# Drying

- **Definition:**
  Drying is the process of removal of small amount of liquid(water/volatile liq./moisture) by application of heat to obtain dry solid or solid product.
- In general drying process involves to operations; 1) Heat transfer 2) Mass transfer
- Drying and Evaporation are relatively same term but are distinguish in terms of removal of water. Here are some differences between drying and evaporation;



| | Drying | Evaporation |
|---|---|---|
| 1. | In drying processes, the main operation usually carried out on solid materials, e.g. powders, or products | In evaporation processes, the main operation usually carried out on liquid materials, e.g. solution, or products |
| 2. | Drying in most of the cases means the removal of relatively small amounts of water from solids. | Evaporation include the removal of large amounts of water from solutions. |
| 3. | Drying involves the removal of water at temperatures below its boiling point. | Evaporation involves the removal of water by boiling a solution. |
| 4. | In drying , water is usually removed by circulating air over the material in order to carry away the water vapour | While in evaporation , water is removed from the material as pure water vapour mixed with other gases. |

## Applications of drying:

- Preservation of drug products
- Preparation of bulk drugs
- Improved handling
- Improved characteristics
- Reduction in transport cost
- Purification of crystalline products
- Prevention of corrosion



## Preservation of drugs:

- Drying is necessary in order to avoid deterioration. A few examples are...
- blood products, tissues undergo microbial growth
- effervescent tablets, synthetic & semi synthetic drugs undergo chemical decomposition.



## Preparation of bulk drugs:

- Drying is the final stage of processing .
- Eg: dried aluminium hydroxide
- spray dried lactose
- powdered extracts

## Improved characteristics:

- Drying produces materials of spherical shape, uniform size, free flowing & enhanced solubility.

1. Granules are dried to improve the fluidity & compression characteristics. These are essential for production of tablets and capsules.
2. Viscous & sticky materials are not free flowing, Drying modifies these characteristics.

## Improved handling:

- Removal of moisture makes the material light in weight and reduces bulk.
- Thus cost of transportation will be less & storage will be efficient.
- If moisture is present, size reduction of drugs is difficult.
- Drying reduces the moisture content.



## Factors affecting rate of drying:

- Particle size
- Nature of material
- Nature of moisture (bound/unbound)
- Surface area
- Initial and final moisture content
- Thickness of material bed
- Temperature
- Amount of moisture
- Nature of product



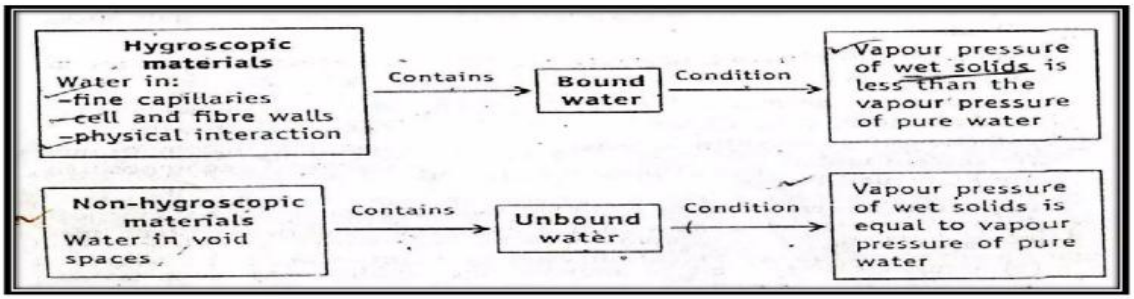
## Theory of drying:

- In a wet solid mass water may be present in two forms;
- 1) Bound water:

  Bound water is the minimum water held by the material that exerts an equilibrium vapour pressure **less than** the **pure water** at the same temperature.
- 2) Unbound water:

  It is the amount of water held by the material that exerts an equilibrium vapour pressure **equal** to that of **pure water** at the same temperature.

- Unbound water exists largely in the voids of solid thus in non- hygroscopic material all the liquid is unbound water.






- Theory of drying can be discussed under two headings
- A) Equilibrium relationships
- B) Rate relationships

A) Equilibrium relationships:

- Air of constant humidity and temperature is passed over wet material after long exposure equilibrium is reached.

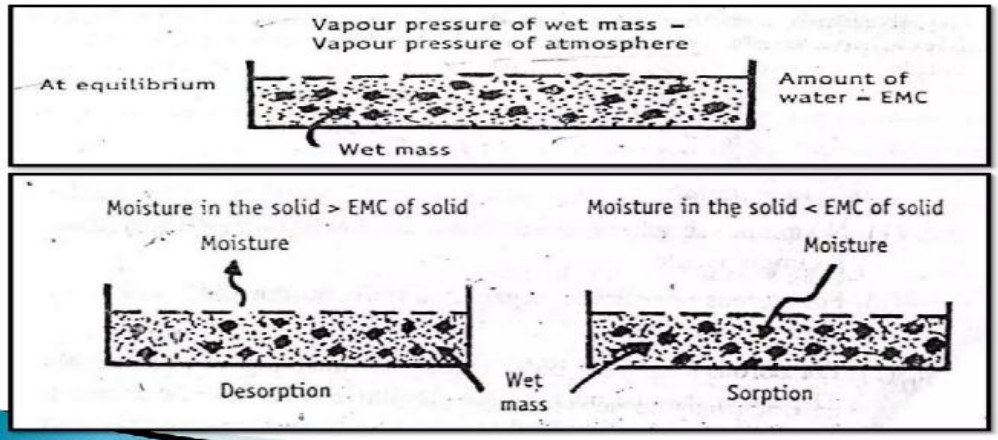
- Equilibrium moisture content (EMC):

  It is amount of water which exerts vapour pressure equal to the vapour pressure of atmosphere surrounding it.



- Based on the conditions of temperature and humidity solid will either lose or absorb the moisture;
- **1)** When air is continuously passed over the solid containing moisture more than EMC then solid **lose water** till the EMC is reached. This phenomenon is known as **Desorption.**
- **2)** When air is continuously passed over the solid containing moisture less than EMC then solid **absorb water** till EMC is reached. This phenomenon is known as **Sorption.**

**Moisture in solid>EMC=desorption (lose water)**
**Moisture in solid<EMC=sorption (gain water)**






- Free moisture content (FMC): It is the amount of water that is free to evaporate from solid.

  FMC = Total water content − EMC

B) Rate relationships:

- Rate relationship is observed by considering a simple model which mimic the conditions of a dryer. In this model wet slab of solid is considered and hot humid air is passed over it. The change in weight is determined by weighing the slab at different time interval and following calculations are made;



$$\%\ Loss\ on\ drying\ (LOD) = \frac{\text{mass of water in sample (kg)}}{\text{total mass of wet sample (kg)}} \times 100$$

$$\%\ Moisture\ content\ (MC) = \frac{\text{mass of water in sample (kg)}}{\text{mass of the dry sample (kg)}} \times 100$$

$$Drying\ rate = \frac{\text{weight of water in sample (kg)}}{\text{time (h)} \times \text{weight of the dry solid (kg)}}$$

- From the data obtaind by the above experiment graph is plotted by taking FMC on x-axis and drying rate on y-axis.
- The curve obtained is known as **drying rate curve.**

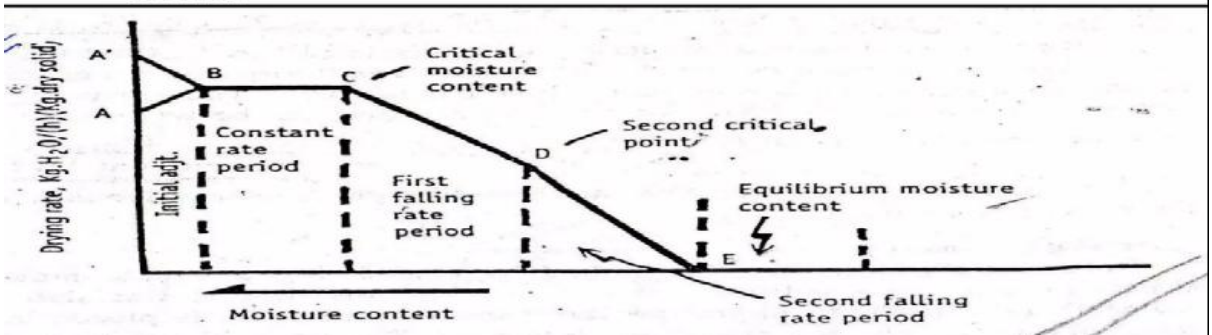



Figure 14-2. FMC curve.

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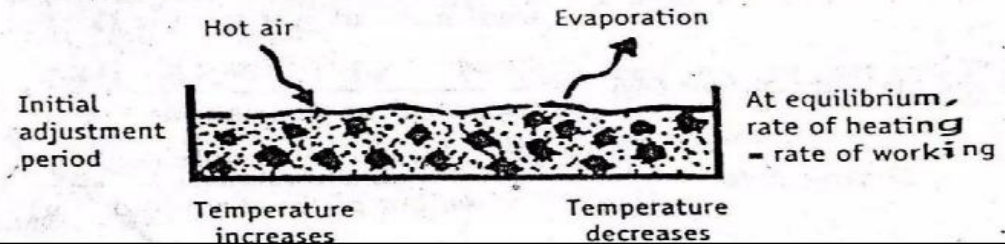
(1) The time corresponding to AB represents the *initial adjustment period*.. During this period, the solids absorb heat and the temperature increases. At the same time, the moisture begins to evaporate and thus tends to cool the drying solid. After some time, the temperature stabilises (heating and cooling rates become equal). This temperature is equal to the wet bulb temperature of the drying air and is referred by the point B.

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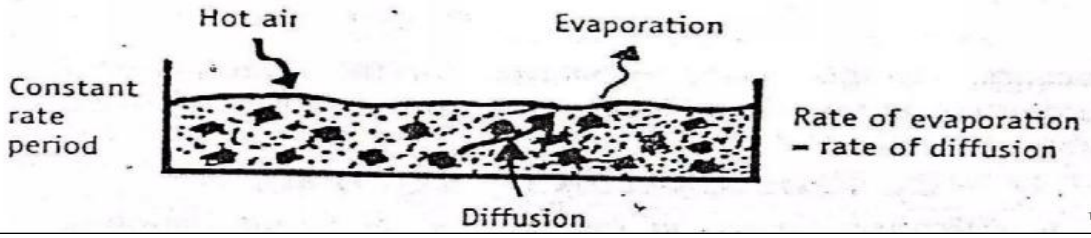
(2) The time corresponding to BC represents the *constant rate period*. The temperature remains constant and rate of drying is constant. The moisture evaporating from the surface is replaced by the water diffusing from the interior of the solid. The rate of diffusion is equal to the rate of evaporation. The moisture content at the end of constant rate (point C) is referred to as the *critical moisture content* (CMC).

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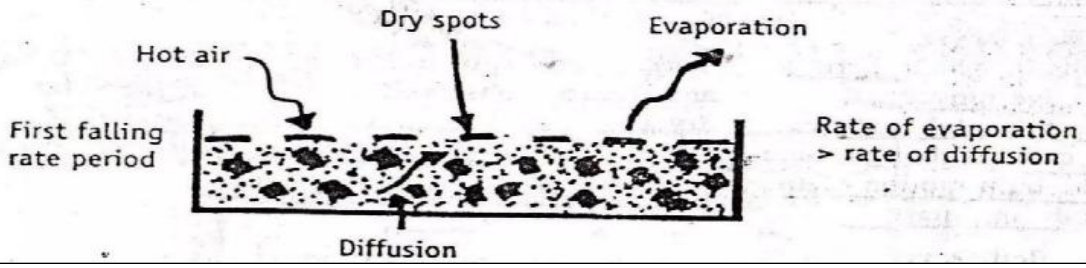
(3) The time corresponding to CD represents the *first falling rate period* (or unsaturated surface drying). During this period, the surface water is no longer replaced at a rate fast enough to maintain a continuous film on the surface. Dry spots begin to appear and the rate of drying begins to fall off. The point D is referred to as the *second critical point.* At this point, the film of surface water is completely evaporated.

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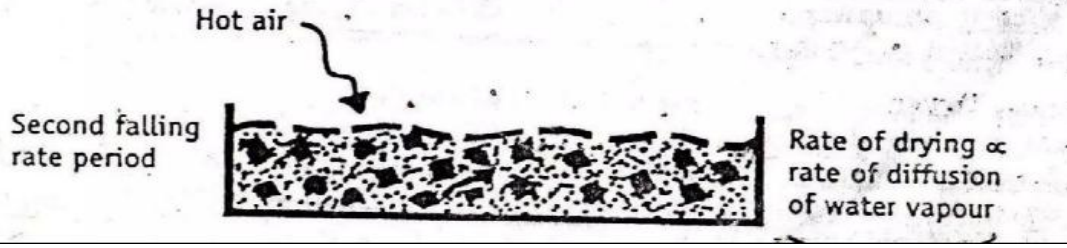
(4) The time corresponding to DE represents the *second falling rate period.* During this period, the rate of drying falls even more rapidly than the first falling rate. During this period, the rate of drying is dependent on the rate of diffusion of vapour of moisture to the surface of the solid. Point E is referred to as the *equilibrium moisture content.*

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(5) Beyond E, the drying rate is equal to zero. Therefore, temperature and moisture content remain constant. Beyond, E, continued drying is waste of time and energy.

The curves may have different shapes for different levels of moisture.If the drying is carried above the level of CMC, only constant rate period occurs.

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## Equations for drying theory:

- Drying process involves both heat transfer and mass transfer operations. It can be understood more easily if the film of liquid at the surface of material being dried.
- The rate of evaporation of this film is related to the heat transfer by following equation;

$$dW/d\theta = q/\lambda \quad \text{-----} \quad (1)$$

Where,

- $dW/d\theta$ = rate of evaporation of water per hour
- $q$ = overall rate of heat transfer
- $\lambda$ = latent heat of vaporization of water



- The rate of diffusion of moisture into air stream is expressed by similar to heat transfer where driving force is humidity difference. The rate equation is given as;

$$dW/d\theta = k'A(Hs-Hg) \quad \text{-----} \quad (2)$$

Where,

- $k'$ = coefficient of mass transfer (it is not constant, but varies with velocity of passing air stream)
- $A$ = area of evaporating surface
- $Hs$ = absolute humidity at evaporating surface
- $Hg$ = absolute humidity in passing air stream



- The coefficient of mass transfer k' is not constant the relationship is in the form:

$$k' = cG^n \quad \text{-----} \quad (3)$$

Where,

- $c$ = proportionality constant
- $G$ = rate of flow of air
- $n$ = fractional exponent, usually about $(0.8)^2$

- After an initial adjustment period the rate of evaporation is equal to the rate of diffusion of water and the rate of heat transfer so,

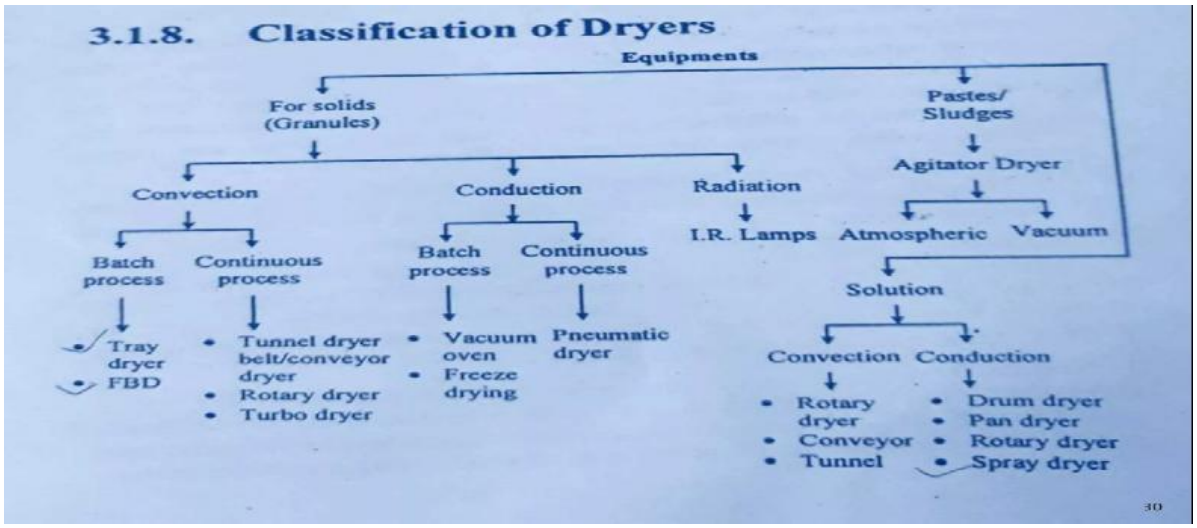
$$dW/d\theta = q/\lambda = k'A(Hs-Hg) \quad \text{-----} \quad (4)$$

- If the overall rate of heat transfer is expressed as the sum of heat transfer by conduction, convection and radiation eq. 4 is expanded to;

$$dW/d\theta = (Qk + Qc + Qr)/\lambda$$
$$= k'A(Hs - Hg) \quad \text{-----} \quad (5)$$

- The rate of drying may be accelerated by increasing any of the individual term in eq.(5)
- Heat must be transferred to the material to be dried in order to supply the latent heat required to remove moisture. Water diffuses through the material to the surface and subsequently evaporates in to air stream. Thus **drying involves** both **heat transfer** and **mass transfer** operations simultaneously.






## List of equipments used for drying:

- Drum dryer,
- Spray dryer,
- Fluidised bed dryer,
- Tray dryer,
- Tunnel dryer,
- Vacuum dryer,
- Microwave,
- Radiant heat dryer (infra red)
- Rotary dryer
- Freeze dryer

# Drum dryer/Roller dryer/ Film dryer:

- Principle:

  In drum dryer heated hollow cylindrical drum is rotate on longitudinal axis,which is dipped into the solution to be dried.

  The solution is carried as a film on the surface of the dryer and dried to form a layer. Dried material is removed with the help of the knife.



- **Construction:**
  - It consist of horizontally mounted **hollow steel drum** of **0.6 to 3m** diameter and **0.6 to 4m** length.
  - Below the drum **feed pan** is placed in such a way that the drum dips partially into the feed.
  - One side of the drum **spreader** and other side **knife** is placed.
  - **Storage bin** is placed to collect the material

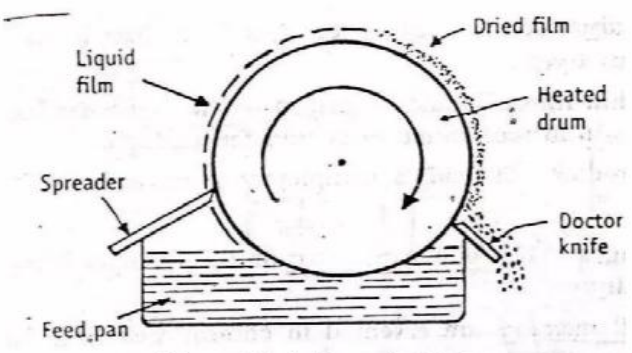


Figure 14-4. Drum dryer.



- **Working:**

Steam is passed inside the drum

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Drum is rotated at 1–10 rotations per minute

↓

The liquid present in feed pan adheres to the surface of drum.

↓

Material is dries during the rotation of drum and collected in storage bin by using knife.

**Note: Drying procees in drum dryer completes in less than one cycle so the time of contact of matireal with drum is only 6 to 15seconds**

**Uses :** Drum dryer is used for drying solutions, slurries, suspensions etc. The products dried are milk products, starch products, ferrous salts, suspensions of zinc oxide, suspension of kaolin, yeast, pigments, malt extracts, antibiotics, glandular extracts, insecticides, DDT, calcium and barium carbonates.

**Advantages :** (1) In drum dryer, drying time is less, only a few seconds. Therefore, heat sensitive materials can be dried.

(2) Drum dryer occupies less space, as it is compact when compared to spray dryer.

(3) As a thin film of liquid is formed on the large heating surface, rates of heat transfer and mass transfer are high.

(4) The product obtained is completely dried and is in the final form.

**Disadvantages :** (1) Maintenance cost of a drum dryer is higher than spray dryer.

(2) Skilled operators are essential to control feed rate, film thickness, speed of rotation and temperature.

(3) It is not suitable for solutions of salts with less solubility.

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# Tray dryer:

- **Principle:**
  In tray dryer hot air is continuously passed over wet mass. Heat transfer takes place by forced convection.
- **Construction:**
  It consist of **rectangular chamber** whose wall is insulated. **Trays** are placed in the chamber according to need.

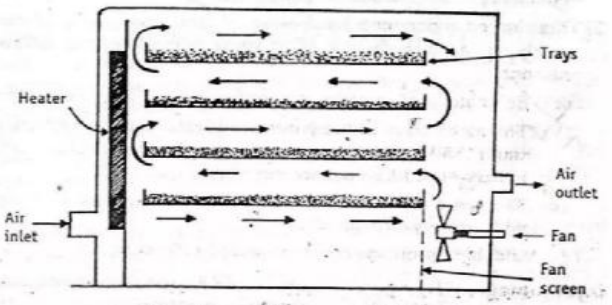

Figure 14-3. Tray dryer.

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- **Working:**

- Wet solid is loaded into trays. Trays are placed in the chamber.
- Fresh air is introduced through inlet which passes through the heaters and heated up.
- The hot air is circulated by means of fans at 2 to 5 meter per second.
- The water is picked up by air and moist air is removed from outlet.
- During the cycle of drying only 10 to 20 % of fresh air is introduced and 80 to 90% air is circulated back.

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**Uses :** Sticky materials, plastic substances, granular mass or crystalline materials, precipitates and pastes can be dried in a tray dryer. Crude drugs, chemicals, powders, tablet granules or parts of equipment are dried.

**Advantages :** (1) In tray dryer, handling of materials (loading and unloading) can be done without losses.

(2) Tray dryer is operated batch-wise. Batch drying is used extensively in the manufacture of pharmaceuticals for the following reasons.

(a) Each batch of material can be handled as a separate entity.

(b) The batch sizes in the pharmaceutical industry are relatively small (250 kg or less per batch) compared with the chemical industry (1000 kg or more per hour).

(c) The same equipment is readily adjusted for use in drying a wide variety of materials.

(d) Valuable products can be handled efficiently.

**Disadvantages :** Tray dryer requires more labour to load and unload. Hence, cost increases. The process is time consuming.

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# Spray dryer:

- **Principle:**

- In spray dryer the fluid to be dried is atomized into fine droplets which are thrown radially into moving stream of hot gas.
- The temperature of the droplets is immediately increased and fine droplets get dried instantly in the form of spherical particles.

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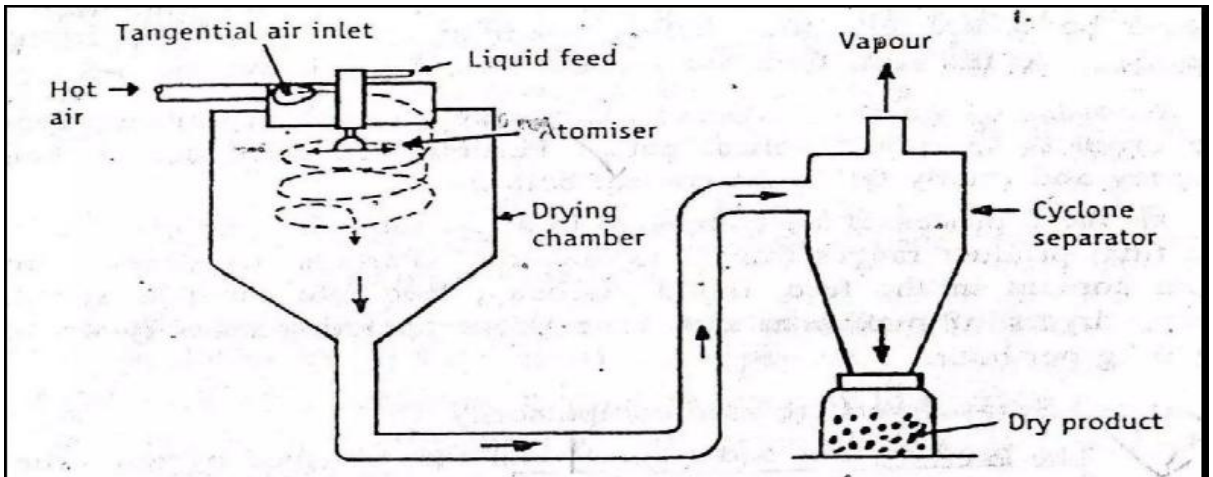


Figure 14-5. Spray dryer.

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- **Construction:**

- It consist of large cylindrical drying chamber with a short conical bottom made up of stainless steel.(diameter 2.5 to 9.0 m and height 25 m or more)
- An inlet for hot air placed in the roof and another inlet carrying spray disk atomizer is also set in the roof.
- The spray disk atomizer is about 300 mm in diameter and rotates at a speed of 3000 to 50,000 rpm.
- Bottom of the dryer is connected to cyclone separator.

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- **Working:**

- Drying of material in spray dryer involves 3 stages;

1. Atomization of liquids: The feed is introduced through the atomizer either by gravity or using suitable pump.
2. Drying of the liquid droplets: Fine droplets are dried in the drying chamber by supplying hot air through the inlet.
3. Recovery of the dried product: Centrifugal force of atomizer drives the droplets to follow helical path. Particles are dried and collected at the conical bottom.

- All these processes are completed in few seconds. Particle size obtained is ranging from 2 to 500 mm. maximum size of spray dryer has capacity of 2000 kg per hour.

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**Uses :** Spray dryers are used compulsorily, if:

(1) The product is a better form than that obtained by any other dryer.

(2) The quantity of the material to be dried is large.

(3) The product is thermolabile, hygroscopic or undergoes chemical decomposition.

**Advantages :** (1) Spray drying is a continuous process and drying is very rapid. Drying completes within 3 to 30 seconds.

(2) Labour costs are low as it combines the function of an evaporator, a crystllizer, a dryer, a size reduction unit and a classifier.

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(3) By using suitable atomizer, the product of uniform and controllable size can be obtained. Free flowing product of uniform spheres is formed which is very convenient for tabletting process.

(4) Fine droplets formed provide large surface area for heat and mass transfer. Product shows excellent solubility.

(5) Either the solution or suspension or thin paste can be dried in one step to get the final product ready for package.

(6) It is suitable for the drying of sterile products.

(7) Reconstituted product appears more or less similar to the fresh material.

(8) Globules of an emulsion can be dried with the dispersed phase inside and layer of the continuous phase outside. On reconstitution, the emulsion will be formed.

**Disadvantages :** (1) Spray dryer is very bulky (height of 25.0 metres and diameter of 9.0 metres) and expensive.

(2) Such a huge equipment is not always easy to operate.

(3) The thermal efficiency is low, as much heat is lost in the discharged gases.

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# Fluidised bed dryer:

- Principle:
- In fluidised bed dryer hot air is passed at high pressure through a perforated bottom of the container.
- The granules are lifted from the bottom and suspended in the stream of air, this condition is called as fluidized state.
- The hot gas surrounding every granule to completely dry them.

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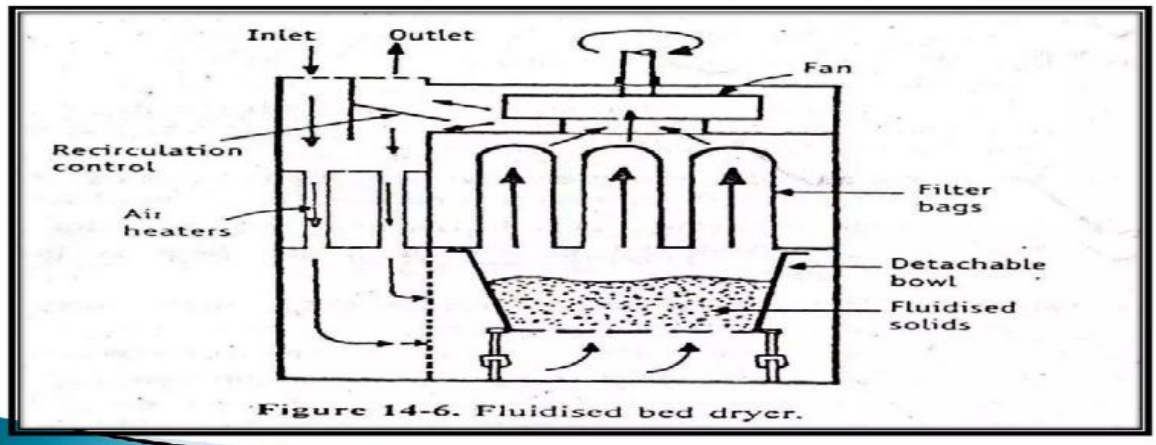


Figure 14-6. Fluidised bed dryer.

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- Construction:
- Fluidised bed dryer is available in two forms;
- i) Horizontal FBD ii) **Vertical FBD**
- The dryer is made up of **SS** or **plastic.**
- A **detachable perforated bowl** is placed at the bottom of the dryer which is used for charging and discharging of material.
- A **fan** is mounted in the upper part for circulating hot air.
- **Fresh air inlet, pre-filter and heat exchanger** are connected serially to the required temperature.
- **Bag filters** are placed over bowl for recovery of fines.



- Working:
- The wet granules to be dried are placed in detachable bowl and bowl is placed into the dryer.
- Fresh air is allowed to pass through pre-filter which subsequently gets heated by passing through heat exchanger.
- Hot air passed through bottom of bowl and fan is allowed to rotate.
- The granules rise in the container because of high velocity gas and later fall back in random motion.



- The gas surrounds every granule to completely dry them. The air leaves the dryer by passing through bag filter.
- The entrained particle remain adhere to the inside surface of bags. Periodically the bags are shaken to remove the particles.
- The material is collected after some time so as to reach the ambient temperature.

- Uses:
- For drying granules.
- It can be used for drying, mixing and granulation process.
- 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 times faster 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.

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- Disadvantages:
- Some organic material may develop electrostatic charge so requires earthing.
- Due to attrition fines are observed in large quantity.

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# 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 lower temperature. Hence evaporate faster.

- Construction:
- It is made up of cast iron heavy jacketed vessel.
- The enclosed space (approx. 1.5 meter cube) is divided into a number of portions by means of 20 hollow shelves which are part of jacket.

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- These shelves provide larger surface area (about 45 to 50 $m^2$) for conduction of heat. Over these shelves metal trays are placed.
- The oven is connected to vacuum pump by placing condenser in between.

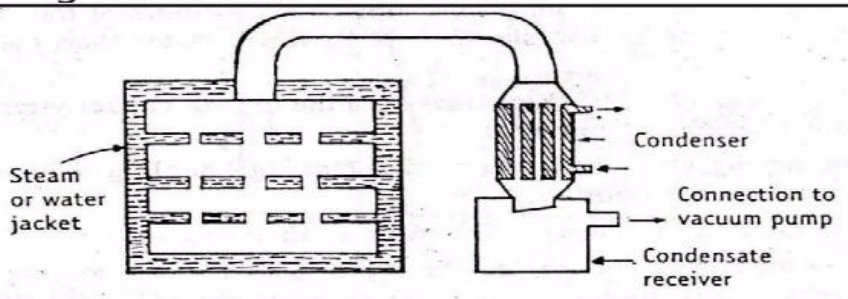


Figure 14-7. Vacuum dryer.



- Working:
- Material to be dried is placed on trays.
- The trays are placed on shelves and pressure is decreased up to 30 to 60 kilopascals.
- Steam or hot air is supplied into hollow space. Drying of material is done by presence of vacuum.
- Water vapour passes into the condenser where it is condensed.
- At the end of drying vacuum line is disconnected and material is collected from trays.



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

(1) Heat sensitive materials, which undergo decomposition.

(2) Dusty and hygroscopic materials.

(3) Drugs containing toxic solvents. These can be separated into closed containers.

(4) Feed containing valuable solvents. These are recovered by condensation.

(5) Drugs which are required as porous end products.

(6) 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:

- Principle:

In freeze drying water is removed from the frozen state by sublimation. (direct change of water from solid into vapour)

- Construction:

Freeze dryer is consist of

1) Drying chamber in which trays are loaded
2) Heat supply in the form of radiation
3) Vapour condensing or adsorption system
4) Vacuum pump or steam jacket or both



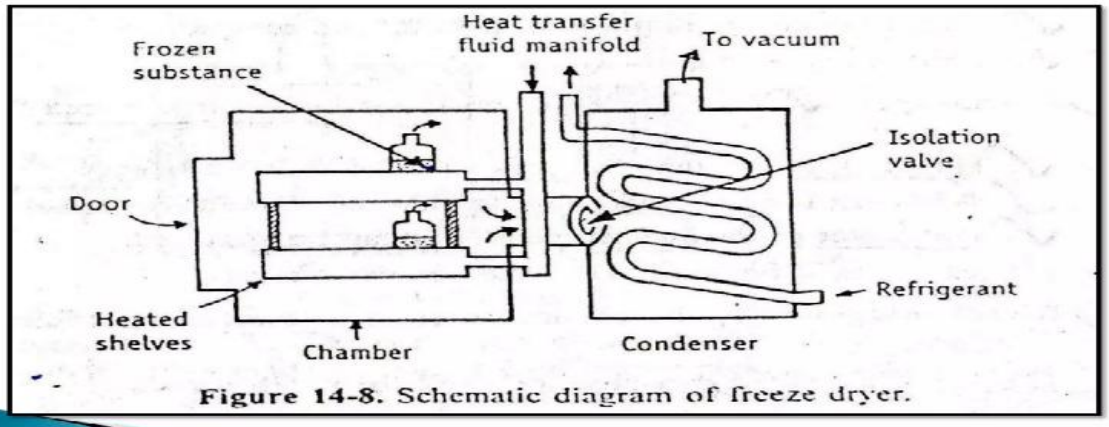


**Figure 14-8.** Schematic diagram of freeze dryer.

- Working:

It involves following steps;

1) Preparation and pretreatment
2) Pre-freezing for solidifying water
3) Primary drying (sublimation of ice)
4) Secondary drying (removal of residual moisture)
5) Packing



*Preparation and pretreatment* : 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 pretreatment is essential. The solution is pre-concentrated under normal vacuum tray drying. This reduces the 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 Kelvin per minute 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 kilopascals, (4.58 mmHg) for the sublimation, when water alone is present.



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

Heat (about 2900 kilojoules per 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

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 vacuum 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 hours.

*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.

(1) Blood plasma and its fractionated products.
(2) Bacterial and viral cultures.
(3) Human tissue (arteries and corneal tissue).
(4) Antibiotics and plant extracts.
(5) 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 carried 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 hours). Time cannot be shortened.