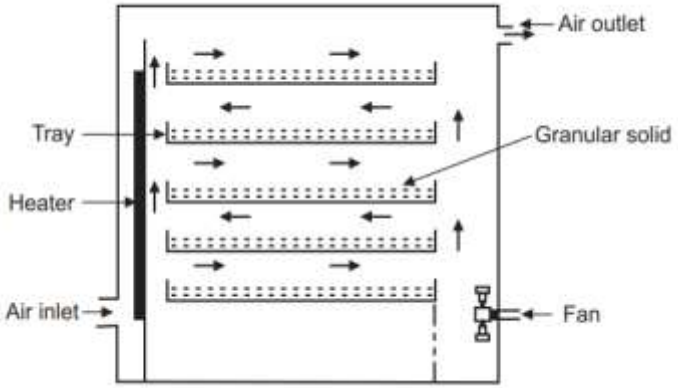
TRAY DRYER

Principle:

In this process, there is no relative movement among the solid particles being dried. It is operated in batch process. This type of dryer operates by passing hot air over the surface of a wet solid that spread over trays arranged in racks and moisture is removed.



Construction:

It consist of chamber whose wall is insulated by thick glass wool or suitable heat insulating material. Inside the chamber there are various trays on which wet materials are placed. The number of trays depend on the size of chamber. For laboratory purpose, minimum 3 trays are required. There should be appropriate distance between upper, middle and lower tray. The dryer is also attached to provision of fresh air inlet and air outlet. The fan in dryer is used to circulate air over the material placed in trays. Heater is also provided inside to facilitate uniform heating. The door of the heater is explosion proof and is locked with the help of spring with suitable pressure. Door lips are having Neoprene rubber gasket to prevent leakages.

Working:

Wet material are spread into trays and placed into the chamber. The doors are closed. The air is introduced through inlet and through heater air heated up. Hot air is circulated by fan over the tray. There should be uniform air flow otherwise proper drying will not be achieved. Even fan are used to distribute the air uniformly over the trays. Some moist air is continuously vented through outlet. The fresh air enters through the inlet. The water evaporate from the interior of the solid due to hot air. At the end, trays an removed from chamber and dried material is collected.

Advantages:

1. Tray Dryer is the most conventional dryer used very widely and still being used where the moisture content is more and where the product has to be dried at low temperature for long hours.

2. It is a batch process and can also be used where quantity of material is less.

3. In the tray dryers the handling of the materials can be done without losses.

4. Valuable products can also handled effectively.

Disadvantages

1. Not suitable for thermolabile substances.

2. The process takes long time.

Pharmaceutical Applications

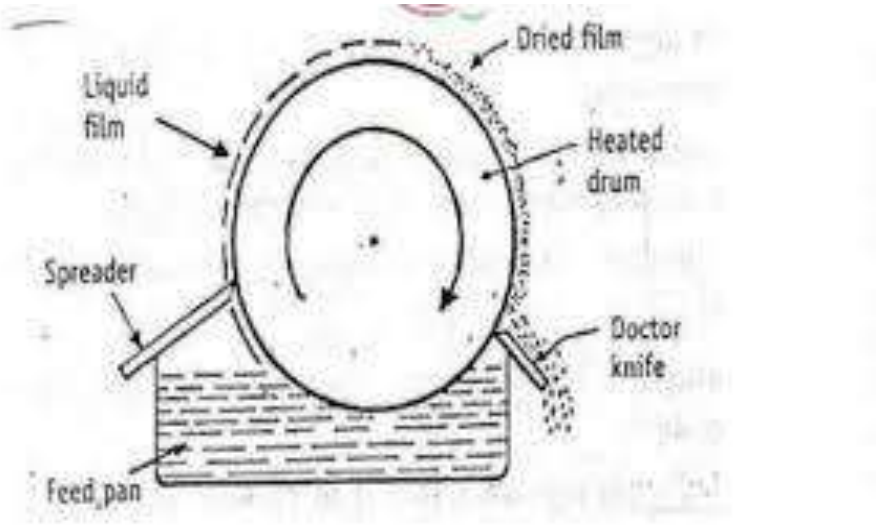
1. Tray dryer is used in the drying of the sticky materials.

2. Tray dryers are used in the drying of the granular mass or crystalline materials.

DRUM DRYER

Principle-

The drum dryer is an equipment used to convert the solutions and suspensions into the solids. The main purpose is to spread the liquid to a large surface area so that drying can occur rapidly.



Construction-

It consists of hollow steel drum of about 0.75-1.5 m in diameter and 2-4 m in length. It is heated internally. usually by steam, and rotated on its longitudinal axis. The external surface of drum is polished. Liquid or slurry is placed as feed in a pan. The drum is partially dipped in pan. The spreader is used to spread liquid film evenly on roller. The rotation of the drum adjusted so that all of the liquid is fully vaporized. The drum is rotated continuously. The dried deposit or material can be scrapped off with the help of knife. The dried material is collected in storage bin.

Working:

As drum rotates, the liquid material get adhere to external surface of drum. The liquid is spread as film onto the surface. The drying of the material is done by the process of steam when passed in to the drum. By the mechanism of the conduction the heat get transferred in to the drum and drying process takes place. The materials is completely dried during the whole process during its revolutions. The dried materials is scrapped by the knife and that falls in to the bin.

Advantages

1. Drying take place in less time.
2. It is suitable for thermo sensitive drugs.
3. It occupies less space.
4. Rapid drying takes place due to rapid heat and mass transfer.

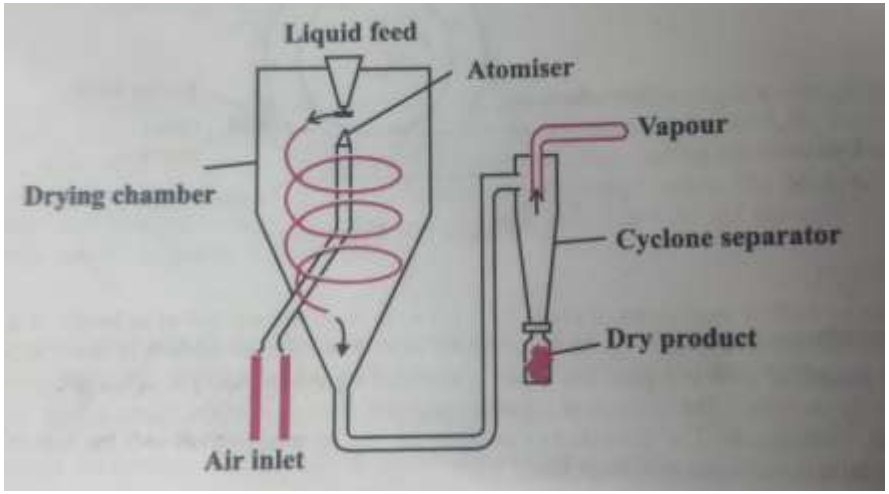
Disadvantages

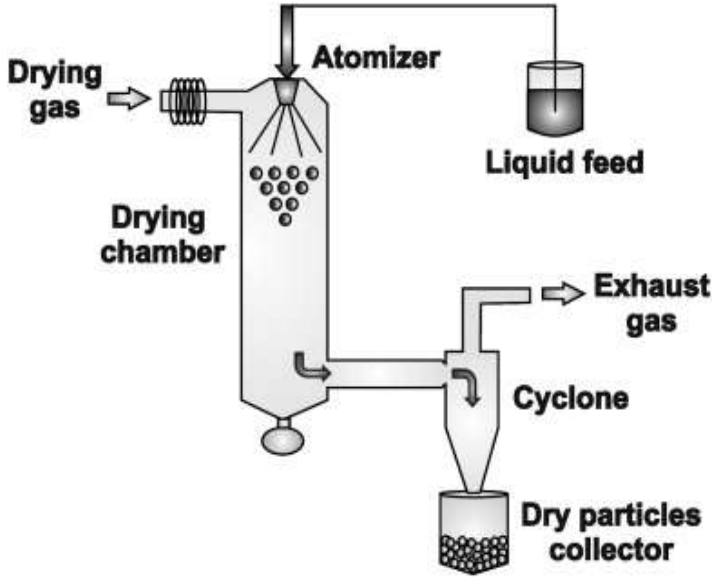
1. Maintenance cost is high.
2. Skilled operators are essential to control thickness of the film.
3. It is not suitable for less solubility products.

SPRAY DRYER

Principle:

Spray drying is an industrial process for dehydration of a liquid feed containing dissolved and/or dispersed solids, by transforming that liquid into a spray of small droplets and exposing these droplets to a flow of hot air.





Construction

It consist of drying chamber having conical base. It is made of stainless steel. The inlet for hot air is provided at bottom and another inlet for spray disk atomizer at the top. Atomization may be achieved by means of nozzles. The drying chamber is connected to cyclone separator. The dry product is collected from the bottom of cyclone separator

Working

The spray-drying process can be divided into four sections

atomization of the fluid,
mixing of the droplets
drying
removal and collection of the dry particles

The feed entered the drying chamber through the atomizer . Hot air passes through the inlet. The air temperature should be adjusted in such a way that the droplet dries completely before reaching the wall of the drying chamber. The residence time of a droplet in the dryer is only a few seconds (5-30 s). The particle should not be overheated. The bottom of drying chamber is connected to cyclone separator. In cyclone separator, the centrifugal force throw out the dried particles to the bottom and dried particle are collected in a bin. The vapors are removed from the top.

Advantages

1. The process of drying completes within 3 to 30 seconds.
2. Less labor costs required as it is continuous process
3. Uniform and controlled size product can be obtained.
4. The solutions or suspensions can be dried easily

Disadvantages

1. The spray dryer is expensive.
2. Difficult to operate.

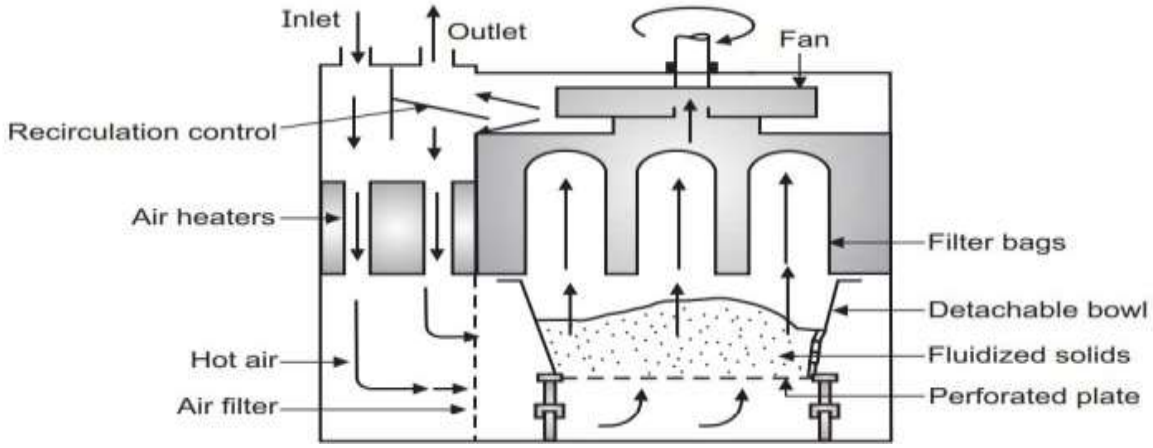
Pharmaceutical Applications

1. Spray dryer can be used for drying both solution or suspension.
2. Spray dryer are very useful for the drying of heat sensitive substances.

FLUIDIZED BED DRYER

Principle

In the fluidized-bed dryer, hot air is passed through a perforated bottom of the container containing granules to be dried. The granules are suspended in the air stream and rise from the bottom. This condition is called a fluidized state. Hot air surrounds each granule to dry it completely. Therefore, the materials or granules are dried uniformly.



Construction

The fluidized bed dryer consists of a steel shell of cylindrical or rectangular cross section. A detachable bowl which has perforated bottom is placed on the bottom of the dryer. It is used to load and unload the materials. The bowl is used to place the materials to be dried. A fan is mounted on the top to circulate hot air. The fresh air inlet and the heat exchanger are connected in series to heat the air to the required temperatures. The temperature of the hot air and the exhaust air are monitored. The bag filters are placed on top of the drying container for the recovery of the fines. The air flow is adjusted by means of the recirculation control. The bags are provided to prevent the passage of the fine particles.

Working

The wet granules to be dried are placed in a detachable bowl. The bowl is inserted in the dryer. Fresh air can pass through a inlet which is then heated when passing through a heat exchanger. Hot air flows through the bottom of the bowl. At the same time, the fan starts to rotate. The air speed increases gradually. When the velocity of the air is greater than the sedimentation rate of the granules, the granules remain partially suspended in the gas stream.

After a specific times, a pressure point is reached in which the friction drag on the particles is equal to the force of gravity. The granules rise in the container due to the high gas velocity of 1.5 to 7.5 m per minute and then fall back. This condition is said to be fluidised state. The gas each granule to dry them completely. The air comes out of the dryer passing through the filters in the bag. The entrained particles remain adhered to the interior of the surface of the bags. Periodically, the bags are shaken to remove entrained particles.

The residence time for drying is approximately 40 min. The materials are sometimes left in the dryer to reach room temperature. The bowl is removed for unloading. The final product is free flowing.

Advantages

1. Drying is achieved at constant rate.
2. It is available in different sizes with the different drying capacity
3. Drying capacity is more than other dryer.

Disadvantages

1. Many organic powders develop electrostatic charges during drying.
2. Chances of attrition of some materials resulting in the production of fines.

Pharmaceutical Applications

1. It is used for the drying of the granules in the production of the tablets.
2. It is used for coating of granules.

VACUUM DRYER

Principle

In vacuum dryer, the materials are dried by vacuum. Due to vacuum, there is decrease in pressure. Therefore water boils at a lower temperature and then the evaporation of water takes place faster.

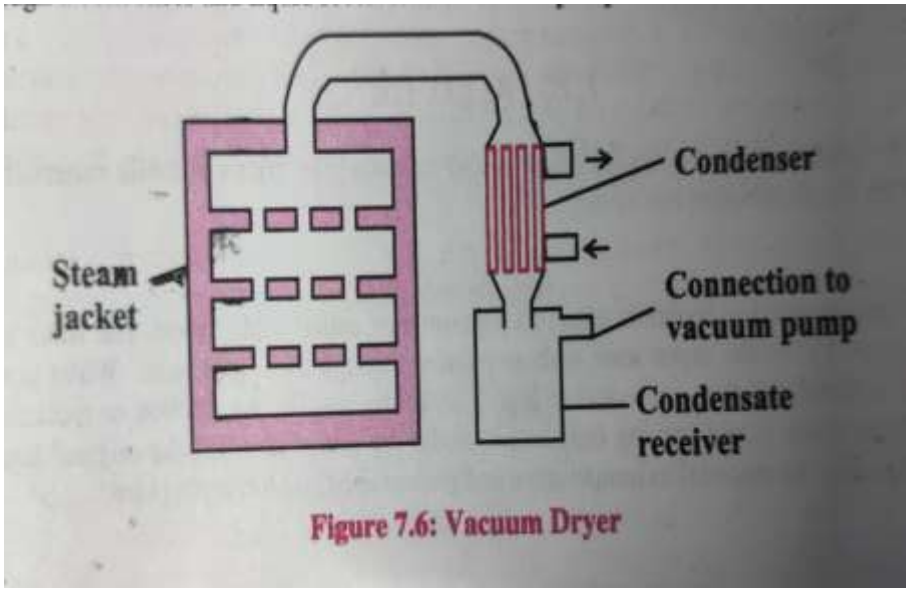


Figure 7.6: Vacuum Dryer

Construction

It consists of a heavy steam jacketed container made of iron. The closed chamber consists of shelves that are used to place metal trays consisting of material. The shelves provide a large area for heat conduction. The chamber is so strong to withstand under vacuum. The oven can be closed by a door. The oven is connected through a condenser and liquid receiver to a vacuum pump.

Working

The trays that are present in the dryer are used to dry the materials that are placed on the shelves and the pressure is reduced to 30 to 60 kps by the vacuum pump. The door closes firmly and steam passes through the jacket space and the shelves. So the heat transfer is carried out by the conduction mechanism. When evaporating under vacuum, the water is evaporated from the material at 25-30°C. The vapor goes to the condenser. After drying the vacuum line is disconnected. Then the materials are collected from the trays.

Advantages

1.Material handling is easy.
2. Hollow shelves which are electrically heated can be used.
3. It provides large surface area. So the heat can be easily transfer throughout the body of the dryer and fast drying action takes place.

Disadvantages

1. Dryer is a batch type process
2. Need high maintenance.
3. There is a danger of over heating due to vacuum.

THANK YOU

EVAPORATION

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A process of converting a liquid into gas by absorption of heat

BOILING POINT

- The temperature at which a liquid start boiling and gets converted into vapor
- It is different for different liquid

VAPOR PRESSURE

- Tendency of a material to change into a gaseous state.
- That's too different for different liquid.

Example-Boiling Point
Water-100 Degrees Celsius
Alcohol-78.37 Degrees Celsius.

Evaporation is a surface phenomenon in which first of all liquid on a surface evaporate.

OBJECTIVE AND APPLICATION OF EVAPORATION

- For getting concentrated product
- Removal of water from aqueous solution
- Removal of water from solid particle for getting dry product
- For reduction of weight
- Drying of drug for further use in manufacturing of medicine

FACTOR INFLUENCING EVAPORATION

1.Temprature
2.Surface Area
3. Vapor Pressure
4.Humidity
5.Wind Speed

Rate of Evaporation- The rate of which evaporation increases or decreases.

1. Temperature

Temperature α Rate of Evaporation

The rate of evaporation increases as the temperature is increases

2. Surface Area

Surface Area α Rate of Evaporation

That is rate of evaporation increases with respect to surface area

3. Vapour Pressure

Vapor Pressure α Rate of Evaporation

On increasing the vapour pressure of liquid rate of evaporation also increases

4. Humidity

Humidity α 1 / Rate of Evaporation

The rate of evaporation decreases as the humidity in atmosphere increases

5. Wind Speed

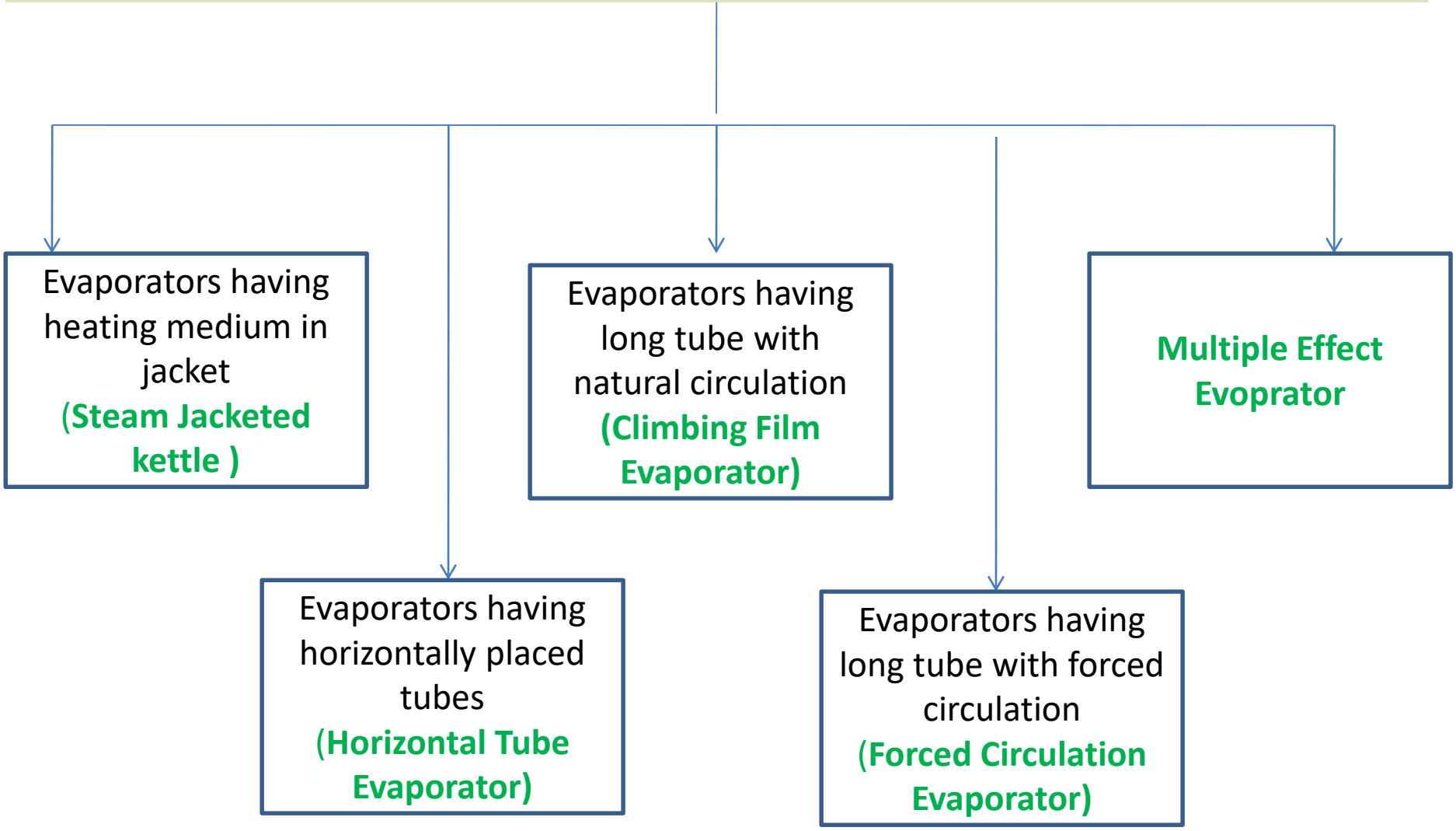
Wind Speed α Rate of Evaporation

Rate of evaporation increases with increase in wind speed

Difference Between Evaporation & Heat Transfer

EVAPORATION	OTHER HEAT PROCESS
The part left over is concentrated liquid	Drying- The part left over is solid
Transformation of liquid into a gas (No need of separation)	Distillation- A process of separation (Separation is compulsory)
Motive is to get concentrated liquid	Crystallization- Motive of concentrating solution to get crystals.
Liquid into gas	Boling- Process of evaporation of a liquid at the boiling point of the liquid

CLASSIFICATION OF EVAPORATOR

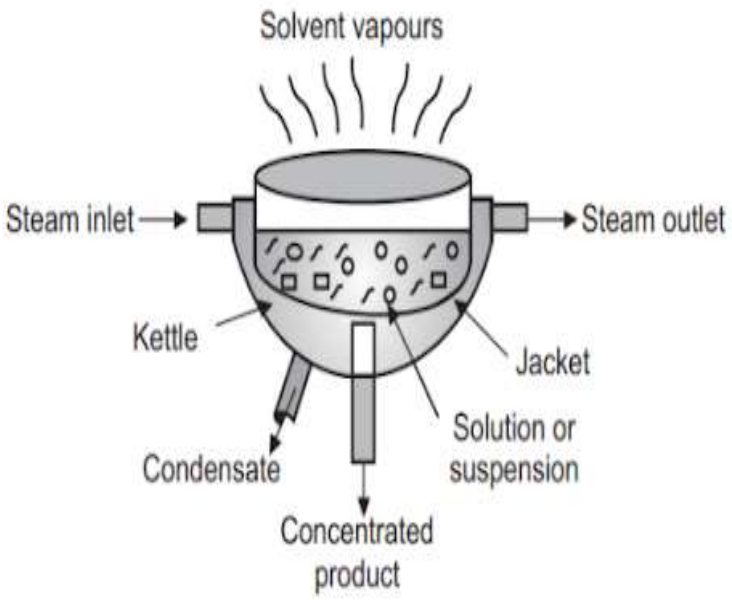


Steam Jacketed Kettle

It is also known as Evaporating Pan

PIRINCIPLE

The mechanism involved in this evaporation process is conduction and convection. The material is placed jacketed kettle. Steam provide heat to a jacketed kettle in which the aqueous extract is placed. The raised temperature increases the tendency of the solvent molecules to escape into the vapors.



Conduction- Heat transfer in a solid series. In this particles are directly connect with each other and heat transfer one by one.
In this particles must be stationary & Heat transfer to Higher temp to Lower temp

Convection- It is transfer of heat through movement of liquid (Fluid)
I which initial positions particle absorb heat then displace with other particle

CONSTRUCTION

The apparatus consists of a hemispherical, or shallower pan which is made of copper or stainless steel. Copper is an excellent material for the kettle, because of its good conductivity. The hemispherical shape provide best surface for heating and vaporization. It is surrounded by a jacket with steam inlet. Steam jacket is also attached to condensate outlet and vent for non condensed gases. The kettle is also fitted with outlet at the bottom for discharge of product.

WORKING

The solution or suspension is placed into the kettle. Steam is allowed to enter through inlet which provide heat to solution or suspension. The condensate leaves through outlet. On small scale kettle is used and also agitation is done manually or mechanically by stirrer. The final product (concentrated product) is collected from the bottom.

ADVANTAGES

1. Simple in construction
2. Easy to operate
3. Maintenance and installation cost is low
4. Product can be easily removed
5. Used for small scale and large scale operations

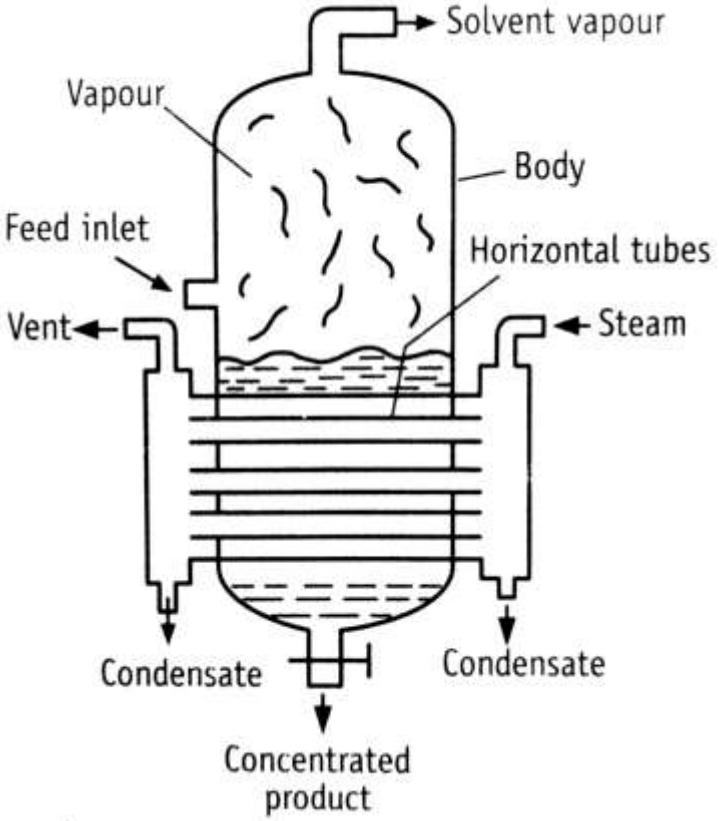
DISADVANTAGES

1. Not suitable for heat sensitive product
2. Used only for aqueous liquids
3. Many product produce foam

HORIZONTAL TUBE EVAPORATOR

PRINCIPLE

Steam is passed through tubes placed horizontally. As a result solvent outside tubes get evaporated a passed from the top and concentrated liquid is discharged from the bottom.



CONSTRUCTION

It consist of vertical cylindrical body having dome shaped top and bottom. The lower part of cylindrical body is fitted to steam inlet and outlet for condensate. Inside the cylinder horizontal tubes are placed. Horizontal tubes are 6 to 8 in number and made of stainless steel. The Lower portion also consist of vent for non condensed gases. Feed inlet is also provided. At the top of the there is one outlet for vapor and concentrated product is discharged from the bottom of the body.

WORKING

Feed is introduced through inlet. Steam is also introduced into the body. Therefore tubes get heated. The condensate pass through outlet. The heat is absorbed by the feed and solvent get evaporated. The vapor formed pass through outlet at the top of the body. The process repeat to get concentrated product which is collected from the bottom.

Advantages

1. Cheap
2.Esay to install
3. Suitable for batch or continuous operation

Disadvantage

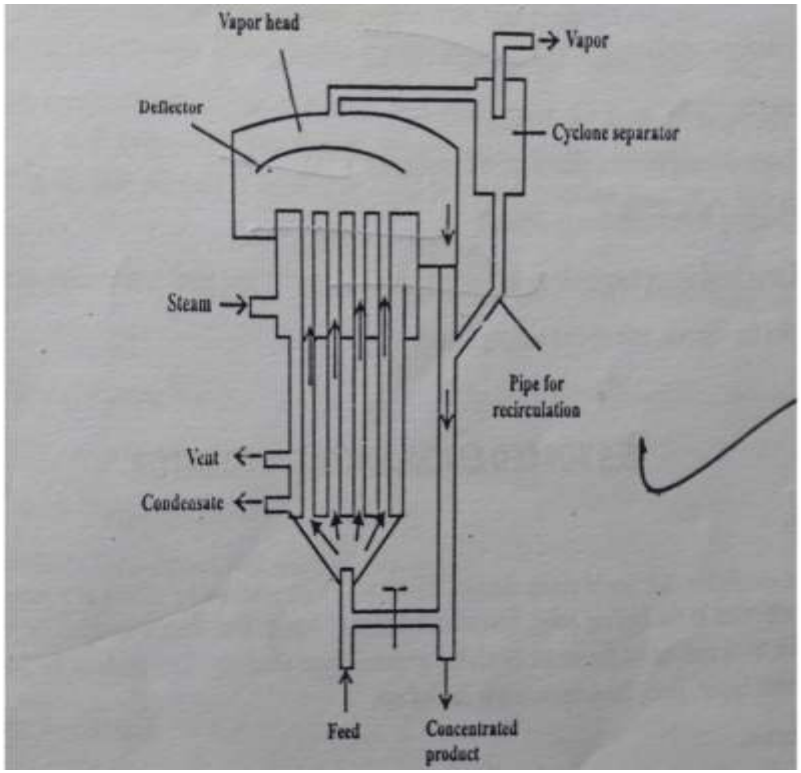
They have smaller capacity than other evaporators

CLIMBING FILM EVAPORATOR

It is also known as Rising Film Evaporator

PRINCIPLE

The tubes are heated outside by steam. The liquor enters from below and flows through the heated tubes. The liquid near the walls becomes steam and forms small bubbles. Larger bubbles flow up with slag and strikes deflector. Deflector throws concentrate down.



CONSTRUCTION

Heating unit consists of steam jacketed tubes. Long and narrow tubes are held between the two plates. Deflector is placed at the top of the vapour head. There is also the provision of Inlets for steam & feed. Outlets are provided for vapour, concentrated product, non condensed gases & condensate. A cyclone separator is placed at the top of vapour head.

WORKING

The preheated liquid enters from the bottom. Steam is passed into the unit through the inlet provided Through the wall, heat is transferred to the liquid. As a result, liquid becomes vapor. Small bubbles are formed that merge or fuse to form large bubbles. The slug of liquid is blown up into the tubes. As a result the liquid will spread like a film on the walls. This film of liquid vaporizes continuously and finally concentrated product is obtained that can be collected from the bottom. The vapor eject from the top of the unit

ADVANTAGES

1. Large area for heat transfer due to large tubes
2. Used for foam forming liquids
3. Instrument need less space

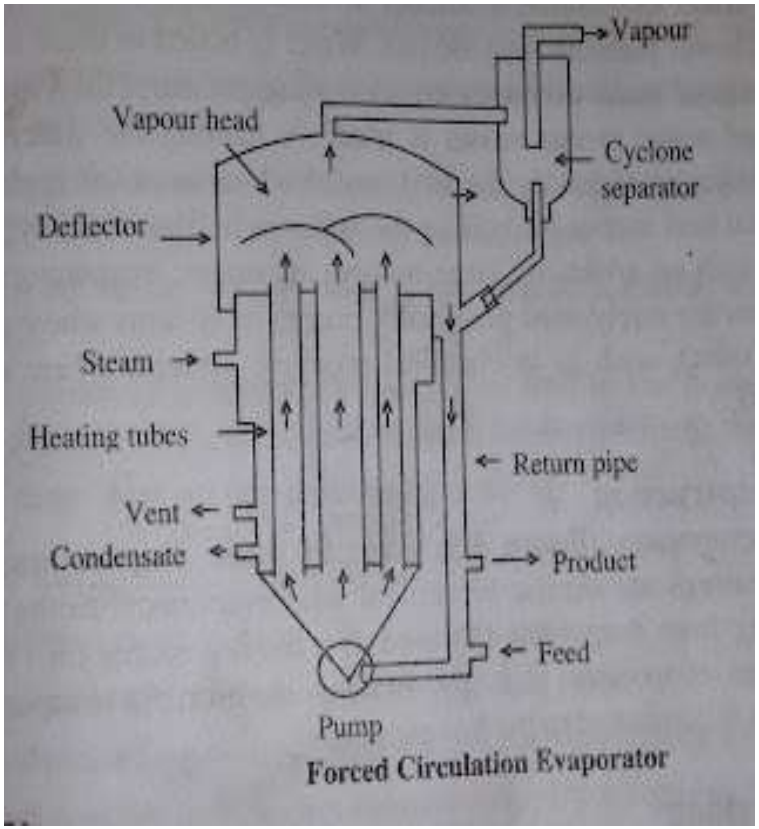
DISADVANTAGES

1. Expensive and construction is quite complicated
2. Cleaning & maintenance is difficult
3. Large head space required
4. Not for viscous liquid

FORCED CIRCULATION EVOPRATOR

PRINCIPLE

In forced circulation, the liquid flows through the tubes at high pressure by means of a pump. Therefore, there is elevation in the boiling point. Forced circulation of liquids also creates agitation. When the liquid leaves the tubes and enters the steam head, the pressure drops suddenly. This leads to the flashing of the super heated liquor. Thus, the evaporation is carried out.



Forced Circulation Evaporator

CONSTRUCTION

The unit consist of longer steam jacketed tubes held between two tube sheets. Tubes are 2.5 meter long. A pump is used to force liquid to the tubes with high velocity. The pump force liquid through tubes into the flash chamber or vapor head. The flash chamber consist of deflectors. The vapor head is also joined to return pipe.

WORKING

Steam is passed into the unit through the inlet provided. The pump sends the feed (liquid) to the tubes at high velocity. The liquid rises and boils as it passes through the tubes. This mixture of vapor and liquid strikes to the deflector. The vapour and liquid get separated. Then the liquid falls down. The steam enters the cyclone separator and leaves the unit. Thus, the continuous circulation is maintained. As a result concentrated product is formed which is collected.

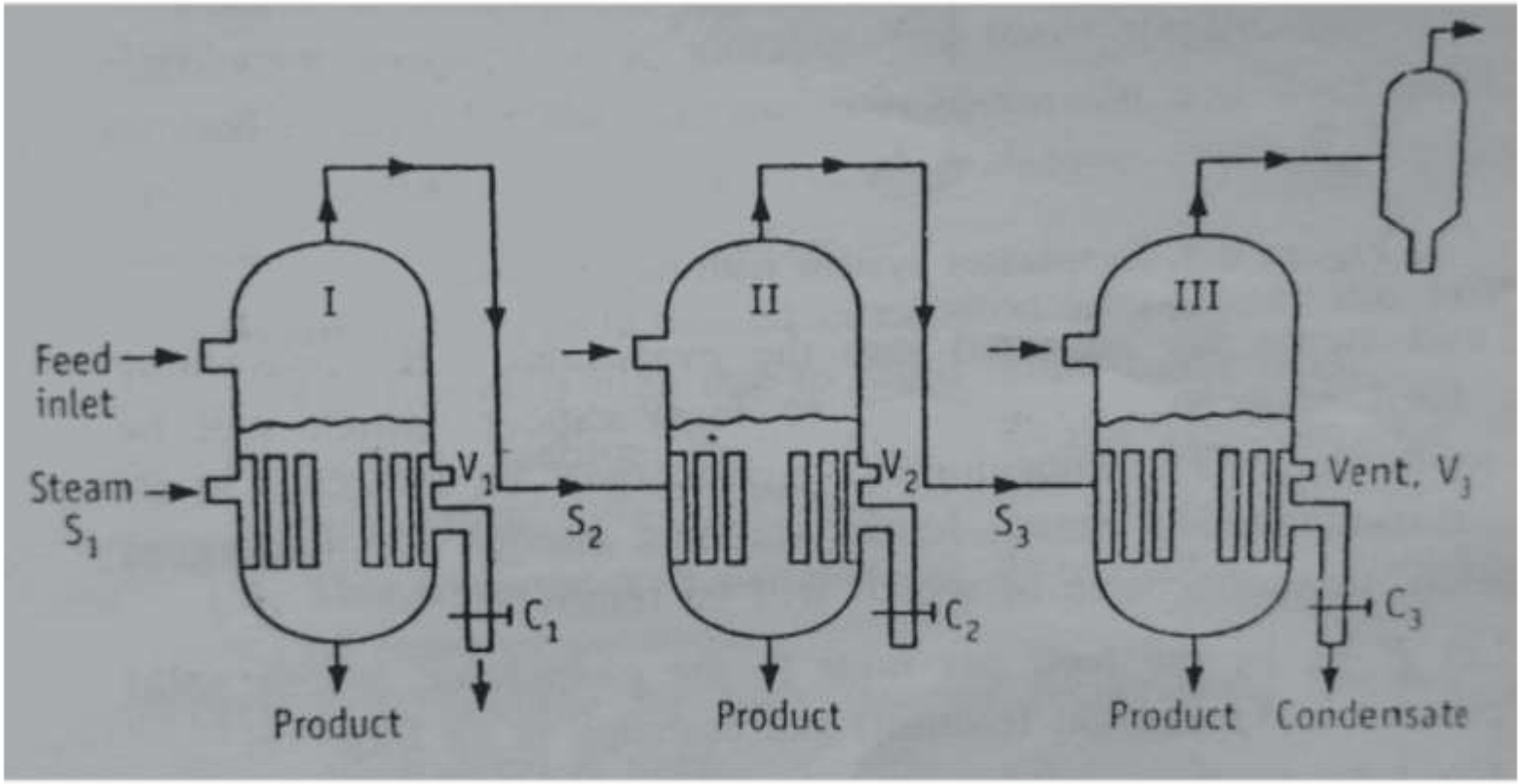
Advantages

1. There is a rapid liquid movement due to high heat transfer coefficient.
2. It is suitable for the viscous preparation because pumping mechanism is used.

Disadvantages

Equipment cost is high due to additional pump requirement

MULTIPLE EFFECT EVAPORATOR



THANK YOU

FILTRATION

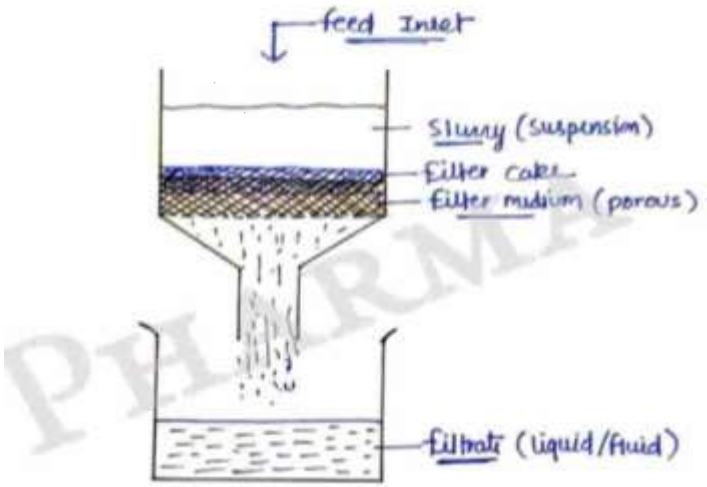
FILTRATION

It defined as Separation of a solids from process of a fluids by passing mixture of slurry (solid + fluid) through a porous medium, that retain the solids, but allows the fluids to pass through

OR

A Process of separation of solids particles from a fluid.

Components –



- The suspension (mixture) to be filtered Is known as **slurry**
- The porous medium used to retain the solids is known as **filter medium**
- The accumulated solid on the filter are known as **filter cake**
- The clear liquid (fluid) passing through the filter is known as **filtrate**

MECHANISM

➢Filtration take place, when the pores of the filter medium smaller than the size of particles to be separated.

➢So, the filter medium resist solid particles and allow the fluid (liquid) to pass

➢Now, filter cake also resist some slurry and act as a secondary filter media or secondary filtration (cake filtration)

OBJECTIVE AND APPLICATION

1. Production of Sterile Product-

Air is filtered through a HEPA filter (High efficiency particulate air) in sterilization process or in a laminar air flow to obtain sterile air .

2. Production of Bulk Drug-

Solid of intermediate and finished product are separated from the reaction mixture by filtration technique.
By this method impurities can be removed.

3. Production of Liquid Oral Formulation-

Filtration is an essential step in the production of liquid oral solution for obtaining the clear solution (clarification)

- De waxing of oils
- Removing suspended oil from aqueous solution

(Example - syrup, elixir , eye drop etc)

4. Waste water treatment -

Waste solid must be separated from the waste liquid prior to its disposable.

Clarification

When the suspending solid particle are 1% or less than 1% in any liquid (fluid) or any slurry.

FACTOR AFFECTING FILTRATION

1. SURFACE AREA OF FILTER MEDIA

The rate of filtration is directly proportional to surface area of filter media.

Surface Area α Filtration

As the surface area increases, the filtration will be increases.

Surface Area ↑ = Filtration ↑

2. PARTICLE SIZE OF SOLIDS

Particle size is directly proportional to the rate of filtration.

Particle Size α Filtration

As the particle size increases, the rate of filtration will be increases.

Particle Size ↑ = Filtration ↑

3. FILTER CAKE (RESISTANCE)

Filter cake is inversely proportional to the rate of filtration

Filter Cake α 1 / Filtration

Filtration is decrease on increasing the filter Cake.

Filter Cake ↑ = Filtration ↓

4.VISCOSITY

Viscosity is inversely proportional to the rate of filtration.

Viscosity α 1/Filtration

As the viscosity increases, the filtration will be decreases.

Viscosity ↑ = Filtration ↓

5. PRESSURE DIFFERENCE

The rate of filtration is directly proportional to the overall pressure drop across the filter media and filter cake.

The pressure difference is directly proportional to rate of filtration.

Pressure Difference α Filtration

As the pressure difference increases, the rate of filtration is increases.

Pressure Difference ↑ = Filtration ↑

FILTER MEDIA

Those substance which help in the filtration process, which retained the solid residue (filter cake) and allowed to pass liquid and also provide mechanical support for filter cake.

CHARACTERISTIC

- It must be inert (not sensitive)
- It should be a sufficient mechanical strength.
- It should allow the maximum passage of liquid
- Resistant to corrosive

MATERIAL USED AS A FILTER MEDIA

- Filter paper
- Woven material (made-up of wool, cotton, silk, etc.)
- Asbestos
- Membrane filter etc.

FILTER AID

Filter aid forms a surface deposit which screen out the solid and also prevent the plugging of the supporting filter medium.

CHARACTERISTIC

- It must be inert
- Porous rather than dense
- Recoverable

Example - Talc, charcoal, betonite, etc.

CLASSIFICATION OF FILTER EQUIPMENT

1. Pressure Filter
   - Plate and Frame Filter Press
   - Meta Filter

2. Vacuum Filter
   - Filter Leaf

3. Rotary Drum Filter

4. Cartridge Filter

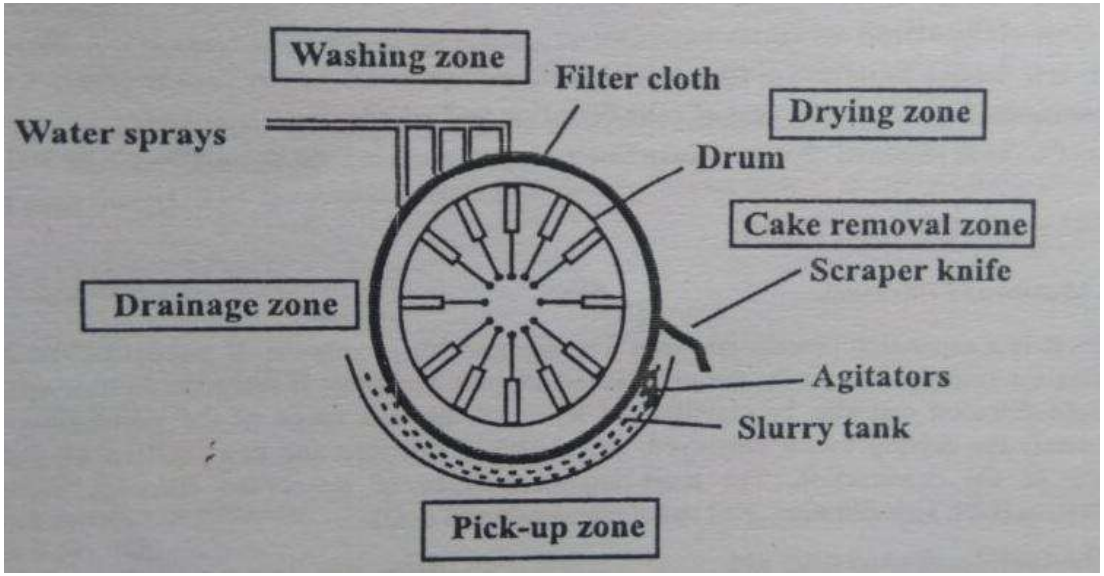
5. Membrane Filter

6. Seitz Filter

ROTARY DRUM FILTER

Principle- It is based on the principle of filtering the slurry through sieve like mechanism on the rotating drum surface, under the condition of vacuum.
In addition, compression, drying and removing filter cake is possible.

Construction-



- It consists of a metal cylinder mounted horizontally.
- Drum have diameter 3 meter, length 3.5 meter and surface area 20 meter square.
- Filter cloth is used as a filter media.
- Each of it is connected via internal pipe to the center of the drum through a rotating wall.

Working-

- The drum is deep into the slurry (Speed of drum is less than 1 revolution per minute) and vacuum is applied to the outlet, which is connected to the filtered receiver (Pick up Zone)

- When the cake has formed, the cake drained or partially dried by vacuum (draining zone)

- The drum is sprayed with water to wash the cake (washing zone)

- Retaining the vacuum connection drains the cake and produces partial dryness (draining zone).

- Then cake is removed by doctor knife (cake removal zone)

Advantages

- Labor cost is low due to the automatic and continuous process.
- Filter has large surface area
- Cake is removed simultaneously.

Disadvantages

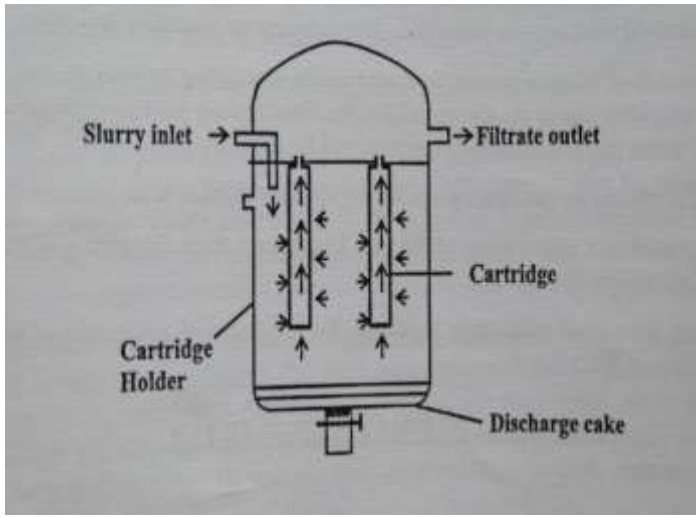
- Expensive equipment
- Cake tend to crack

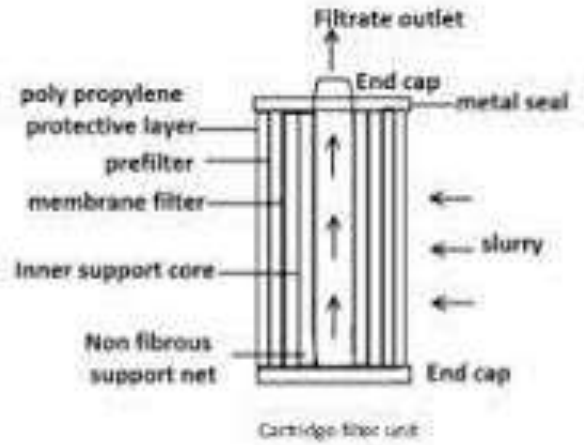
CARTRIDGE FILTER

Principle

Cartridge filter is a thin porous membrane in which pre-filter and membrane filter are combined into a single unit.

The filtration action is mainly sieve like and the particles are rated on the surface.





Construction

- Cartridge filter has a cylindrical vessel With changeable filter media.
- These are made-up of plastic or metal.
- It is made-up of two membrane filter made-up of polypropylene, pre filter and actual membrane filter (0.2 Micron)

Working

- The slurry is pumped into a cartridge holder.
- It passes through a cartridge filter unit by the mechanism of straining.
- The clear liquid passes to the center and moves up to the collect through the outlet.

Uses

Use for preparation of particulate free solution for parental ophthalmic use

PHARMACEUTICAL ENGINEERING

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UNIT-I **10 Hours**

- **Flow of fluids:** Types of manometers, Reynolds number and its significance, Bernoulli's theorem and its applications, Energy losses, Orifice meter, Venturimeter, Pitot tube and Rotometer.
- **Size Reduction:** Objectives, Mechanisms & Laws governing size reduction, factors affecting size reduction, principles, construction, working, uses, merits and demerits of Hammer mill, ball mill, fluid energy mill, Edge runner mill & end runner mill.
- **Size Separation:** Objectives, applications & mechanism of size separation, official standards of powders, sieves, size separation Principles, construction, working, uses, merits and demerits of Sieve shaker, cyclone separator, Air separator, Bag filter & elutriation tank.

UNIT-II **10 Hours**

- **Heat Transfer:** Objectives, applications & Heat transfer mechanisms. Fourier's law, Heat transfer by conduction, convection & radiation. Heat interchangers & heat exchangers.

- **Evaporation:** Objectives, applications and factors influencing evaporation, differences between evaporation and other heat process. principles, construction, working, uses, merits and demerits of Steam jacketed kettle, horizontal tube evaporator, climbing film evaporator, forced circulation evaporator, multiple effect evaporator& Economy of multiple effect evaporator.

- **Distillation:** Basic Principles and methodology of simple distillation,flash distillation, fractional distillation, distillation under reduced pressure, steam distillation & molecular distillation

UNIT- III **08 Hours**

- **Drying:** Objectives, applications & mechanism of drying process, measurements & applications of Equilibrium Moisture content, rate of drying curve. principles, construction, working, uses, merits and demerits of Tray dryer, drum dryer spray dryer, fluidized bed dryer, vacuum dryer, freeze dryer.

- **Mixing:** Objectives, applications & factors affecting mixing, Difference between solid and liquid mixing, mechanism of solid mixing, liquids mixing and semisolids mixing. Principles, Construction, Working, uses, Merits and Demerits of Double cone blender, twin shell blender, ribbon blender, Sigma blade mixer, planetary mixers, Propellers, Turbines, Paddles & Silverson Emulsifier,

UNIT-IV **08 Hours**

- **Filtration:** Objectives, applications, Theories & 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.
- **Centrifugation:** Objectives, principle & applications of Centrifugation, principles, construction, working, uses, merits and demerits of Perforated basket centrifuge, Non-perforated basket centrifuge, semi continuous centrifuge & super centrifuge.

UNIT- V **07 Hours**

- **Materials of pharmaceutical plant construction, Corrosion and its prevention:** Factors affecting during materials selected for Pharmaceutical plant construction, Theories of corrosion, types of corrosion and there prevention. Ferrous and nonferrous metals, inorganic and organic non metals, basic of material handling systems.

SIZE REDUCTION

INTRODUCTION

Size reduction or Pulverizations is the process of reducing large substances into smaller particles. Size reduction machines for large particle sizes (i.e. particles above 40 mm), are known as crushers (**a machine which breaks up solids by pressing them)**, while the particle sizes below this are processed by mills 9Milling **involves the application of mechanical energy to physically break down coarse particles to finer ones**). Size Reduction is an important operation in many pharmaceutical applications. For pharmaceutical purpose, mono- size particles are best of use. For solid materials, grinding and cutting are used as a size reduction process, while emulsification or atomization for liquid material are used.

ADVANTAGES OF SIZE REDUCTION

1. For effective mixing particles of uniform size are desirable. As content uniformity is important in case of potent and low dose drugs formulations

2. Size reduction increase surface area of material and this further improve rate of dissolution.

3. During compression of tablet, particle size should be small so that powder can easily flow into dies.

4. Extraction of active constituent become effective if smaller particles are used.

5. Drying will be effective when small sized granules or powders are used.

DISADVANTAGES OF SIZE REDUCTION

1. Thermo labile substances get decomposed during size reduction. This is due to heat produced during milling

2. There are chances of contamination during milling and grinding.

OBJECTIVE OF SIZE REDUCTION

1. Increase Surface Area- Reduced size leads to increased surface area.
For example: The dissolution rate of the solid drug particles increases many times after the size reduction. Micronized form of Griseofulvin, an antifungal drug, shows about five times better absorption.

2.Ease of Mixing- Reducing the size of particles in a narrow range makes mixing easier.

3. Stability of Suspension: The fine particle size in pharmaceutical suspensions decreases sedimentation rate.

4. Dosage form: Pharmaceutical capsules, insufflations (i.e, inhaled powders directly into the lungs), suppositories and ointments require the particle size to be less than 60 mm in size.

5. Stability of emulsion: The stability of emulsion can be increased by decreasing size of oil globules.

6. Reduce Irritation: Ophthalmic preparations and preparation meant for external application to skin should be free from coarse and gritty particles to avoid irritation.

7. Increase Absorption: The rate of drug absorption will be higher if the particle size will be small.

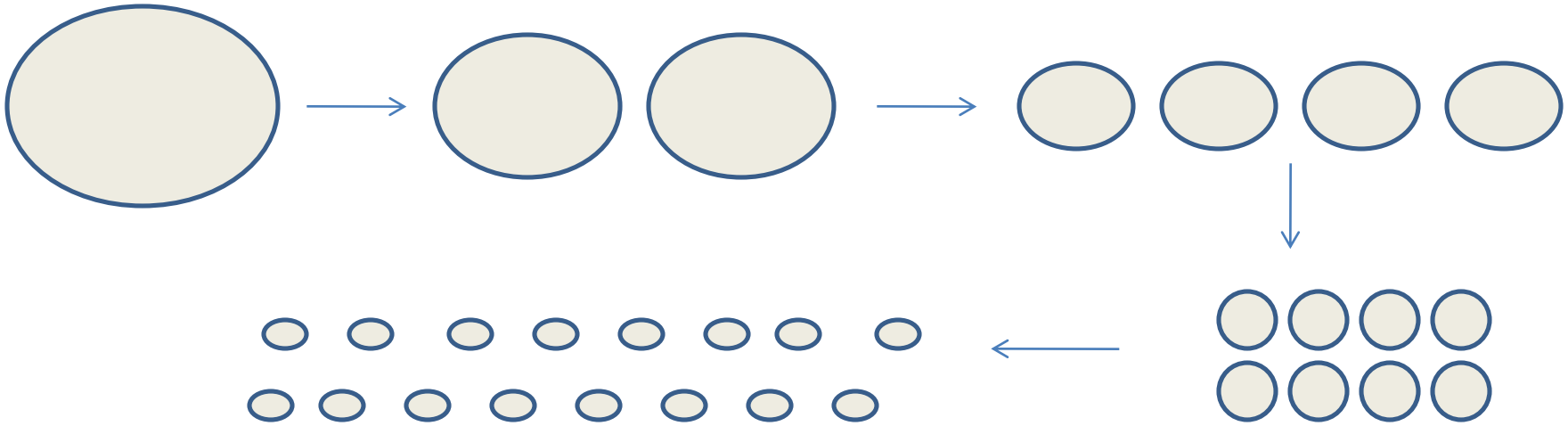
8. Appearance: By reducing particle size, physical appearance of ointment, creams, paste can be enhanced.

LAWS GOVERNING SIZE REDUCTION

1. **Kick's Law**
2. **Rittinger's Law**
3. **Bond's Law**

MECHANISM-

The more we reduce the particles, the more energy will be used to make it smaller.



KICK'S LAW

According to this law, energy required to reduce size of particle is proportional to ratio of initial size of a typical dimension to the final size of that dimension.

Where

$$E = K_k \, In\{ d1/d2 \}$$

E-energy required per mass of feed

K_k -Kick's constant

d1- Initial size of pieces (m)

d2- Final Size of particles (m)

d1/d2 size reduction ratio

RITTINGER'S LAW

This law states that the energy required for the size reduction of unit mass is proportional to the new surface area produced.

Where

$$E = K_R \{1/d1 - 1/d2\}$$

E-energy required per mass of feed

K_R-Rittinger's constant

d1- Initial size of pieces (m)

d2- Final size of ground particles

BOND'S LAW

This law states that energy used for size reduction is proportional to new crack length.

Where

$$E/W = \sqrt{100/d2} - \sqrt{100/d1}$$

E-energy required per mass of feed

W-Bond Work Index work required to reduce a unit weight

d_1 = diameter of sieve aperture that allows 80% of mass of feed to pass (in meters)

d2-diameter of sieve aperture that allows 80% of mass of ground material to pass (in meters)

FACTORS AFFECTING SIZE REDUCTION

1. **Hardness -** It is a surface property of the material. The hardness of material is measured by a devised known as Moh's Scale The Moh scale is from 1 to 10. Material of hardness 1 to 3 are classified as soft , 4 to 7 intermediate and 8 to 10 hard . The harder the material the more difficult it is to reduce in size

2. Toughness- The Toughness of a material is sometimes more important than the hardness. A soft but tough material may present more problems in reducing size than a hard but brittle substance. For example, it is difficult to break the rubber than a blackboard chalk stick.

3. Abrasiveness - Abrasiveness is a property of hard materials. During the grinding of abrasive substances, the final powder may be contaminated with more than 0.1 percent of the grinding mill's worn metal.

4. Stickiness- Stickiness is a property which causes considerable difficulty in reducing the size because the material get adhere to the grinding surfaces or the screen meshes may blogged

5. Slipperiness- It is the opposite of stickiness. It can also lead to size reduction difficulties as the material acts as a lubricant and decreases the efficiency of grinding surfaces.

6. Softening temperature- During size reduction process, sometimes heat is generated which can cause some substances to soften, and the temperature at which this occurs may be important. Waxy substances, such as stearic acid, or drugs containing oils or fats are examples that may be affected. Some methods can be used to overcome this like cooling the mill, either by a water jacket or by passing a stream of air through the equipment.

7. Moisture content- The moisture content influences a number of properties that may affect size reduction, For example, hardness, toughness or viscosity. In general the materials must be dry or moist and merely moist. Usually, less than 5 percent moisture is adequate if the substance is to be ground de or more than 50 if it is being subjected to wet milling

CLASSIFICATION OF SIZE REDUCTION EQUIPMENTS

1. Impact- In this, drug molecules converted into small particles through any type of impact. Amount of drug remain constant.
Example- Hammer mill

2. Attrition - In this method, Drug particles collides with each other and reduce there size from large molecule to small molecule (Particles)
Example- Fluid energy mill

3. Combined Impact and Attrition- In this method, Impact & Attrition both apply simultaneously
Example- Ball Mill

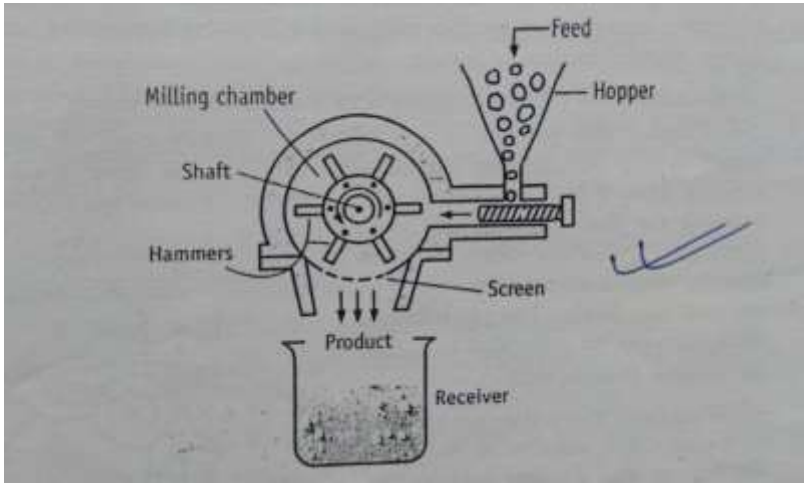
4. Cutting - In this method, we reduced large drug molecules into small through Cutting, it is mostly used for soft & fibrous drugs.
Example- Cutter mill

5. Compression- The Drug molecules reduces through compression or by applying pressure on it.
-It is mostly use for hand nature drugs.
Example- Edge and End Runner Mill

HAMMER MILL

PRINCIPLE

Hammer mill based on principle of impact between hammer (which is in continuous motion) and powder. Due to high speed material get pulverized



CONSTRUCTION

Hammer mill consists of a housing frame, enclosing a central shaft to which four or more hammers are attached. Hammers are made of stainless steel. Shaft is either horizontal or vertical type. Hammers blades can be flat edges or sharp edges or both on each side. The lower part of the casing consists of screen through which materials can pass after size reduction. Screen are prepared by metal sheet with perforated holes. The screen can be changed according to the particle size required. The material is collected in a container placed below the screen. The unit is enclosed in a chamber.

WORKING

The hopper is used to place the feed material. The material from the hopper flows vertically and then horizontally. The hammers are in continuous motion and rotating at a speed of 8000 to 15000 revolutions per minute. When the feed material strikes with the rotary hammer, the material breaks down into smaller pieces. Then these particles pass through the screen.

ADVANTAGES

1. It is rapid in action, and is capable of grinding many different types of materials.
2. The fineness of product can be regulated by variation of rotor speed, hammer type and size and shape of mesh
3. Operation is done in chamber. So dust can be minimized
4. Small space requires for setup of mill

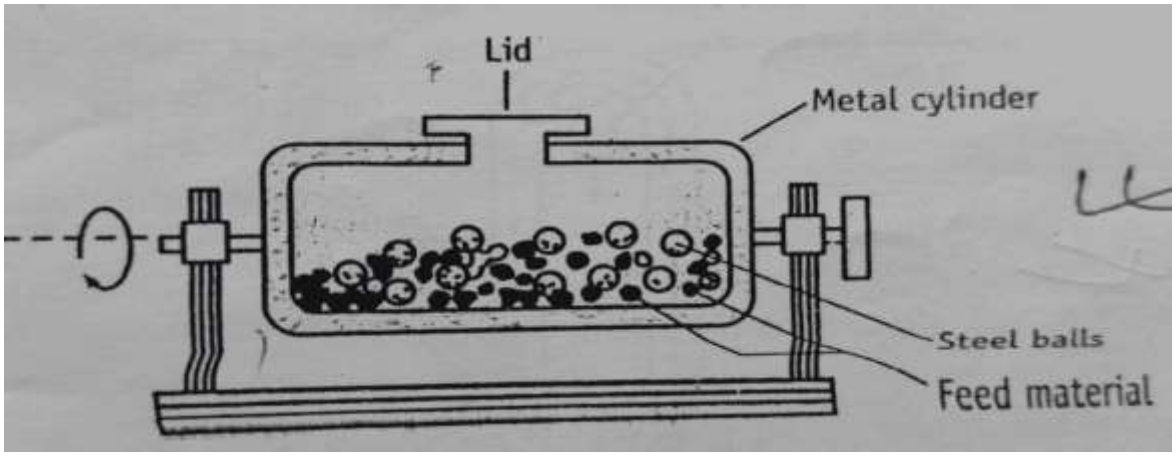
DISADVANTAGES

1. High speed generates heat that may affect thermo labile materials.
2. Due to the high speed of operation, the hammer mill can be damaged if some foreign materials such as stone, metal parts etc are present in the feed
3. Chances of clogging of screen.

BALL MILL OR PEBBLE MILL

This is also known as tumbling mills.
Method of size reduction: Impact and Attrition

PRINCIPLE In the ball mill, Impact and Attrition both are responsible for the size reduction. Rapidly moving balls are used for comminution of brittle material.



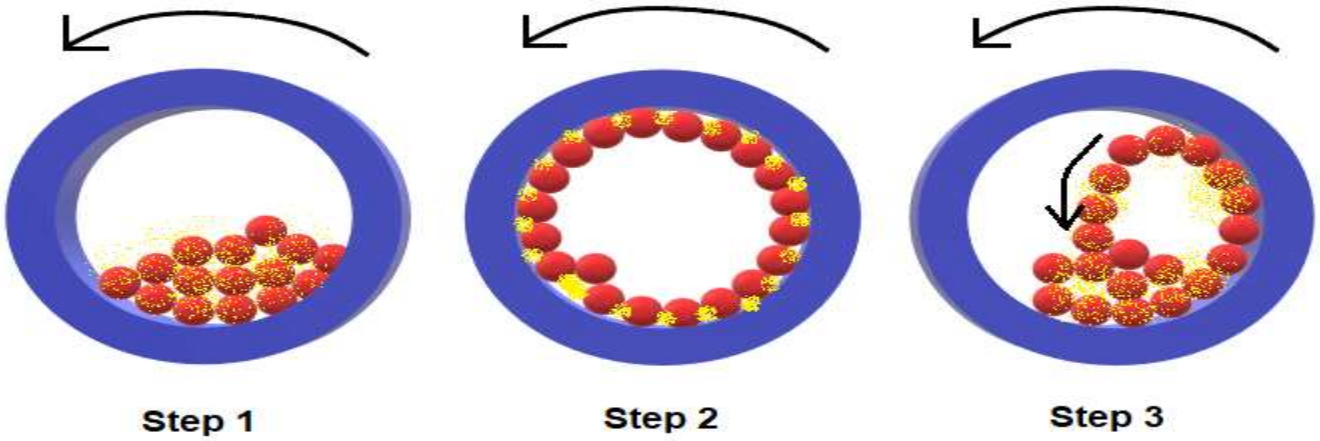
CONSTRUCTION

The ball mill consists of a hollow metal cylinder mounted on shaft and rotating about its horizontal axis. The cylinder can be made of metal, porcelain or rubber. Inside the cylinder balls or pebbles are placed. The balls occupy between 30 and 50% of the volume of the cylinder. The diameter of the balls depends on the size of the feed and the diameter of the cylinder. The diameter of the balls varies from 2cm to 15cm. The balls can be made of metal, porcelain or stainless steel. The ball acts as a grinding medium

WORKING

The material to be ground is kept in a hollow cylinder. The material is placed up to 60% of the volume. A fixed number of balls is placed in the cylinder and then the cylinder is closed. The mill is allowed to rotate Speed of rotation is an important point of consideration.

At low speed, the mass of balls will slide or roll up one over another and will only produce an insignificant amount of size reduction. At high speeds, the balls are thrown to the cylinder wall due to centrifugal force and no grinding will occur. At 2/3rd speed centrifugation just occurs which is called the critical speed of the ball mill. At this speed the balls are carried almost to the top of the mill and then fall in a cascade across the diameter of the mill. In this way, the maximum size reduction is obtained by impact of the particles between the balls and by attrition between the balls.



ADVANTAGES

1. Very fine powder can be obtained
2. Suitable for both wet and dry grinding processes.
3. Toxic substances can be ground, as closed cylinder are used.
4. In ball mill, installation, operation and labor costs are low.
5. Suitable for batch process.
6. Grinding medium is cheap
7. Cost of installation and production is low.

DISADVANTAGES

1. The ball mill is a very noisy machine especially when metal cylinder is used.
2. Ball mill is a slow process.

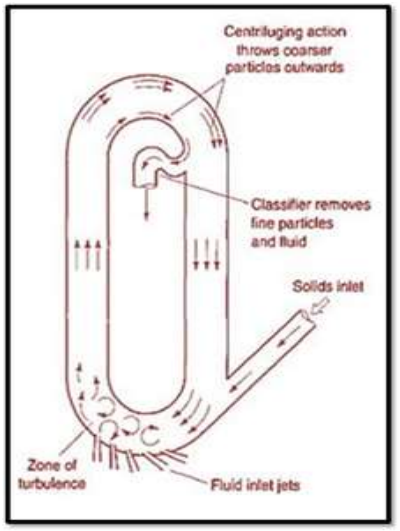
FLUID ENERGY MILL OR JET MILL

This is also known as micronizers or ultrafine grinders.

Method of size reduction: Attrition

PRINCIPLE

Fluid energy mill based on principle of Attrition. Size reduction take place by high velocity collision between particle.



CONSTRUCTION

It consists of a loop of pipe, which has a diameter of 20 to 200 mm. The height of the loop may be up to 2m. Several nozzles are fitted at the bottom of the pipe. Generally 2 to 6 grinding nozzles are present. A classifier is fitted at the product collection point. Normally compressed air are used.

WORKING

The compressed air is injected at very high pressure through nozzles at the bottom. This results in a high circulation velocity that produces turbulence. The solids are introduced into the stream through the feed inlet. As a result of the high degree of turbulence, attrition occur between the particles. A classifier is installed in the system so that only finer sized particles are collected as products and larger particles are sent back into the air stream for further reduction of size. The feed to the mill is previously of reduced size and passed through a screen. The size of the product may be 5mm or below

ADVANTAGES

1. This mill is suitable for thermo labile substance because no heat is produced
2 The particle size of the product is smaller when compared to other methods.
3. There is no contamination of the product

DISADVANTAGES

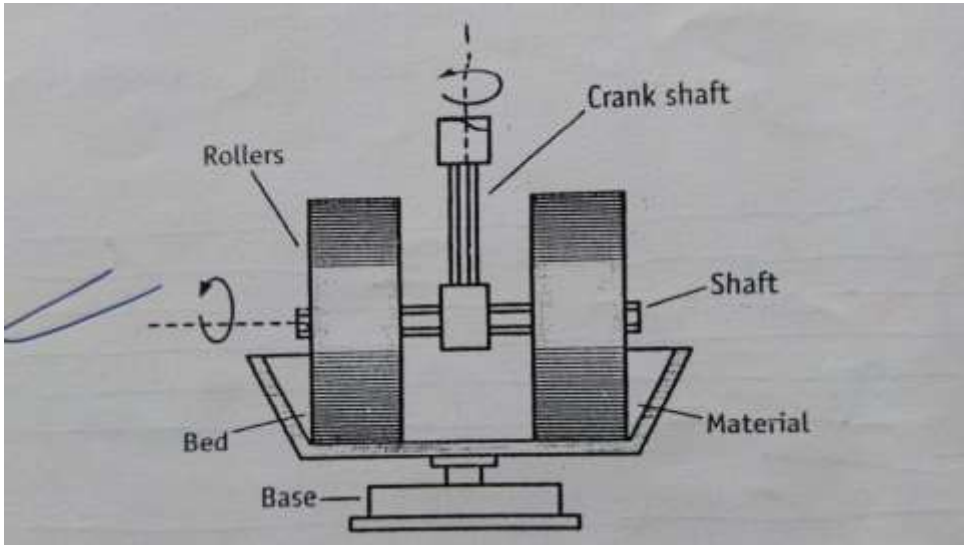
1. Not suitable for soft material milling
2. Expensive.

EDGE RUNNER MILL

PRINCIPLE

The edge runner mill mainly works on the compression. Size reduction is obtained by Crushing & shearing force involved during the movement of stones.

Method of size reduction: Crushing and shearing (Compression)



CONSTRUCTION

It consist of two heavy rollers which moves on the bed. The bed is made of stone or iron. The roller rotate on central shaft. The rollers mounted on a horizontal shaft & also move around the bed.

WORKING

The material to be ground is placed on the bed. The scraper is used which continuously remove material that get adhere to side of pan and return back it to crushing zone. The stones continuously revolve on its axis. Size reduction is obtained by shearing along with crushing.

The material is ground for a definite period and then it is passed through the sieves to get the powder of the required size.

ADVANTAGES

1. It is mostly used for all types of the drugs.
2. Very fine particle size is obtained.
3. The major advantage is that during operation less attention is required.
4. Simple in structure.

DISADVANTAGES

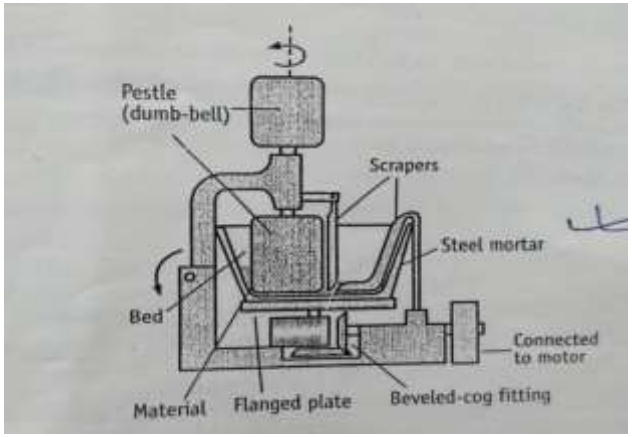
1. It occupy more space than other size reduction machine.
2. Chances of contamination of product due to roller material.
3. Not used for sticky materials.
4. It cause noise pollution.

END RUNNER MILL

Method of size reduction: Crushing and shearing (Compression)

PRINCIPLE

Size reduction is done by crushing due to weight of pestle and shearing also involves due to pestle and mortar movement.



CONSTRUCTION

It consist of mechanical mortar and pestle. Mortar is made of steel and attached to a flanged plate. Mortar with plate can be rotated at high speed. The pestle is dumb bell shaped. The bottom of pestle is flat rather than round. Pestle can be raised to done emptying and cleaning. It consist of bed of stone which can be rotated. A scrapper is also present which force the material to grinding surface.

WORKING

The material whose size is to be reduced is placed in mortar. The scrapper is used to push the material in crushing zone. The mortar revolves at high speed and the revolving mortar causes the pestle to revolve. The resulting material passed through sieve to get powder of sufficient size. During the process, size reduction is achieved by crushing and shearing.

ADVANTAGE

1. It produces fine particles.
2. Require less attention during milling operation

DISADVANTAGE

1.It is not suitable for milling sticky materials.
2. Machine noise leading to noise pollution.

THANK YOU

SIZE SEPRATION

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INTRODUCTION

Sire separation is also known as sieving, sifting, screening. Size separation is a process that involves the separation of particles of desired size from the mixture of various size particles.

Size reduction gives particles of varying sizes. Sifting is done to get narrow size particles.

Size reduction alone is not sufficient to obtain mono-size or narrow size range powder. Therefore, size reduction and size separation should be combined to obtain powders of desired size

APPLICATION OF SIZE SEPARATION

Size separation can be used

1. To prepare granules of desired size to ensure good flowability.

2. To separate undesirable particles

3. To measure particle size and size distribution

4. To know the efficiency of size reduction equipments

5. To get uniform dosage form.

6. Monosize particles undergo less segregation

7. During filling the capsule, the particles must be uniform in weight and doses.

MECHANISAM OF SIZE SEPRATION

1. AGITATION

In this sieves are agitated in number of ways

1. Oscillation- The sieve is rack mounted that oscillate back and forth. It is a simple method, but the material can roll on the surface of the sieve. The reciprocating movement is induced by ordinary eccentric on the rotating shaft.

2. Vibration- In this method, sieve is vibrated at high speed by mean of eccentric device either electrically or mechanically

3. Gyration- In this method, the sieve is rubber mounted and connected to the eccentric flywheel. This gives a small amplitude rotational movement to the sieve, which in turn gives rotational movement to the particles that help pass them through the sieve.

Advantages:

A. Simple method

B. Inexpensive

Disadvantage:

A. Chances of clogging of sieve if powder is not dried

B. Size reduction occur due to collision between particle during agitation

2 BRUSHING METHOD:

In this method, the brush is used to move the particles on the surface of the sieve and to keep the mesh clear. The brush is rotated in the centre in the case of a circular sieve, but the spiral brush is rotated in longitudinal axis in the case of a horizontal cylindrical sieve. It is used to separate the size of the greasy sticky powder.

3 CENTRIFUGAL METHOD:

In this method, the high speed rotor is fixed inside a vertical cylindrical sieve, so that during rotation particles are thrown out by the centrifugal force. The air stream can be generated by means of air equipment, which helps in the separation of particles.

Example: Cyclone Separator and Air Separator

Advantage: Chances of blockage of sieve is less.

SIEVES

Sieves are used for size separation. Sieves determine the efficiency of screening devices. Most of Sieves used for pharmaceutical purpose are of wire mesh type. Each sieve is given a definite number which denotes number of the meshes present in a length of 2.54 cm or one inch.

MATERIAL OF CONSTRUCTION

Sieves are woven from wire of brass, bronze, stainless steel. Sieves should not be coated with any material. It should be non reactive with material used. The material used for construction should be resistant to corrosion.

Generally Iron wire is used as screen material because it is cheap but their disadvantage are corrosive nature and chances of contamination by iron. These disadvantages can be overcome by coating iron with galvanizing agent which increase the strength and also make it corrosion resistant.

Brass, Phosphor-bronze and stainless steel are the metals used due to their corrosion resistant, good strength and non contamination qualities.

For special purpose punched plates or perforated screens are used. These sieves are made by drilling holes of varying thickness in metal plate. The holes may be round, rectangular or square.

STANDARDS OF SIEVE

Holes in the screen is called Mesh. Mesh number indicate number of holes included in a length of 1 inch. Aperture of screen is the clear space between wires of screen opening and it is given in term of mm.

The common standards used for used for sieves are

a. Tyler Standard sieve series: It is U.S. standard sieve. They are available in various mesh number

b. U.S. Standard sieve series: It is used in U.S.A. 4-325.

c. British Standard sieve series (B.S.S): It is available in mesh number 5-300. It is extensively used in UK.

d. U.S.ASTM: It is "American Society of Testing Materials" standard sieve series. It is available in mesh number 4-325.

e. German DIN (Deutsche Industrienormein): It is commonly used in Europe and Germany.

f. F.S.S.: It is French Standard Sieve. It is available in mesh number 17-38.

g. IP standard Sieve series: It is commonly used in India

h. International test sieve series (ISO): It is used world wide.

SEIVE SHAKER

PRINCIPLE:

The particles of different sizes are separated by passing them through number of sieves in nest which oscillates back and forth continuously to reciprocating motion.

CONSTRUCTION:

Shaking screen consists of metal frame to which a screens are fixed. The standard sieves of different mesh numbers (as mentioned in IP and USP) are used.

WORKING:

The sieves are arranged in such a way that the largest aperture will be at the top and smallest aperture will be at the bottom. A sieve nest consist of 6 to 8 sieves. Powder sample having weight of 50 gm is placed on top most sieve. Then close the sieve set and fixed it on to mechanical shaker apparatus. Then start shaking the sieve set for stipulated time (20 minutes). Then all sieves are disassembled. The powder retained on each sieve is collected and weighed.

Advantages:

1. It require less power requirement.
2. Inexpensive
3. Easy to use

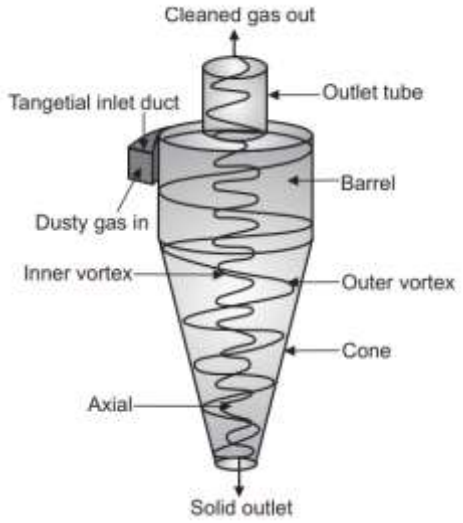
Disadvantages:

1. During shaking, attrition may occurs.
2. Chances of clogging of sieve if powder is not properly dry.

CYCLONE SEPARATOR

PRINCIPLE:

Cyclone separators are mainly used to separate the solids from fluids. This is based on principle of centrifugal force. The separation process depends on particle size as well as on density of particles.



CONSTRUCTION:

It consist of vertical, cylindrical vessel. It has conical base or bottom. The upper portion of vessel is fitted with tangential inlet. The outlet is arranged at base. The outlet for air or vapour is provided at centre of top, which extend inwardly into separator to prevent air short circuiting directly from inlet to outlet of fluids.

WORKING-

The solids are suspended in gas or air stream and are introduced tangentially at very high velocity into the vessel. A tangential inlet is the most common type of inlet because it is least expensive and most efficient. Then the rotary motion takes place inside the vessel. As a result vortex formation occur. The centrifugal force and the vortex throw the solids to the wall. The coarse solid particles fall into the conical bottom and are collected through solid outlets. The air or vapour can escape from the central outlet at the top.

PHARMACEUTICAL USES

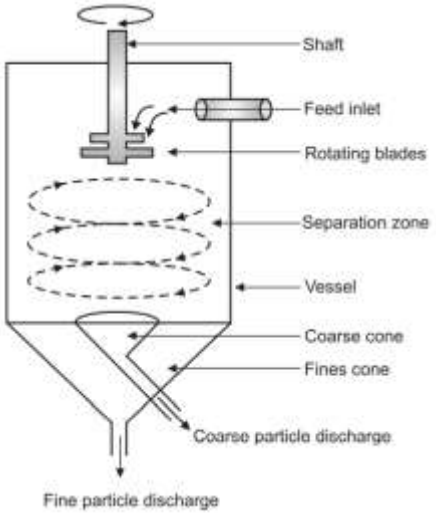
1. Used to separate solids from fluids.

2. Used for separating heavy or coarse fraction from fine dust.

AIR SEPARATOR

PRINCIPLE

The air separator uses the same principle as the cyclone separator. However, for size separation of fine material cyclone separator alone is not efficient. For such separations, an air stream combined with the centrifugal force is used. The finer particles are entrained by the air and the coarser particles are thrown by centrifugal force, which fall into the bottom.



CONSTRUCTION

Air separator consists of a cylindrical vessel having feed inlet at upper part of vessel. The base is conical. A rotating disc and rotating blades are fitted on a shaft placed at the centre of the vessel. Two outlets are provided at the base of the vessel: one is for the finer particles and the other for coarse or heavy particles.

WORKING

The feed (powder) enters the centre of the vessel through the feed inlet. The admitted feed falls on the rotating blade and the rotating disc. The rotating disc produces an air jet in the direction indicated in the diagram. The fine particles are collected by the air stream and brought into the space of the settling chamber, where the air velocity is sufficiently reduced so that the fine particles fall out and are eliminated through the fine particle outlet. Particles too heavy to be collected by the air stream are removed at the outlet of coarse particles.

The main features of Air separator is ease of installation, the rotor speed is adjustable, high product capacity, air flow can be adjusted and easy to maintain.

THANK YOU