14

Drying

Theory of Drying
Classification of Drying Equipment
Equipment

Drying is defined as the removal of small amounts of water or other liquid from a material by the application of heat.

The liquid medium may be removed from the solids mechanically by a filter press or centrifuge or thermally by vaporisation. It is cheaper to remove liquid by mechanically than thermally. Therefore, it is advisable to reduce the liquid as much as possible before subjecting to the drying process.

Drying and evaporation are distinguishable by the relative quantities of liquid removed from the solid. In evaporation, the product obtained is either concentrated solution or suspension or wet slurry. In drying, dry solid is the product.

The liquid to be vaporised may remain:

- on the surface of the solid, as in drying of salt crystals,
- entirely inside the solid, as in solvent removal from a sheet of polymer,
- partly outside and partly inside, as in case of solvent removal from pharmaceutical powders.

Drying is possible when the environment is unsaturated with the water vapour. Hence, humidity in the environment is an important determinant for drying of the solids. Though several methods are available, thermal methods and freeze drying technique (nonthermal method) are included here. This chapter deals with the theoretical principles and equipment employed for drying.

Applications

Preparation of bulk drugs : In the preparation of bulk drugs, drying is the final stage of processing. A few examples are:

- dried aluminium hydroxide
- spray dried lactose
- powdered extracts

Drying step is essential after certain operations such as crystallization and filtration.

Preservation of drug products : Drying is necessary in order to avoid deterioration. A few examples are:

Crude drugs of animal and vegetable origin	- chemical decomposition
Blood products, skin, tissue	- microbial growth
Synthetic and semisynthetic drugs	- chemical decomposition
Effervescent tablets (aspirin, penicillins)	- chemical decomposition

Improved characteristics : Drying produces materials of spherical shape, uniform size, free flowing and enhanced solubility. Some specific areas of importance are:

(1) Granules are dried to improve the fluidity and compression characteristics. These are essential for the production of tablets and capsules.

(2) Viscous and sticky materials are not free flowing. Drying modifies these characteristics. Examples are male fern extract, malt extract and oleoresin.

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

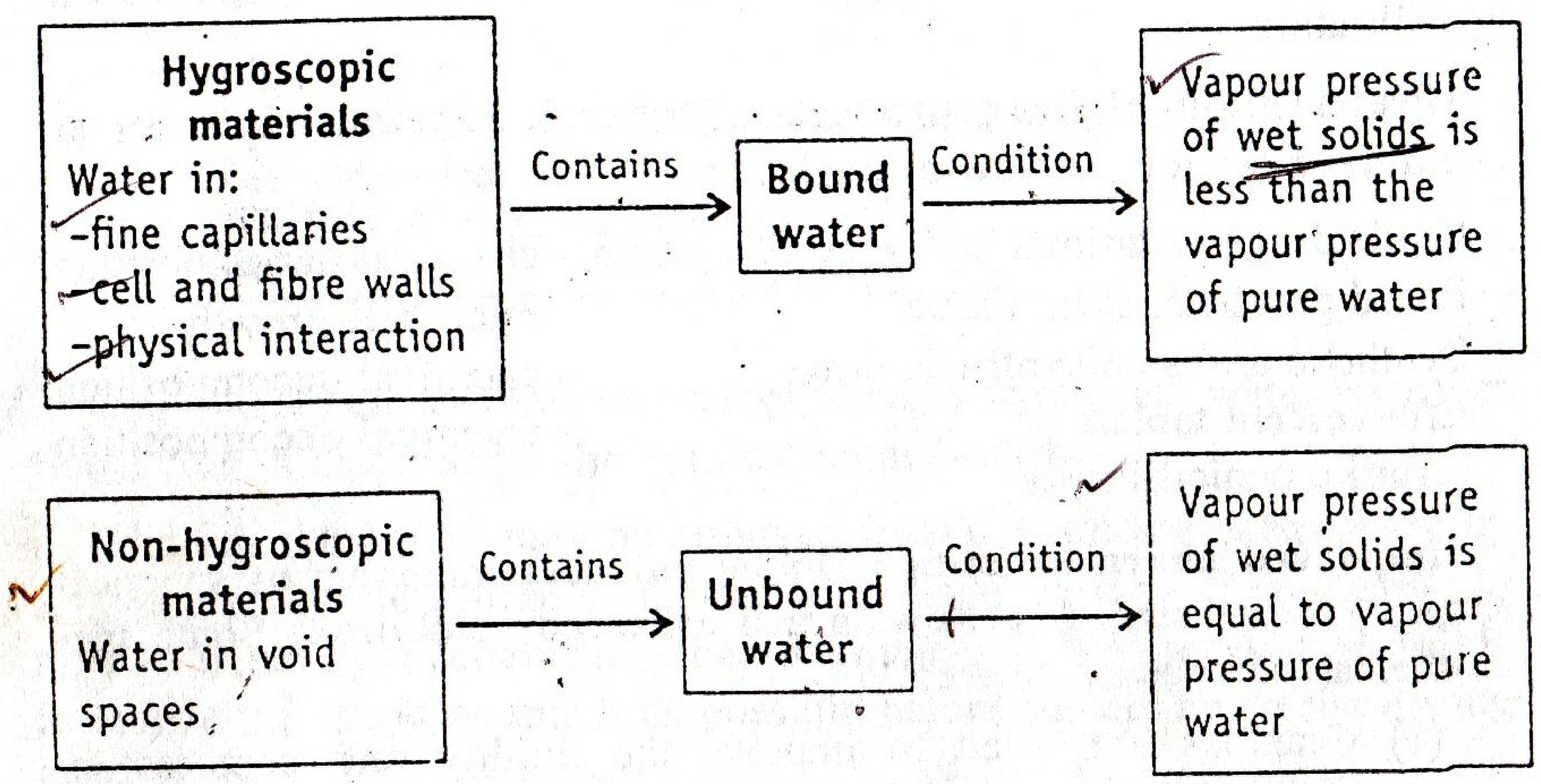
THEORY OF DRYING

In a wet solid mass, water may be present as bound water and unbound water.

Bound water (moisture) is the minimum water (moisture) held by the material that exerts an equilibrium vapour pressure less than the pure water at the same temperature.

Unbound water (moisture) is the amount of water (moisture) 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 the solid. Thus, in a non-hygroscopic material, all the liquid is unbound water. In a hygroscopic material, the unbound moisture is the liquid in excess of the equilibrium moisture content, corresponding to saturation humidity. Substances containing bound water are often called *hygroscopic substances*. The distinction between bound and unbound water depends on the material itself. These are described below.



Heat must be transferred to the material to be dried in order to supply the latent heat required for vaporisation of the moisture. Water diffuses through the material to the surface and subsequently evaporates into the air stream. Thus drying involves both heat transfer and mass transfer operations simultaneously.

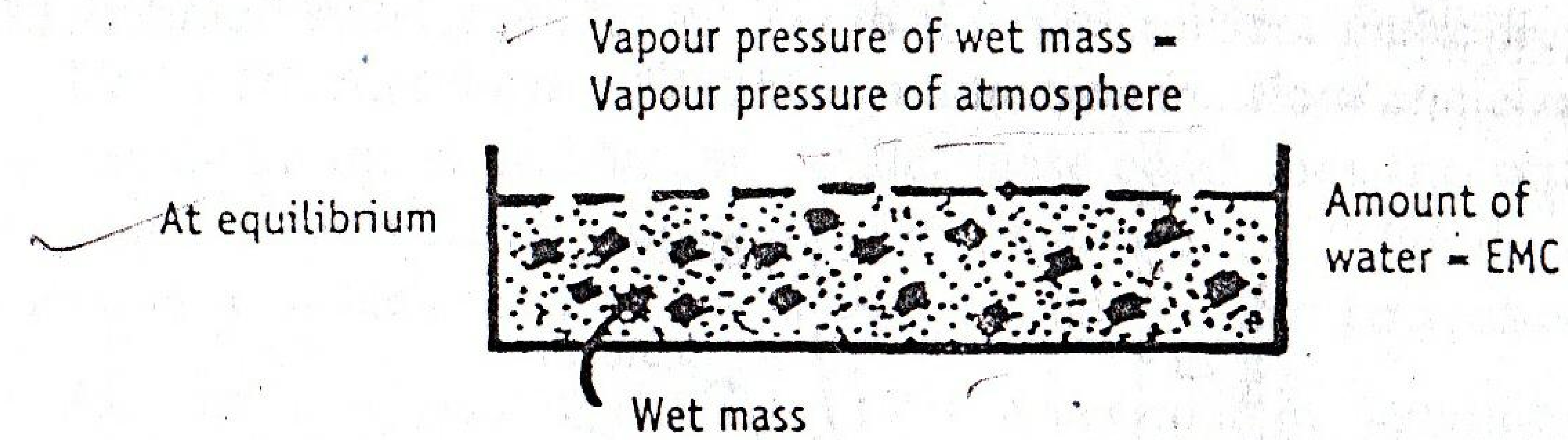
Theory can be discussed in two heads namely equilibrium relationships and rate relationships.

Equilibrium Relationships

Air of constant temperature and humidity is passed over the wet solid. After a long exposure, equilibrium is reached. On attaining equilibrium, further exposure will not alter moisture content in the solid. At this stage, vapour pressure of the wet solids is equal to that of the surrounding atmosphere. Hence there is no driving force for mass transfer.

Equilibrium moisture content (EMC) : It is the amount of water present in the solid which exerts a vapour pressure equal to the vapour pressure of the atmosphere surrounding it.

Equilibrium moisture content in a wet mass is shown below.

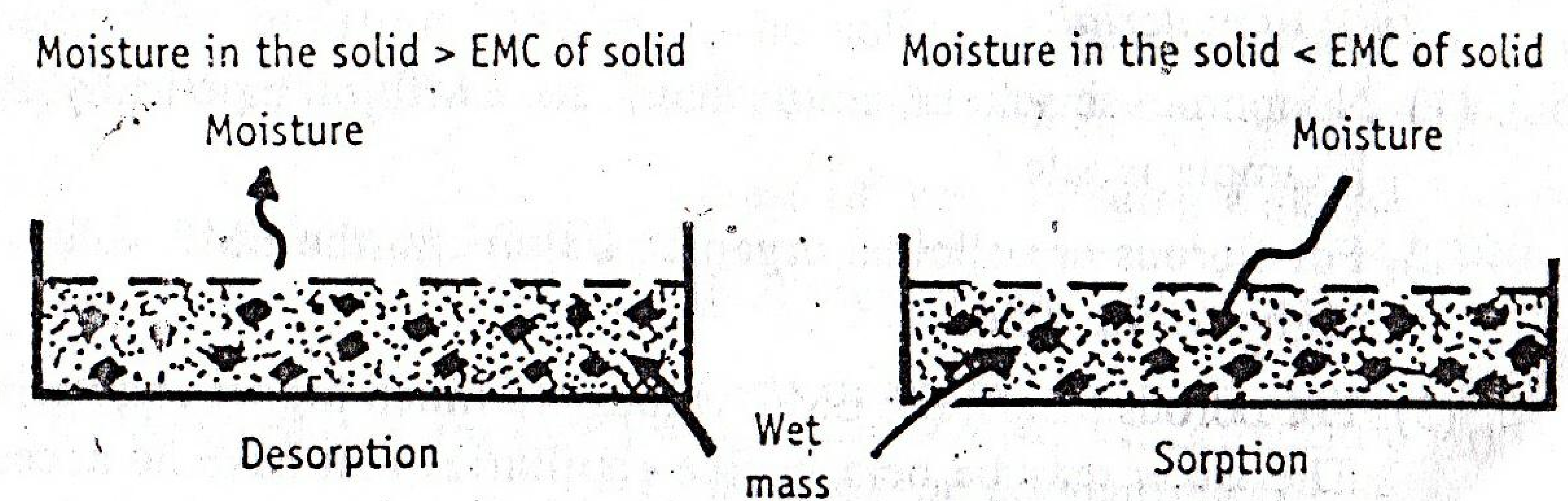


The characteristics of air such as temperature and humidity are maintained constant. Depending on these conditions, the solids may absorb or lose moisture.

(a) When air (of constant temperature and humidity) is continuously passed over the solid containing moisture more than EMC, then solid loses water continuously till EMC is reached. This phenomenon is known as *desorption*.

(b) When air (of constant temperature and humidity) is continuously passed over the solid containing moisture less than EMC, then solid adsorbs water continuously till EMC is reached. This phenomenon is known as *sorption*.

The behaviour of desorption and sorption is shown below. From the above observations it is clear that, material can be dried up to EMC but not below it.



At a given temperature and humidity of air

Measurement of EMC : The EMC of a material can be determined as follows. The solid samples are placed in a series of closed chambers such as desiccators. Each chamber consists of solution (desiccant), which maintains a fixed relative humidity in the enclosed air spaces. In other words, the solid samples are exposed to several humidity conditions. The exposure is continued until the material attains a constant weight (equilibrium conditions). The difference in the final and initial weights gives the moisture content.

Equilibrium moisture curve is drawn by taking relative humidity (%) on x-axis and moisture content on y-axis (Figure 14-1).

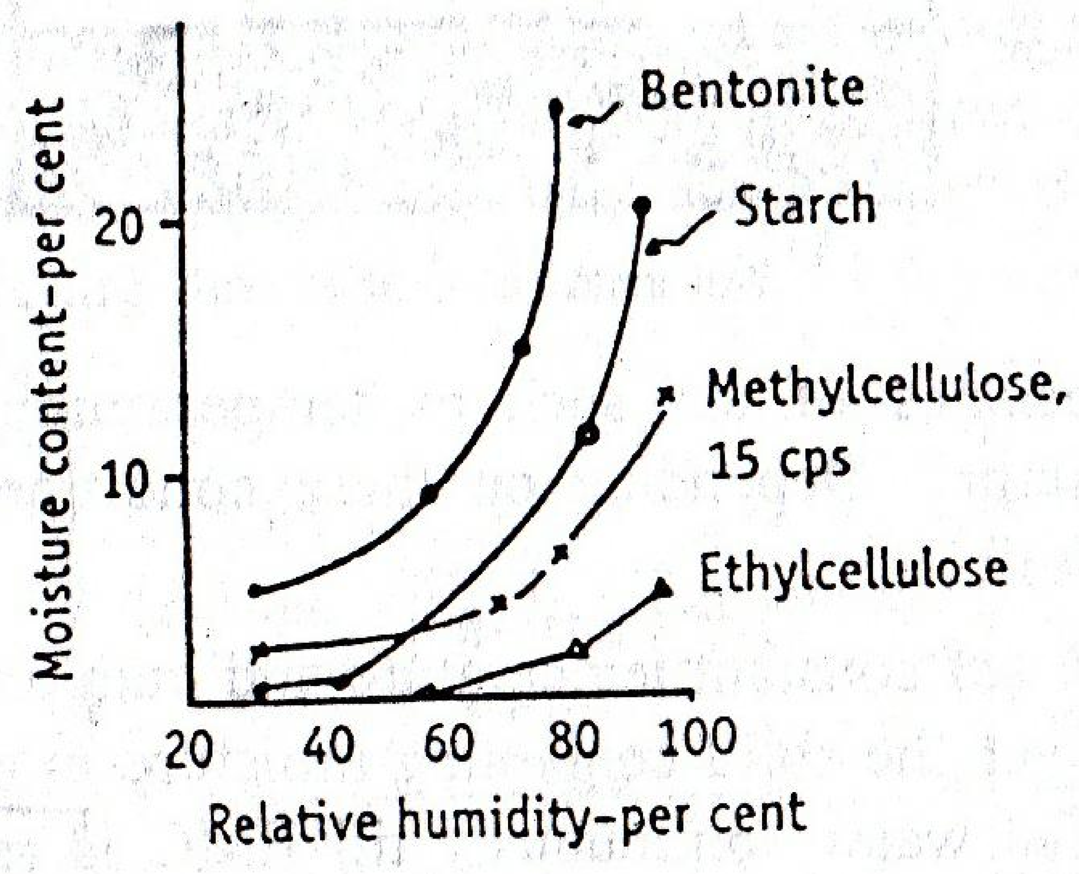


Figure 14-1. EMC curves for tabletting material.

Application of EMC : The EMC curve permits the selection of the experimental conditions to be used for drying of the product. Drying should be stopped when the moisture content reaches the level of the EMC under the exposed conditions. Over drying can be avoided.

Factors affecting EMC : EMC values vary depending on a number of factors.

Nature of material:

(1) Nonporous insoluble solids have an EMC of practically zero. Example is talc.

(2) For fibrous or colloidal organic substances, the EMC values are high and variable.

(3) For porous solids, the EMC values are much higher and variable. The water may be held in fine capillaries that have no access to the surface. The dissolved solid may reduce the vapour pressure and water may be molecularly bound.

Nature of air:

(1) For air of zero humidity, EMC of all materials is zero.

(2) As the temperature of air increases, the EMC of solid decreases.

EMC of solids is a constant for a given temperature and humidity of air. If the equilibrium curves (Figure 14-1) are continued up to 100% RH, the moisture content so obtained (EMC with saturated air) is the least moisture at which the material can exert a vapour pressure as high as that exerted by liquid water at the same temperature.

Free moisture content (FMC) : *Free moisture content* (FMC) is the amount of water that is free (easy) to evaporate from the solid surface.

Under the conditions of saturation humidity (100% RH), the EMC is the minimum moisture content. Under these conditions the water must be bound water which is minimum at that temperature. The remaining is unbound for which the FMC for a given condition can be written as:

Free moisture content (FMC) = total water content − equilibrium moisture content (EMC)

The distinction between free and equilibrium moisture depends on the drying conditions.

Rate Relationships

Rate relationships can be studied by considering a simple model, which mimic the conditions of a dryer. In this model, wet slab of material of sufficiently high moisture content to be dried is placed in a tray whose bottom and sides are insulated. The air is blown over the solid under constant drying conditions (air velocity, temperature, humidity and pressure are maintained constant). The superficial water diffuses through the surrounding stationary air film and is carried away rapidly by the moving air stream.

Periodically the slab is weighed. The difference in the weights of two successive periods gives the loss of moisture content, i.e., amount dried. The moisture present in the solid can be expressed on a wet weight or dry weight basis. Then the following calculations are made:

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

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

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

Drying rate is plotted against the midpoints of the time period. Similarly midpoints of the moisture content values can be plotted.

From the data obtained by the above experiment, a graph is plotted by taking FMC on x-axis and drying rate on y-axis. The curve so obtained (Figure 14-2) is called *drying rate curve*. It represents different changes during drying as explained below.

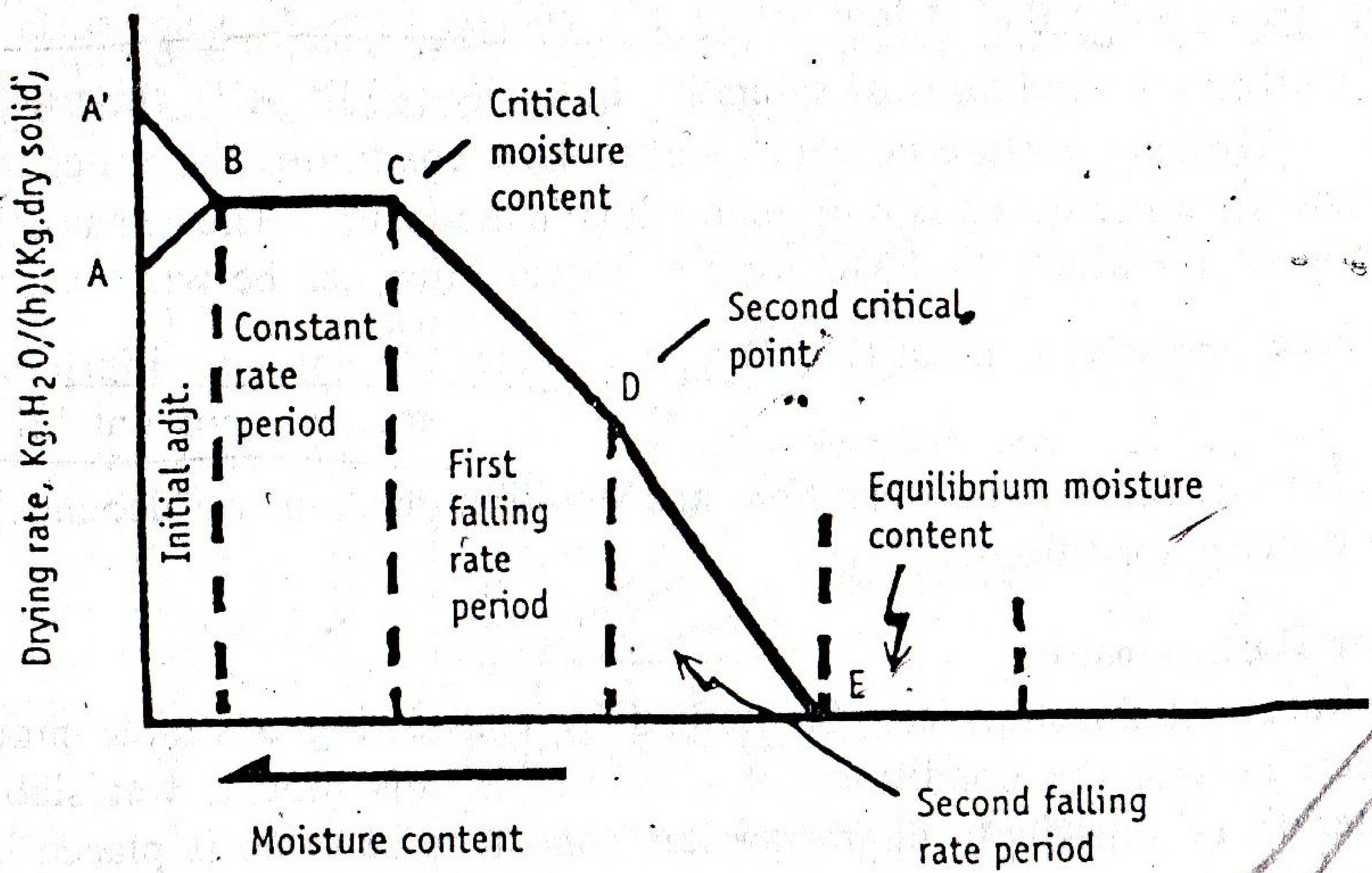
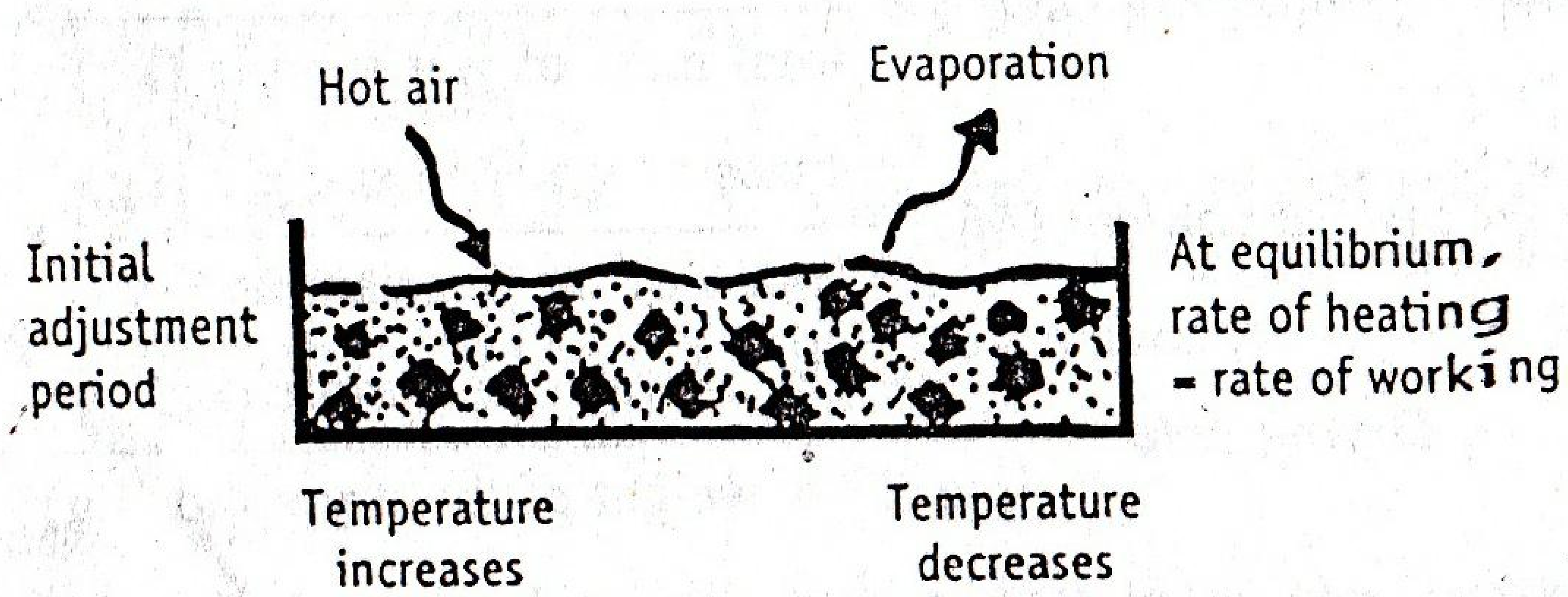
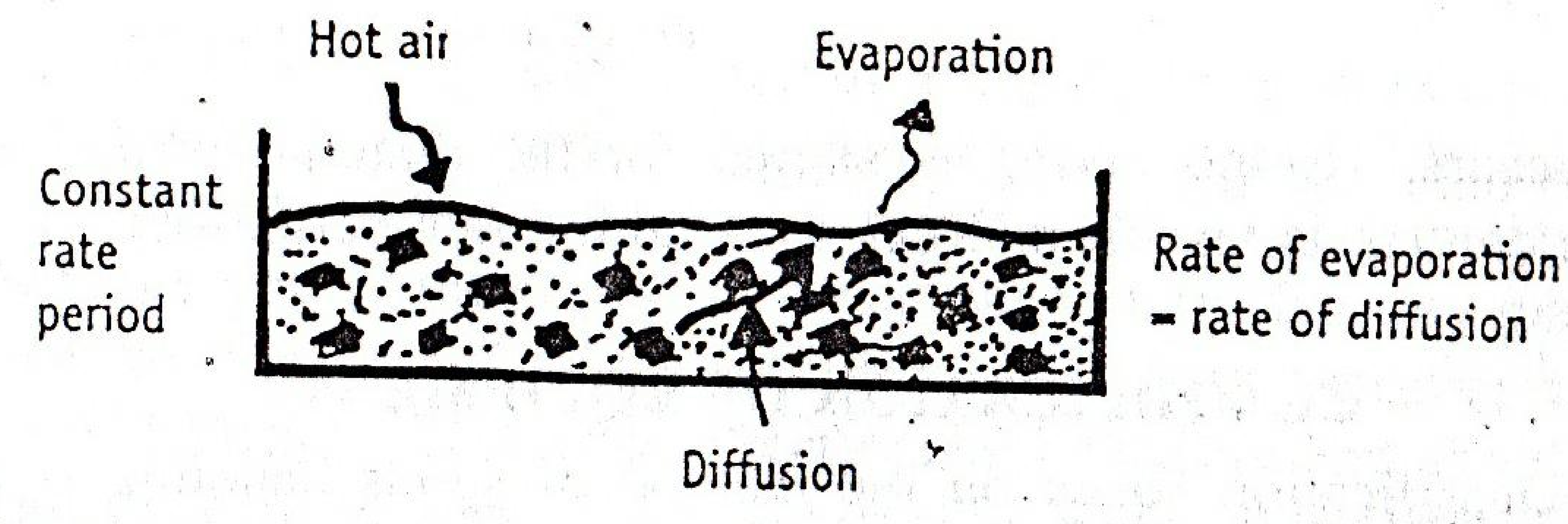


Figure 14-2. FMC curve.

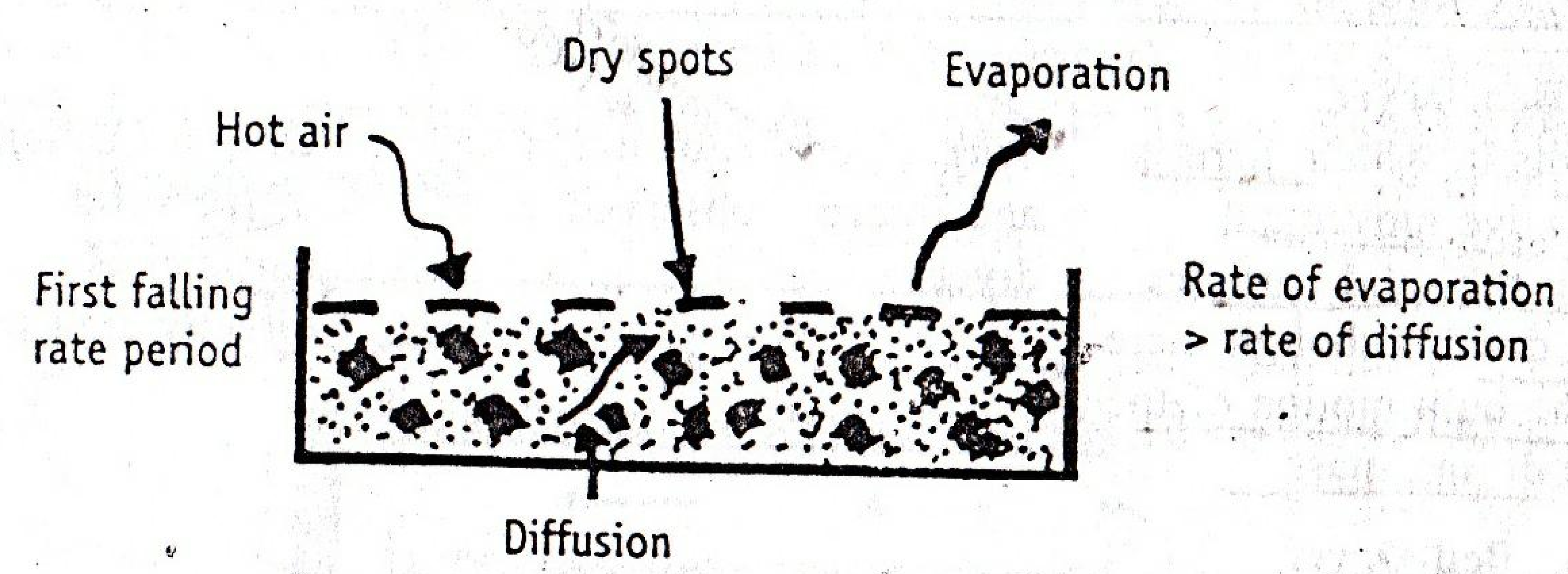
(1) The time corresponding to AB represents the *initial adjustment period.*. During this period, the solids absorb heat and the temperature increases. At the same time, the moisture begins to evaporate and thus tends to cool the drying solid. After some time, the temperature stabilises (heating and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



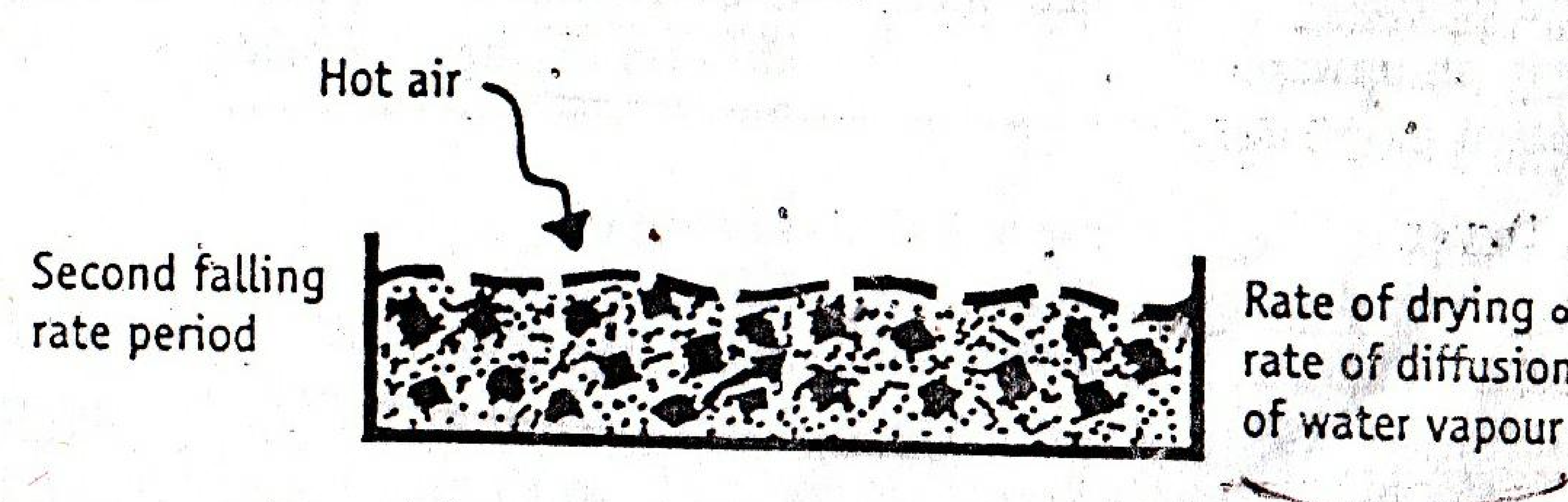
(2) The time corresponding to BC represents the *constant rate period.* The temperature remains constant and rate of drying is constant. The moisture evaporating from the surface is replaced by the water diffusing from the interior of the solid. The rate of diffusion is equal to the rate of evaporation.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



(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.



(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.*



(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. If the drying is started for the material whose initial moisture content is less than CMC, then falling rate period occurs.

CLASSIFICATION OF EQUIPMENT

The classification based on the method of solids handling is more suitable when special attention is given to the nature of material to be dried. Dryers are classified as given in Table 14-1.

TABLE 14-1
Classification of Dryers—Mechanism, Examples, Advantages and Disadvantages

Type of dryer and mechanism	*Examples*	*Advantages*	*Disadvantages*
Static Bed Dryer Systems in which there is no relative movement among the solid particles being dried, although there may be bulk motion of the entire drying mass.	Tray dryer and freeze dryer	Attrition is not observed	Only a fraction of the solid particles is directly exposed
Moving Bed Dryer Systems in which the drying particles are partially separated so that they flow over each other	Drum dryer	The entire material is continuously exposed to heat source	Attrition is possible
Fluidised Bed Dryer Systems in which the solid particles are partially suspended in an upward moving heated gas system	Fluidised bed dryer	Solid-gas contact is excellent, uniform drying	Attrition of particles takes place
Pneumatic Dryer Systems in which drying particles are entrained and conveyed at a high velocity gas stream	Spray dryer	Efficient and rapid drying	

EQUIPMENT

TRAY DRYER

Principle : In tray dryer, hot air is continuously circulated. Forced convection heating takes place to remove moisture from the solids placed in trays. Simultaneously, the moist air is removed partially.

Construction : The construction of a tray dryer is shown in Figure 14-3. It consists of a rectangular chamber whose walls are insulated. Trays are placed inside the heating chamber. The number of trays may vary with the size of the dryer. Dryers of laboratory size may contain a minimum of three trays, where as dryers of industry size may contain more than 20 trays. Each tray is rectangular or square and about 1.2 to 2.4 metres square in area. Trays are usually loaded from 10.0 to 100.0 millimetres deep. The distance between the bottom of upper tray and (upper) surface of the substance loaded in the subsequent tray must be 40.0 millimetres.

Alternately the trays can be placed in trucks on wheels, which can be rolled into and out of chamber. Two such trucks can be arranged inside dryer. Dryer is fitted with a fan for circulating air over the trays. Electrically heated elements are provided inside (rather than outside) to heat the air (steam also can be used as it is cheaper). In the corner of the chamber, direction vanes (not shown in Figure 14-3) are placed to direct air in the expected path.

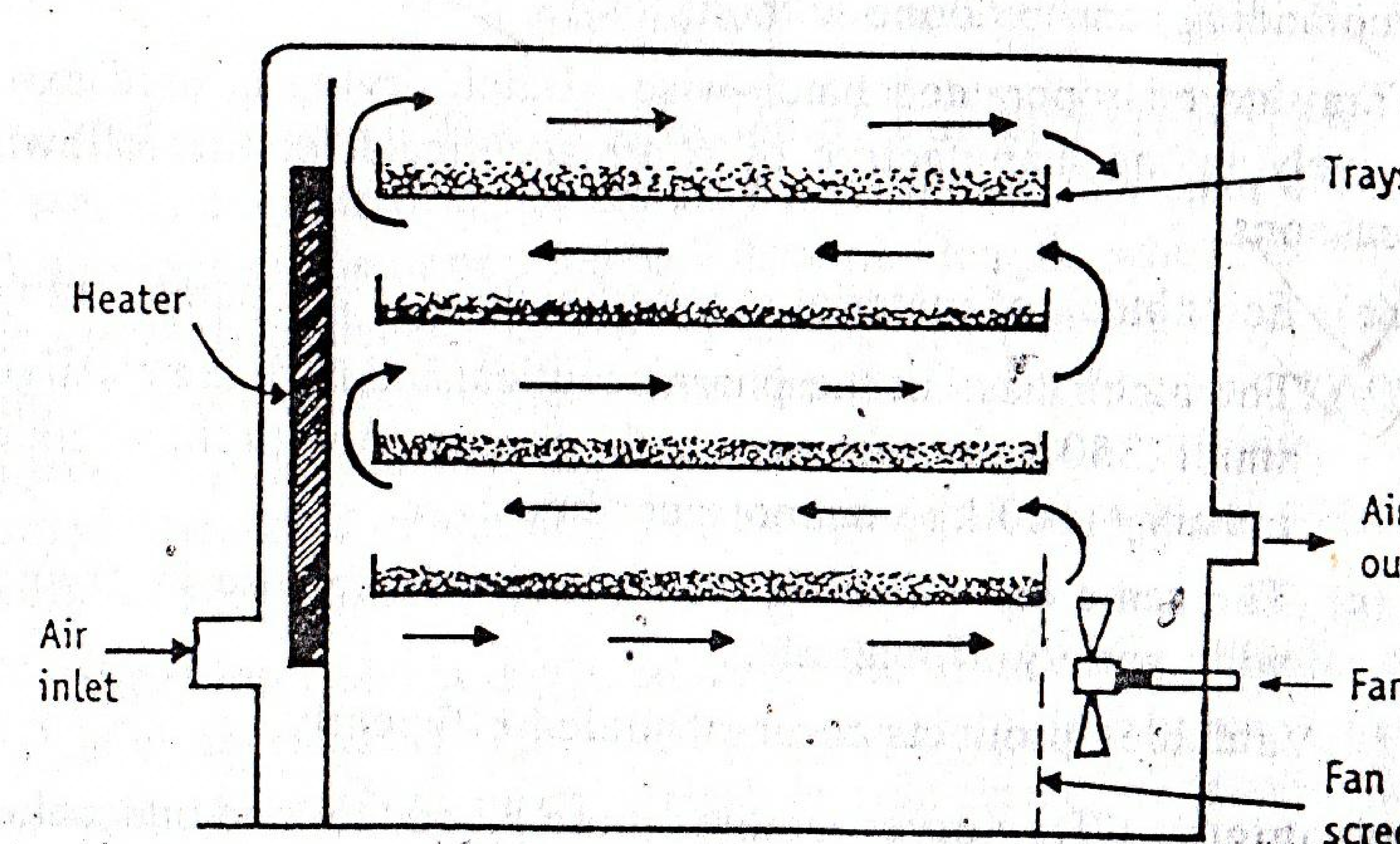


Figure 14-3. Tray dryer.

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 gets heated up. The hot air is circulated by means of fans at 2 to 5 metre per second. Turbulent flow lowers the partial vapour pressure in the atmosphere and also reduces the thickness of the air boundary layer. The water is picked up by air. As water evaporates from the surface, the water diffuses from the interior of the solid by capillary action. These events occur in a single pass of air. The time of contact is short and the amount of water picked up in a single pass is small. Therefore the discharged air to the tune of 80 to 90% is circulated back through fans. Only 10 to 20% of fresh air is introduced. Moist air is discharged through outlet. Thus constant temperature and uniform airflow over the material can be maintained for achieving uniform drying.

In case of wet granules (as in tablets and capsules) drying is continued until the desired moisture content is obtained. At the end of drying, trays (trucks) are pulled out of the chamber and taken to a tray dumping station.

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.

Variants : Tray dryer may be operated under vacuum, often with indirect heating. This is done in special vacuum tray dryers for drying vitamins and other heat-sensitive products.

Tunnel dryer : In this type, trucks are loaded with wet material at one end of the tunnel. The tunnel comprised of a number of units, each of which is electro-statically controlled. The solids get dried and the product is discharged at the other end of the tunnel.

DRUM DRYER OR ROLLER DRYER
(or Film Drum Dryer)

Principle : In drum dryer, a heated hollow metal drum rotates on its longitudinal axis, which is partially dipped in the solution to be dried. The solution is carried as a film on the surface of the dryer and dried to form a layer. A suitable knife scraps the dried material, while the drum is rotating.

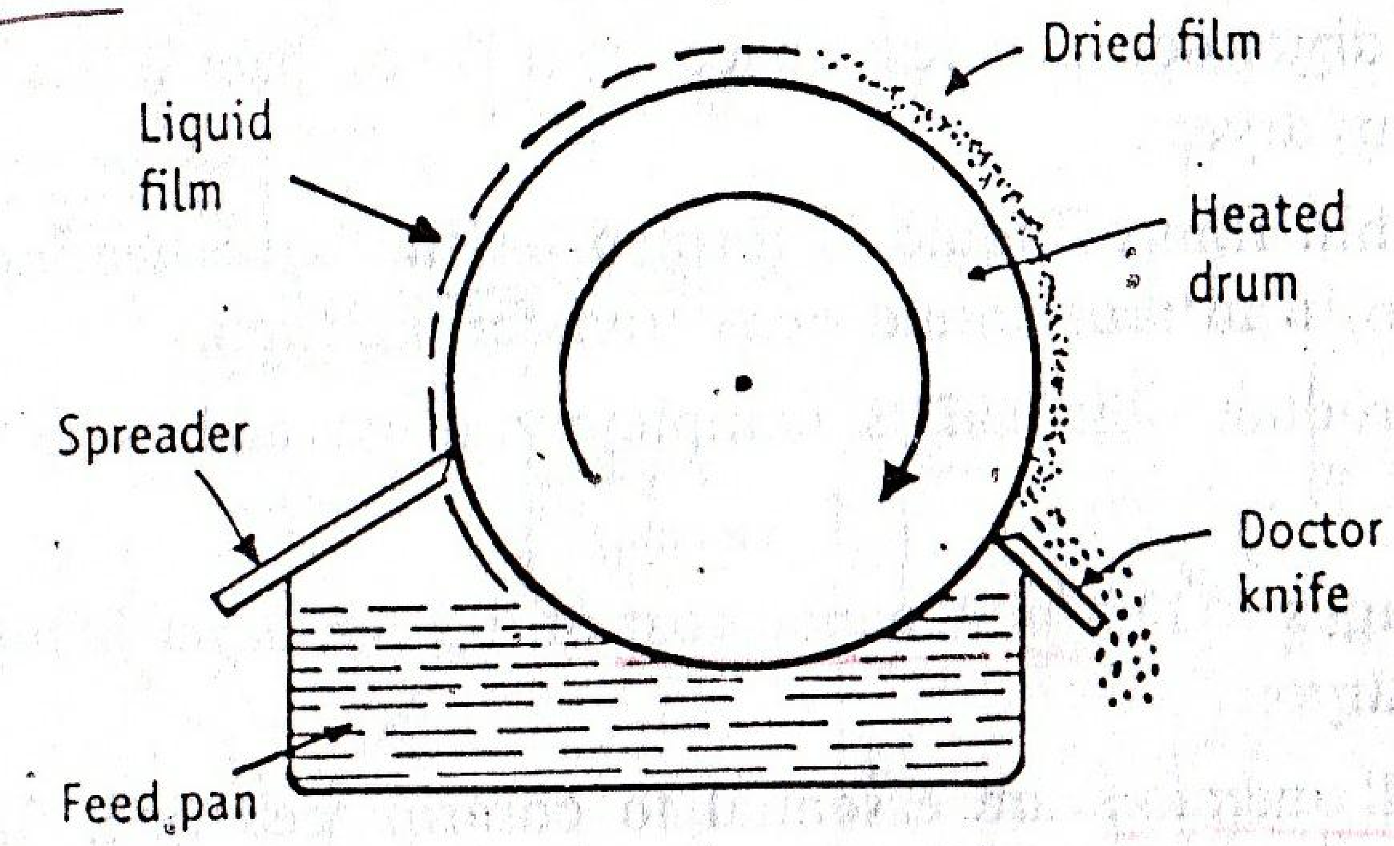


Figure 14-4. Drum dryer.

Construction : The construction of a drum dryer is shown in Figure 14-4. It consists of a horizontally mounted hollow steel drum of 0.6 to 3.0 metres diameter and 0.6 to 4.0 metres length, whose external surface is smoothly polished. Below the drum, feed pan is placed in such a way that the drum dips partially into the feed. On one side of the drum a spreader is placed and on the other side a doctor's knife is placed to scrap the dried material. A storage bin (or a conveyor) is placed connecting the knife to collect the material.

Working : Steam is passed inside the drum. Heat transfer coefficient of the drum metal is high. Drying capacity is directly proportional to the surface area of the drum. Heat is transferred by conduction to the material. Simultaneously drum is rotated at a rate of 1-10 revolutions per minute. The liquid material present in the feed pan adheres as a thin layer to the external surface of the drum during its rotation. The material is completely dried during its journey in slightly less than one rotation (i.e., from one side to another side of the drum). The dried material is scrapped by the doctor's knife, which then falls into a storage bin. The time of contact of the material with hot metal is 6 to 15 seconds only. Therefore, processing conditions such as film thickness, steam temperature are closely controlled.

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.

Variants : A vacuum drum dryer encloses both drum and feed line in a vacuum chamber to facilitate drying of heat sensitive materials. It is suitable for drying of drugs susceptible to oxidation and to recover solvents.

In a large scale, instead of one drum, two drums are set in parallel, rotating in opposite directions with a common feed inlet. The feed can be introduced on to the drum either by spraying it on the top or by introducing from the bottom.

SPRAY DRYER

Principle : In spray dryer, the fluid to be dried is atomized into fine droplets, which are thrown radially into a moving stream of hot gas. The temperature of the droplets is immediately increased and fine droplets get dried instantaneously in the form of spherical particles. This process completes in a few seconds before the droplets reach the wall of the dryer.

Construction : The construction of a spray dryer is shown in Figure 14-5. It consists of a large cylindrical drying chamber with a short conical bottom, made up of stainless steel (diameter of the drying chamber ranges between 2.5 to 9.0 metres and height is 25.0 metres or

more). An inlet for hot air is placed in the roof of the chamber. Another inlet carrying spray-disk atomizer is set in the roof (shown in Figure 14-5). The spray disk atomizer is about 300 millimetres in diameter and rotates at a speed of 3,000 to 50,000 revolutions per minute. Bottom of the dryer is connected to a cyclone separator.

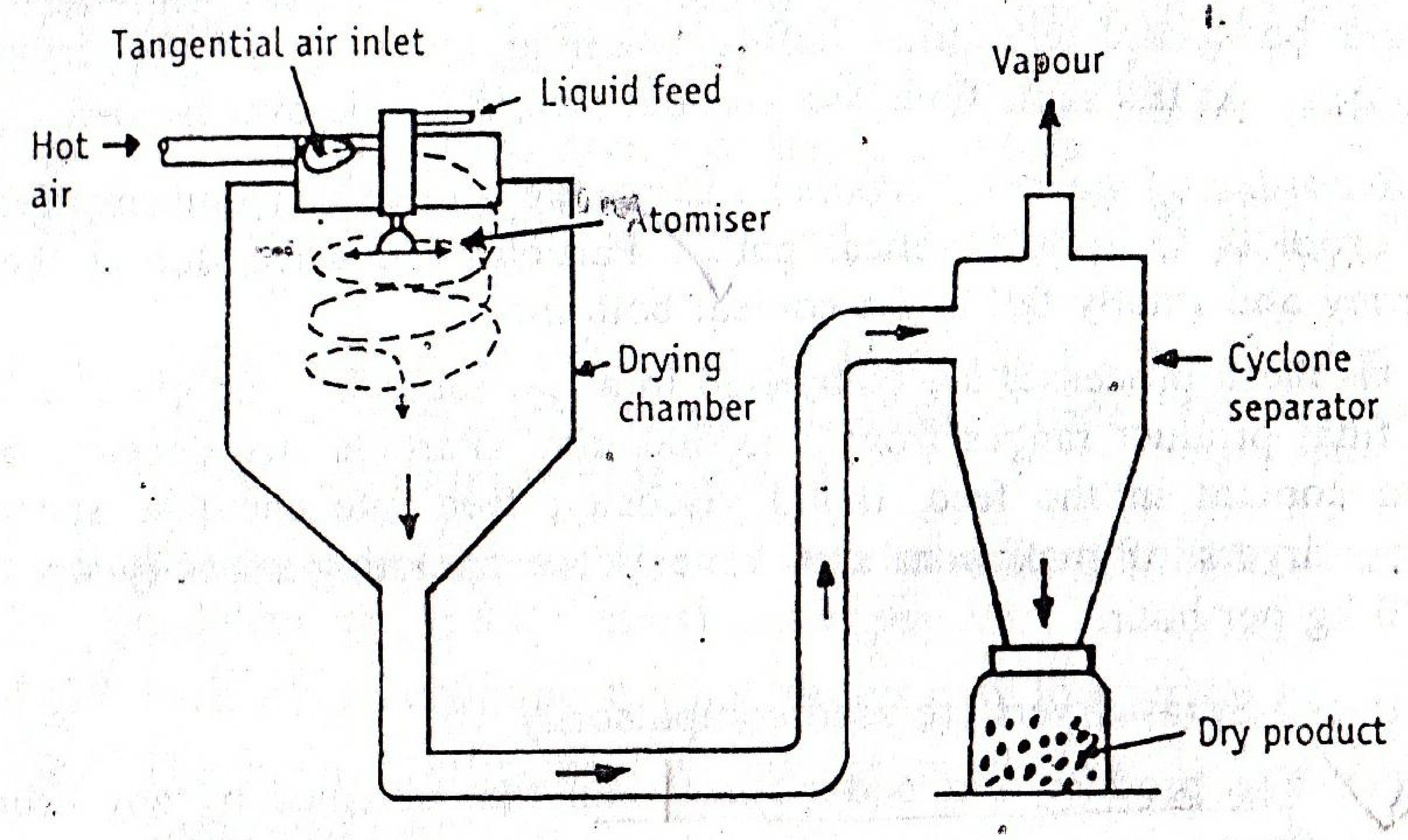


Figure 14-5. Spray dryer.

Working : Drying of the material in spray dryer involves 3 stages.

(1) Atomization of the liquid.

(2) Drying of the liquid droplets.

(3) Recovery of the dried product.

Atomization of the liquid to form liquid droplets : The feed is introduced through the atomizer either by gravity or by using suitable pump to form fine droplets. The properties of the final product depend on the droplet form, hence, the selection of the type of atomizer is important. Atomizer of any type: pneumatic atomizer, pressure nozzle and spinning disc atomizer may be used.

The rate of feed is adjusted in such a way that the droplets should be completely dried before reaching the walls of the drying chamber. At the same time, the product should not be over heated.

Drying of the liquid droplets : Fine droplets are dried in the drying chamber by supplying hot air through the inlet.

The surface of the liquid drop is dried immediately to form a tough shell. Further, the liquid inside must escape by diffusing through the shell at a particular rate. At the same time, heat transfer from outside to inside takes place at a rate greater than liquid diffusion rate. As a result, heat inside mounts up which allows the liquid to evaporate at a faster rate. This tendency of a liquid leads to rise in the internal pressure, which causes the droplets to swell. The shell's thickness decreases where as permeability for vapour increases. If the shell is neither elastic nor permeable, it ruptures and the internal pressure escapes.

The temperature of air is adjusted in such a way that the droplets should be completely dried before reaching the walls of the drying chamber. At the same time, the product should not be over heated.

Recovery of the dried product : Centrifugal force of atomizer drives the droplets to follow helical path. Particles are dried during their journey and finally fall at the conical bottom.

All these processes are completed in a few seconds. Particle size of the final product ranges from 2 to 500 mm. Particle size depends on solid content in the feed, liquid viscosity, feed rate and disc speed. Spray dryers of maximum size have got evaporating capacity up to 2000 kg per hour.

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.

A few products that are dried using spray dryer are:

acacia	chloramphenicol succinate	gelatin	plasma
adrenaline	citric acid	hexamine	serum
bacitracin	coffee extract	hormones	soaps
barium sulphate	detergents	lactose	sodium phosphate
blood	dextran	methyl cellulose	starch
borax	extracts	milk	sulphur
boric acid	ferrous sulphate	pancreatin	vaccines
calcium sulphate	fruit juices	penicillin	vitamins
		pepsin	yeast

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.

(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.

Variants : (1) Spray dryer can be constructed in such a way as to suit sterile products.

(2) It can be operated under closed conditions to recover solvents.

(3) It can be operated under oxygen free environment.

(4) It can be constructed in such a way that the flow of liquid and gas may be co-current, counter current or a combination of both in the same unit.

(5) The same equipment can be used for spray congealing.

(6) It is useful for encapsulation (coating) of solid and liquid particles.

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 granules 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 14-6. 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 part 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.

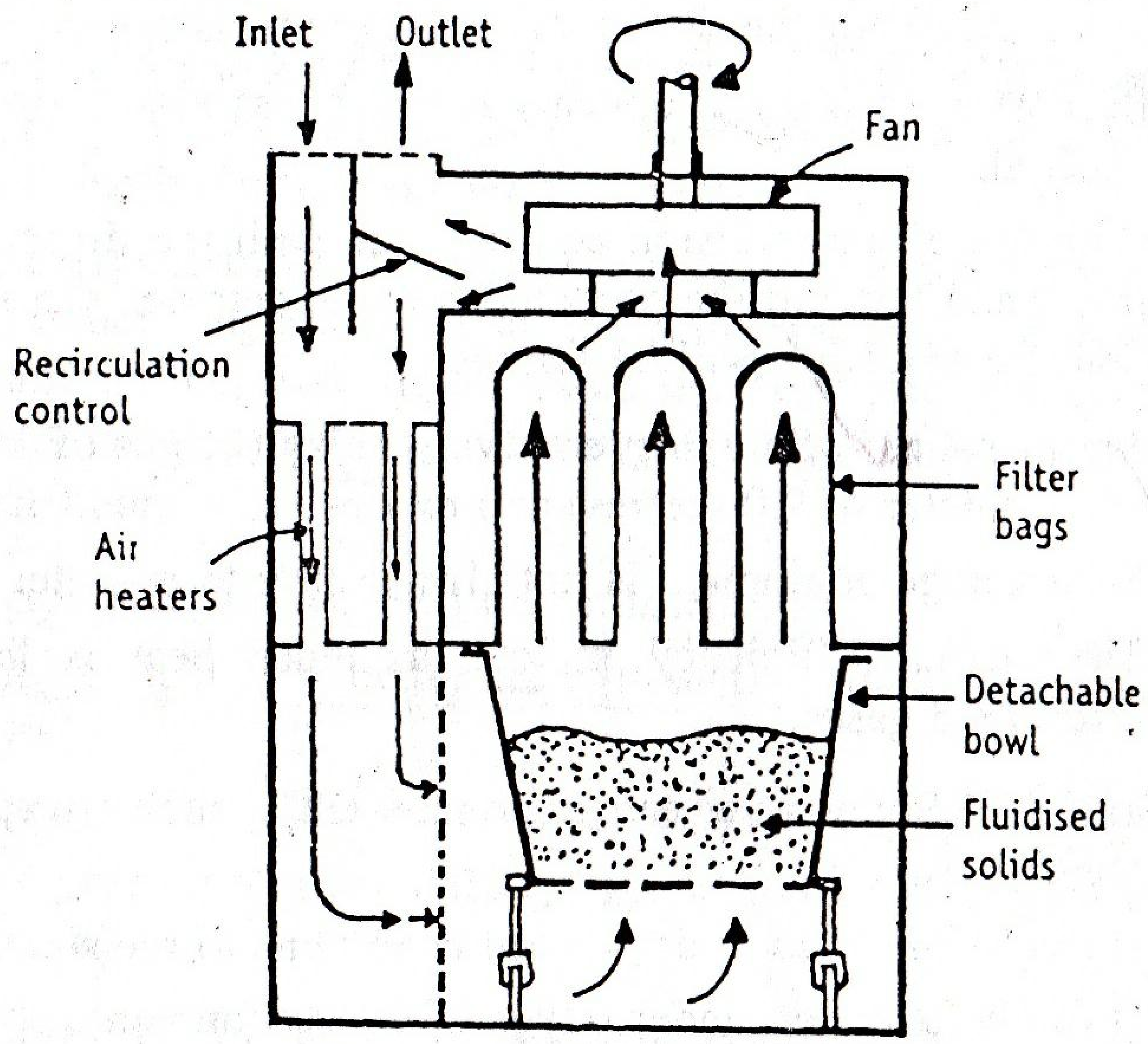


Figure 14-6. Fluidised bed dryer.

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.

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 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.

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 discharge.

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.

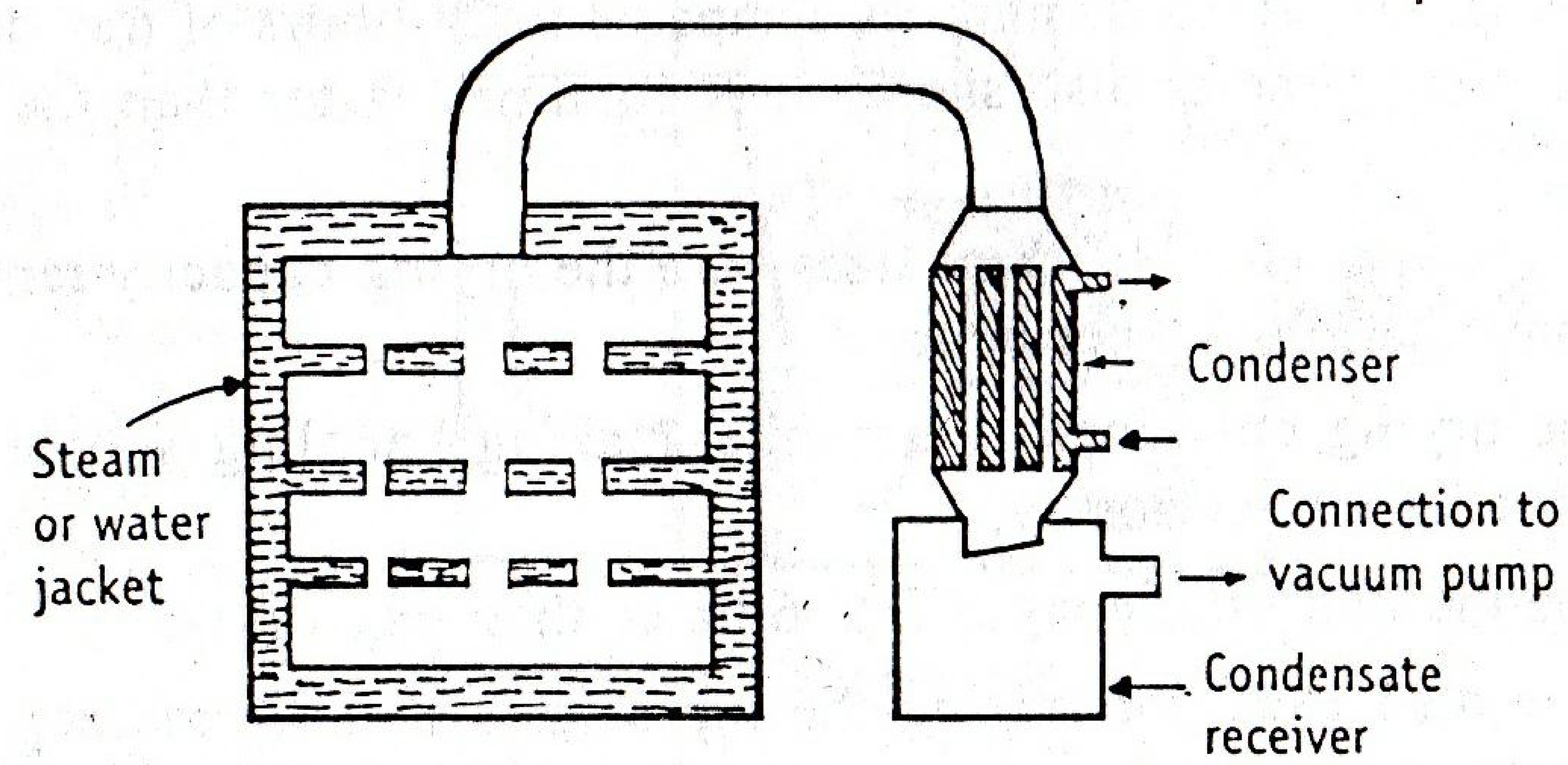


Figure 14-7. Vacuum dryer.

Construction : The construction of a vacuum dryer is shown in Figure 14-7. 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. 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.

(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

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 by 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 14-8. 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.

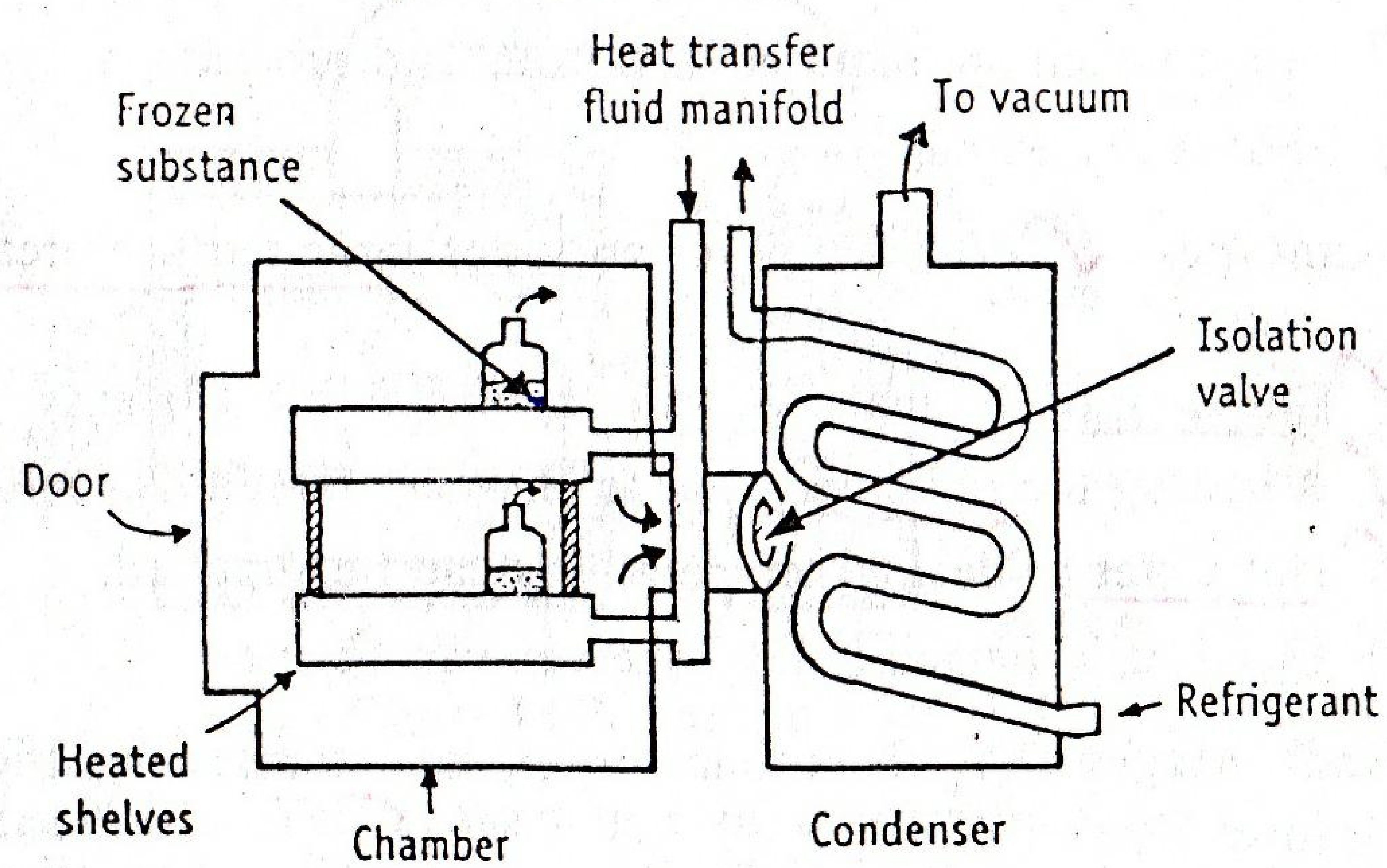


Figure 14-8. Schematic diagram of freeze dryer.

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 evaporated 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 pretreatment

(2) Prefreezing for solidifying water

(3) Primary drying (sublimation of ice under vacuum)

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

(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.

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 K below eutectic point is sufficient.

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

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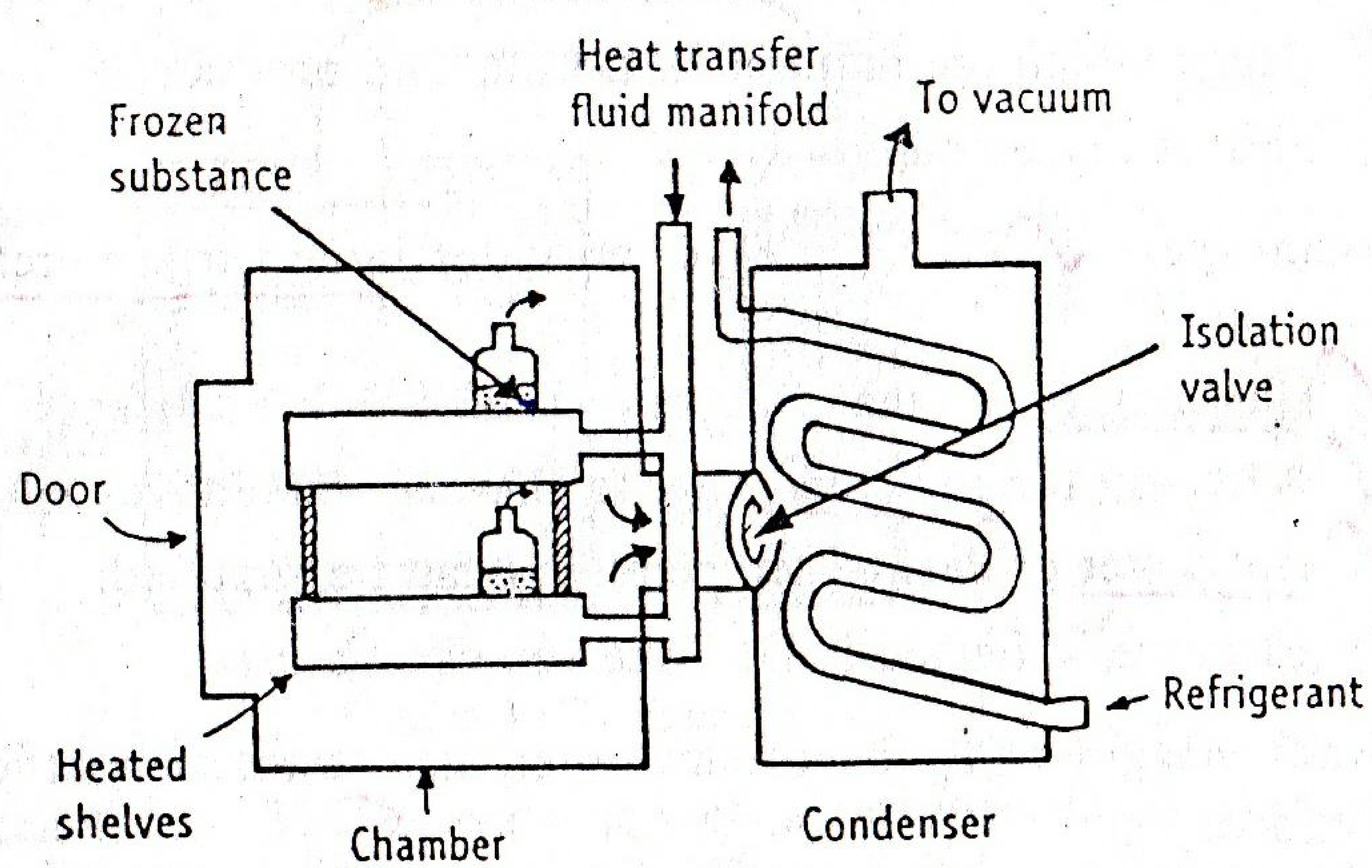

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Heat (about 2900 kilojoules per kg) is supplied which transfers as latent heat and ice sublimes directly into vapour state. The heat controls the movements of ice layer inwards. It has to be controlled in such a manner so as to get highest possible 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 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.

QUESTION BANK

Each question carries 2 marks

1. Distinguish between drying and evaporation.
2. Give suitable dryers.
   (i) Granular free flowing solids
   (ii) Wet bricks before sending to kilns
   (iii) Sticky pastes
   (iv) Food products like Horlicks.
3. Define critical moisture content and equilibrium moisture content.
4. Define bound moisture and free moisture content.
5. Mention the factors affecting constant drying rate.
6. Classify dryers giving suitable examples.
7. List the critical conditions for drying of various substances.
8. Define drying. Give its importance in the formulation of dosage forms.
9. Explain how agitator dryers are useful to dry pasty and sludgy materials.
10. How do you obtain the rate of drying curve for a given drying operation? Give its applications.

Each question carries 5 marks

1. Recommend a suitable dryer for drying the following substances and substantiate your answer with at least two reasons.
   (A) Liver extracts
   (B) Granular solids
   (C) Pasty materials

(D) Granules of heat sensitive drugs
(E) Vitamin B complex granules
(F) Colloidal solution

2. Describe the drying rate curve for a nonporous granular solid.
3. Describe the principle with the help of a labeled diagram of fluidised bed dryer.
4. List the pharmaceutical applications of freeze drying process. Give salient features of the process.
5. Describe the construction and working of a fluidized bed dryer.
6. Explain the principle of spray drying with suitable labeled diagram.
7. Describe the drying rate curve. Explain its applications.
8. Explain the principle and working of drum dryer.
9. Compare spray drying with drying in a vacuum shell dryer.
10. Explain the factors to be considered in the selection of a suitable dryer.
11. Explain the operation and applications of fluidised bed dryer.
12. Describe the function of drum dryer and its uses.
13. Describe the rate of drying curve for a crude fibrous drug.
14. Explain the principle of freeze drying. What are its applications in pharmacy?
15. Compare the operations of spray dryer and tray dryer.
16. Describe the concept of spray dryer. What are its advantages? Compare the spray drying with other methods of drying.

Each question carries 10 marks

1. Explain the construction, operational details of freeze dryer. Describe its applications in pharmacy.
2. Describe the concept of spray drying. Describe the specific advantages of spray dried product over drum dried material. Also list the pharmaceutical applications.
3. How do you classify dryers? Describe in detail the constant rate and falling rate periods. Add a note on critical moisture content.
4. For drying of milk, we can use either drum dryers or spray dryers. Which dryer will you prefer and why? Discuss drum dryers in detail.
5. Discuss the construction, working, advantages and disadvantages of spray dryer.
6. Explain the theory behind drying and rate of drying with suitable graphs.