UNIT: III **(DRYING)**

CLASS:24

TOPIC: PRINCIPAL, CONSTRUCTION, WORKING, USES, MERITS AND DEMERITS OF FLUIDISED BED DRYER, FREEZE DRYER

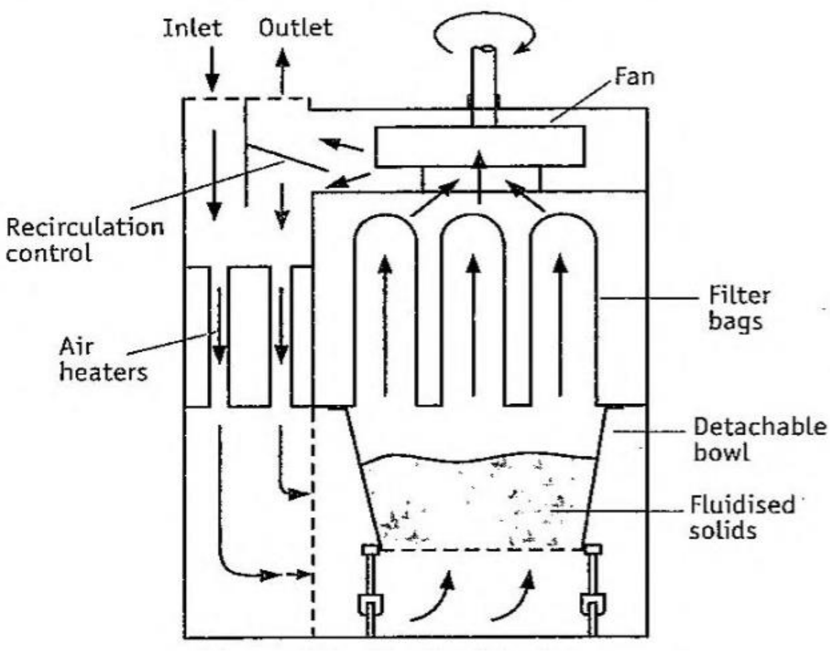
Fluidised Bed Dryer (FBD)

Principle: In fluidised bed dryer, hot air (gas) is passed at high pressure through a perforated bottom of the container containing gran- ules to be dried. The granules are lifted from the bottom and suspended in the stream of air. This condition is called fluidized state. The hot gas is surrounding every granule to completely dry them. Thus, materials or granules are uniformly dried.

Construction: Two types of bed dryers are available, vertical fluid bed dryer and horizontal fluid bed dryer.

The construction of a vertical fluidised bed dryer is shown in Figure. The dryer is made up of stainless steel or plastic. A detachable bowl is placed at the bottom of the dryer, which is used for charging and discharging. The bowl has a perforated bottom with a wire mesh support for placing materials to be dried. A fan is mounted in the upper pan for circulating hot air. Fresh air inlet, prefilter and heat exchanger are connected serially to heat the air to the required temperatures. The temperature of hot air and exit air are monitored. Bag filters are placed above the drying bowl for the recovery of fines.

Working: The wet granules to be dried are placed in the detachable bowl. The bowl is pushed into the dryer. Fresh air is allowed to pass through a prefilter, which subsequently gets heated by passing through a heat exchanger. The hot air flows through the bottom of the bowl. Simultaneously fan is allowed to rotate. The air velocity is gradually increased.



Fluidised bed dryer.

When the velocity of the air is greater than settling velocity of granules, the granules remain partially suspended in the gas stream After some time, a point of pressure is reached at which frictional drag on the particles is equal to the force of gravity. The granules rise in the container because of high velocity gas (1.5 to 7.5 metres per minute) and later fall back in a random boiling motion. This condition is said to be *fluidised state*. The gas surrounds every granule to completely dry them. The air leaves the dryer by passing through the bag filter. The entrained particles remain adhered to the inside surface of the bags Periodically the bags are shaken to remove the entrained particles.

Intense mixing between granules and hot gas provides uniform conditions of temperature, composition and particle size distribution, Drying is achieved at constant rate and falling rate period is very short. Any attempt to increase the air velocity may result in entrainment.

The residence time for drying is about 40 minutes. The material is left for some time in the dryer for reaching ambient temperature. The bowl is taken out for discharging The end product is free flowing

Uses: Fluidised bed dryer is popularly used for drying of granules in the production of tablets. Fluidised bed dryer can be used for three operations such as mixing, granulation and drying. It is modified for coating of granules.

Advantages:

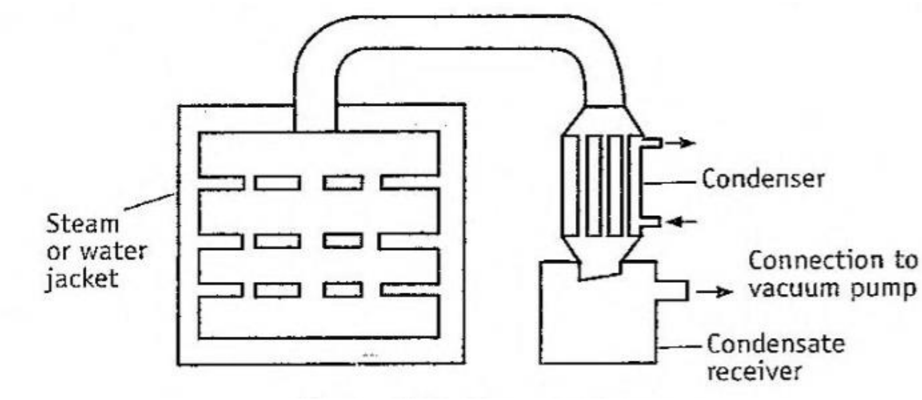
(1) Fluidised bed dryer requires less time to complete drying, i.e., 20 to 40 minutes compared to 24 hours of tray dryer. Handling time is also short. It is 15 fasters than the tray dryer.
(2) It is available in different sizes with the drying capacity ranging from 5 to 200 kg per hour.
(3) The drying containers are mobile, making handling simple and reducing labour costs.
(4) The thermal efficiency is 2 to 6 times than tray dryer.
(5) It is also used for mixing the ingredients and its mixing efficiency is also high.
(6) Hot spots are not observed in the dryer, because of its excellent mixing and drying capacities.
(7) Higher drying temperatures can be used that are not possible in Tray dryer and truck dryer.
(8) It facilitates the drying of thermolabile substances, since the contact time for drying is short.
(9) It can be used either as batch type or continuous type.
(10) It has a high output from a small floor space
(11) The free movement of individual particles eliminates the risk of Soluble material migrating as may occur in static beds.

Disadvantages: Many organic powders develop electrostatic charges during drying. To avoid this, efficient electrical earthing of the dryer is essential. The turbulence of the fluidised state of granules may cause attrition of some materials resulting in the production of fines. But using a suitable binding agent this problem can be solved. Fine particles may become entrained and must be collected by bag filters.

Variants: *Plug flow dryer*: It is a rectangular fluid bed dryer having different compartments for fluidisation. The material is made to move from inlet through different compartments to outlet. Different drying conditions can be maintained in the compartments. Often the last compartment is fluidised with cold gas to cool the solids before dis- charge.

Vacuum Dryer

Principle: In vacuum dryer, material is dried by the application of vacuum. When vacuum is created, the pressure is lowered so that water boils at a lower temperature. Hence, water evaporates faster. The heat transfer becomes efficient, i.e., rate of drying enhances substantially.



Vacuum dryer.

Construction: The construction of a vacuum dryer is shown in Figure. It is made of a cast iron heavy-jacketed vessel. It is so strong that it can withstand high vacuum within the oven and steam pressure in the jacket. The enclosed space (approximately 1.5 metre cube) is divided into a number of portions by means of 20 hollow shelves, which are part of the jacket. These shelves provide larger surface area (about 45 to 50 metre square) for conduction of heat. Over the shelves, metal trays are placed for keeping the material. The oven door can be locked tightly to give an air tight seal. The oven is connected to a vacuum pump by placing condenser in between.

Working: The material to be dried is spread on trays. The trays are placed on the shelves. Door is closed firmly. Pressure is decreased up to 30 to 60 kilopascals by means of a vacuum pump. Door is closed firmly. Steam or hot air is supplied into the hollow space of jacket and shelves. Heat transfer by conduction takes place. At this vacuum. Evaporation of water from the material takes place at 25-30 °C, on account of lowering of boiling point. Water vapour passes into the condenser where condensation takes place.

At the end of the drying, vacuum line is disconnected. The material is collected from the trays.

Uses: Vacuum dryer can be used for drying of the following.

(1) Heat sensitive materials, which undergo decomposition.
(2) Dusty and hygroscopic materials. Drugs containing toxic solvents. These can be separated into closed containers,
(3) Feed containing valuable solvents. These are recovered by condensation.
(4) Drugs which are required as porous end products.
(5) Friable dry extracts.

Advantages:

(1) Vacuum dryer provides large surface area for heat transfer.
(2) Handling of the material, trays and equipment is easy
(3) It is easy for switching over to the next materials.
(4) Hot water of desired temperatures can be supplied.
(5) Electrically heated hollow shelves can be used.

Disadvantages:

(1) In vacuum dryer, heat transfer coefficients are low.
(2) It has a limited capacity and used for batch process.
(3) It is more expensive than tray dryer. Labour and running costs are also high.

(4) Sometimes, there is a danger of over heating as the material is in contact with steam heated surface for longer period.

Freeze Dryer

Freeze drying is also known as *lyophilization*, i.e., system is made solvent loving for removing the same.

Principle: In freeze drying, water is removed from the frozen state by *sublimation*, i.e., direct change of water from solid into vapour without conversion to a liquid phase. Solid-liquid-vapour equilibrium phase diagram of water is useful to decide the experimental conditions. The drying is achieved by subjecting the material to temperature and pressures below the triple point (in practice, below eutectic temperature is essential). Under these conditions, any heat transferred is used as latent heat and ice sublimes directly into vapour state. The water vapour is removed from the system my condensation in a cold trap maintained at a temperature lower than the frozen material.

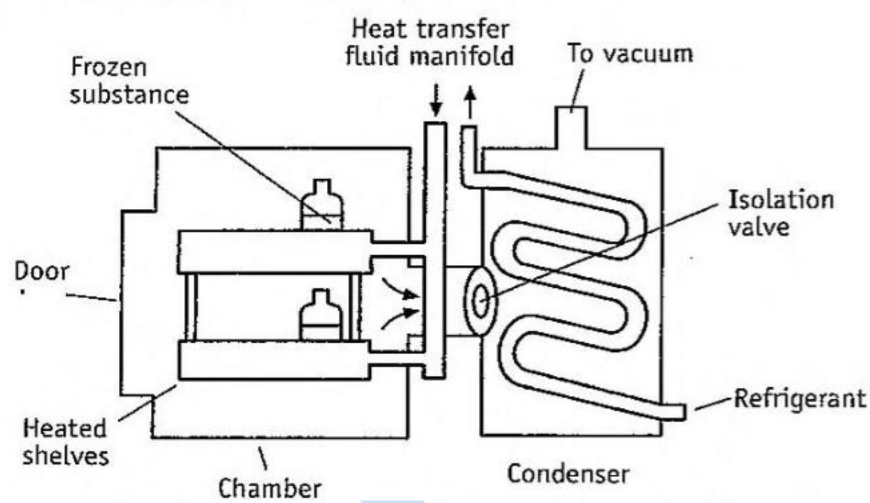
Construction: The construction of a freeze dryer is shown in Figure. It consists of:

(1) Drying chamber in which trays are loaded.

(2) Heat supply in the form of radiation source, heating coils.

(3) Vapour condensing or adsorption system.

(4) Vacuum pump or steam ejector or both.



Freeze Dryer

The chamber for vacuum drying is generally designed for batch operation. It consists of shelves for keeping the material. The distance between Subliming surface and condenser must be less than the mean path of molecules. This increases the rate of drying. The condenser consists of a relatively large surface cooled by solid carbon dioxide slurred with acetone or ethanol. The temperature of the condenser must be much lower than the evaporating surface of frozen substance. In order to maintain this condition, the condenser surface is cleaned repeatedly.

Working : The working of freeze dryer consists of the following steps.

(1) Preparation and pre-treatment
(2) Prefreezing to solidify water
(3) Primary drying (sublimation of ice under vacuum)

(4) Secondary drying (removal of residual moisture under high vacuum)
(5) Packing

Preparation and pre-treatment: The volume of solution introduced into the container is limited by its capacity. Satisfactory freeze drying beyond a certain limit of depth of liquid is not possible. Therefore, pre-treatment is essential. The solution is pre-concentrated under normal vacuum tray drying. This reduces tire actual drying by 8 to 10 times. The final product becomes more porous. Liquid or solid desiccants are also used for this purpose.

Prefreezing to solidify water: Vials, ampoules or bottles in which the aqueous solution is packed are frozen in cold shelves (about -50 °C) During this stage, cabinet is maintained at low temperature and atmospheric pressure. The normal cooling rate is about 1 to 3 K/min so that large ice crystals with relatively large holes are formed on sublimation of ice. This is also responsible for giving a porous product.

Primary drying (sublimation of ice under vacuum) : In this step the material to be dried is spread as much large surface as possible for sublimation. The temperature and pressure should be below the triple point of water, i.e. 0.0098 °C and 0.533 kPa, (4.58 mmHg) for the sublimation when water alone is present.

When a solution of solid is dried, the depression of freezing point of water occurs. Hence, it is essential that the temperature be brought below the eutectic point. The pressure and temperature at which the frozen solid vaporises without conversion to a liquid is referred to as the eutectic point Depending on the drug substance dissolved in water, the eutectic point is determined. The usual range is from -10 °C to 30 °C. The condition of 1 to 8 °C below eutectic point is sufficient.

Vacuum is applied to the tune of about 3 mmHg (0.4 kPa) on the frozen sample. The temperature is linearly increased to about 30 °C m a span of 2 hr.

Heat (about 2900 kj/kg) is supplied which transfers as latent heat and ice sublimes directly into vapour state. The heat controls the movements of ice layer inwards. It has to be controlled in such a manner so as to get highest possible water vapour at ice surface without melting the material as soon as vapour molecules are formed, these are removed. The overall driving force is the temperature difference (also vapour pressure difference) between evaporating surface and condenser.

As the drying proceeds, thickness of the frozen layer decreases and the thickness of partially dried solids increases. Primary drying stage removes easily removable moisture. During this stage, about 98 to 99 % water is removed. Still traces of moisture is present in the sample.

Secondary drying (Removal of residual moisture under high vacuum): During this stage, traces of moisture is removed. The temperature of the solid is raised to as high as 50 to 60 °C, but pressure is lowered below that is used in primary drying (50 mmHg). The rate of drying is very low and it takes about 10 to 20 h.

Packing: After vacuum is replaced by inert gas, the bottles and vials are closed.

Uses: Freeze dryer is most commonly used in the production of dosage forms, such as injections, solutions and suspensions. It is used for drying of a number of products.

- Blood plasma and its fractionated products.
- Bacterial and viral cultures.
- Human tissue (arteries and corneal tissue).
- Antibiotics and plant extracts.

- Steroids, vitamins and enzymes.

Several other products such as food items (prawns, mushrooms, meat and poultry products), coffee and tea concentrates and citrus fruit juices are dried.

Advantages: The entire operation is earned out well below the freezing point. This offers several advantages.

(1) Thermolabile materials (heat sensitive materials) can be dried.
(2) The product retains its bulk volume. It is porous and uniform. The reconstitution of the material is easy.
(3) Denaturation does not occur.
(4) Migration of salts and other solutes does not take place.
(5) Loss of volatile material is less.
(6) Moisture level can be kept as low as possible without decomposition.
(7) Material can be dried in its final container such as single dose and multiple dose vials.
(8) Sterility can be maintained.
(9) The final product can be stored at ambient temperature, if well sealed by providing inert atmosphere.

Disadvantages

(1) The product is prone to oxidation, due to high porosity and large surface area. Therefore, the product should be packed in vacuum or using inert gas or in a container impervious to gases.
(2) Equipment and running costs are high.
(3) It is difficult to adopt the method for solutions containing non- aqueous solvents.
(4) The period of drying is high (rarely less than 10 h). Time cannot be shortened.

MCA Each carry 1 mark

(1) Fluidised bed dryer has one of the following advantages?
   a. Attrition is not observed
   b. **Entire material is continuously exposed to a heat source**
   c. Fluffy mass is formed
   d. Humidity can be increased
(2) Which one of the following dryer is known as lyophiliser?
   a. Fluidised bed dryer
   b. Freeze dryer
   c. Spray dryer
   d. Vacuum dryer
(3) Migration of salts & solutes does not occur in one of the following equipment for drying
   a. Freeze dryer
   b. Spray dryer
   c. Tray dryer
   d. Vacuum dryer
(4) Which materials are not used in drying in a freeze dryer?
   a) Seafood
   b) Fruits
   c) Pharmaceuticals
   d) Dyes
(5) Eutectic point is an important factor for one of the following drying processes?

   a. Drum drying
   b. Fluidised bed drying
   c. Freeze drying
   d. Tray drying

(6) High vacuum is applied in one of the following drying processes?
   a. Drum drying
   b. Freeze drying
   c. Spray drying
   d. Tray drying

(7) Low temperature drying is obtained in one the following drying process-
   a. Drum drying
   b. Fluidised bed drying
   c. Lyophilization
   d. Tray drying

(8) Fluidised bed dryer can be utilised for the following operations.
   a. Drying
   b. Mixing and granulation
   c. Mixing, granulation and drying
   d. Mixing, granulation, coating and drying

Each carry 2 marks

(1) Suggest a method of drying in which containers (product) can be directly used. Explain the procedure.
(2) Suggest a drying method, which permits the handling of product at ambient conditions.
(3) Give the equipment which produces overheating during drying with reasons.
(4) Which is the dryer used for multiple operations: spray dryer or fluidized bed dryer? Give the operations.

Each carry 5 marks

(1) Explain the principle of freeze drying. What are its applications in pharmacy?
(2) List the pharmaceutical applications of freeze-drying process. Give salient features of the process.
(3) Describe the construction and working of a fluidized bed dryer.
(4) Explain the operation and applications of fluidised bed dryer.
(5) Describe the principle with the help of a labelled diagram of fluidised bed dryer.

Each carry 10 marks

(1) Explain the construction and operational details of freeze dryer. Describe its applications in pharmacy.
(2) Explain the construction and operational details of fluidized bed dryer. Describe its applications in pharmacy.