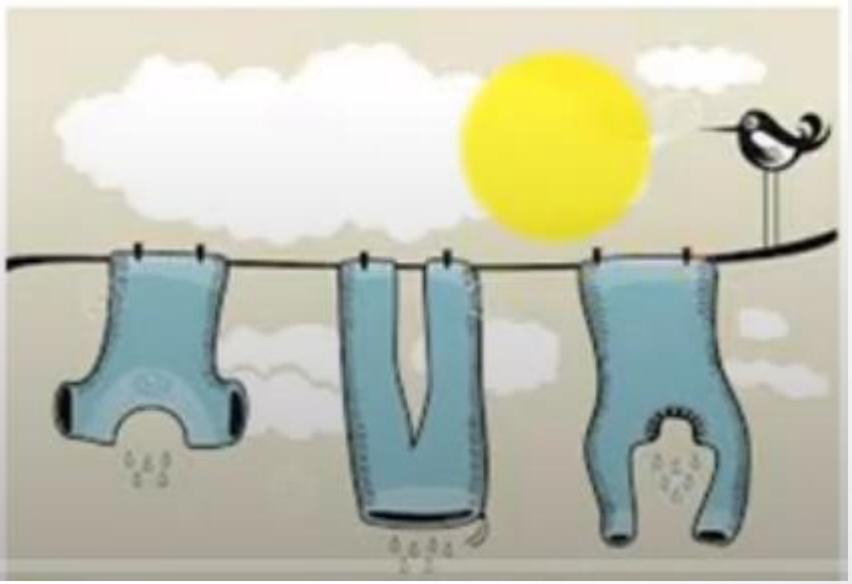
Drying



BY

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Content

- Definition of drying
- Purposes of drying
- Psychrometry (moisture measurement)
- Theory of drying

Introduction

Definitions

- ❑ Drying is the process of removal of small amount of a liquid (water/volatile liq./moisture) from a material by the application of heat to obtain dry solid

How it can be accomplished:

↓

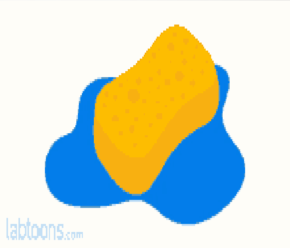
By transfer of a liquid from a surface into an unsaturated vapor phase.

- Drying and evaporation are relatively the same term but are distinguishable merely by the relative quantities of liquid removed from the solid.

- Drying is commonly the last stage in a manufacture process.

Non-thermal drying

Expression of a solid to remove liquid, as squeezing of a wetted sponge



The extraction of liquid from a solid by use of a solvent,

Adsorption: of water from a solvent using desiccants (such as anhydrous calcium chloride),

absorption of moisture from gases by passage through sulfuric acid column

Desiccation: the adsorption of moisture from a solid by placing it in a sealed container with a moisture removing agent (e.g., silica gel in a bottle),

Purpose of drying

1. Unit of process in pharmaceutical manufacturing (e.g., preparation of granules then dispensing in bulk or converted as capsules or tablets).

2. Reduces the bulk and the weight of drying materials → lowering the cost of transportation and storage.

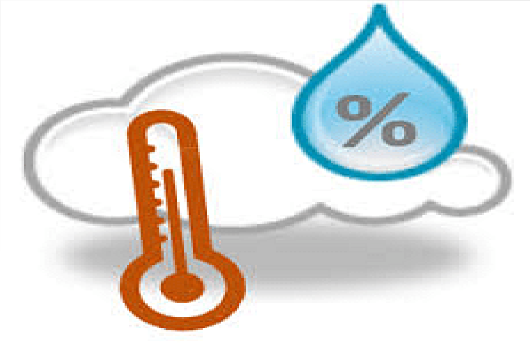
3. Aids in the preservation of animal and vegetable drugs by minimizing mold and bacterial growth.

4. Removing the moisture → facilitate comminution by increasing friability.

5. processing of materials as in Preparation of certain products such as spray dried lactose, dried AlOH and powdered extracts.

6. Drying enhances product stability by reducing chemical reactivity, making effervescent salts, aspirin, hygroscopic powders, ascorbic acid, and penicillin more stable.

Psychrometry



- A critical factor in drying operations is Vapor-carrying capacity of the air, nitrogen, or other gas stream passing over the drying material.
- The carrying capacity determines the rate and the extent of drying material (i.e., the lowest moisture content to which a given material can be dried).

Psychrometry → **is the** determination of the vapor concentration and carrying capacity of the gas.

- Humidity of the gas is **the concentration** of water vapor in a gas.

تسمى الحالة الغازية للماء الموجودة في جزيئات الهواء الرطوبة

Psychrometric or humidity chart

- Absolute humidity (g/m^2) is the total weight of water vapor per unit weight of dry air regardless temp. $AH = \frac{\text{weight of water vapor}}{\text{unit weight of dry air}}$
- Relative humidity (%) is a ratio of the actual amount of water vapor in the air compared to the total amount of vapor that can exist in the air at its current temperature.

- Dew point: is the temperature to which a given mixture of air and water vapor must be cooled (at constant pressure) in order to achieve a relative humidity (RH) of 100% (become saturated) (i.e., hold max. amount of moisture without condensation).

- Dry Bulb Temperature refers basically to the ambient air temperature.

- Wet Bulb temperature is the equilibrium temperature reached by an evaporating surface when the rate of heat transfer to the surface by convection = rate of heat lost by evaporation.

Psychrometric or humidity chart

- The curve represents the relationship between the temperature and humidity of the air-water vapor system at **constant pressure**.
- **horizontal axis** → temperature
- **vertical axis** → absolute humidity.

▪ Absolute humidity (g/m^2) is the total weight of water vapor per unit weight of dry air.

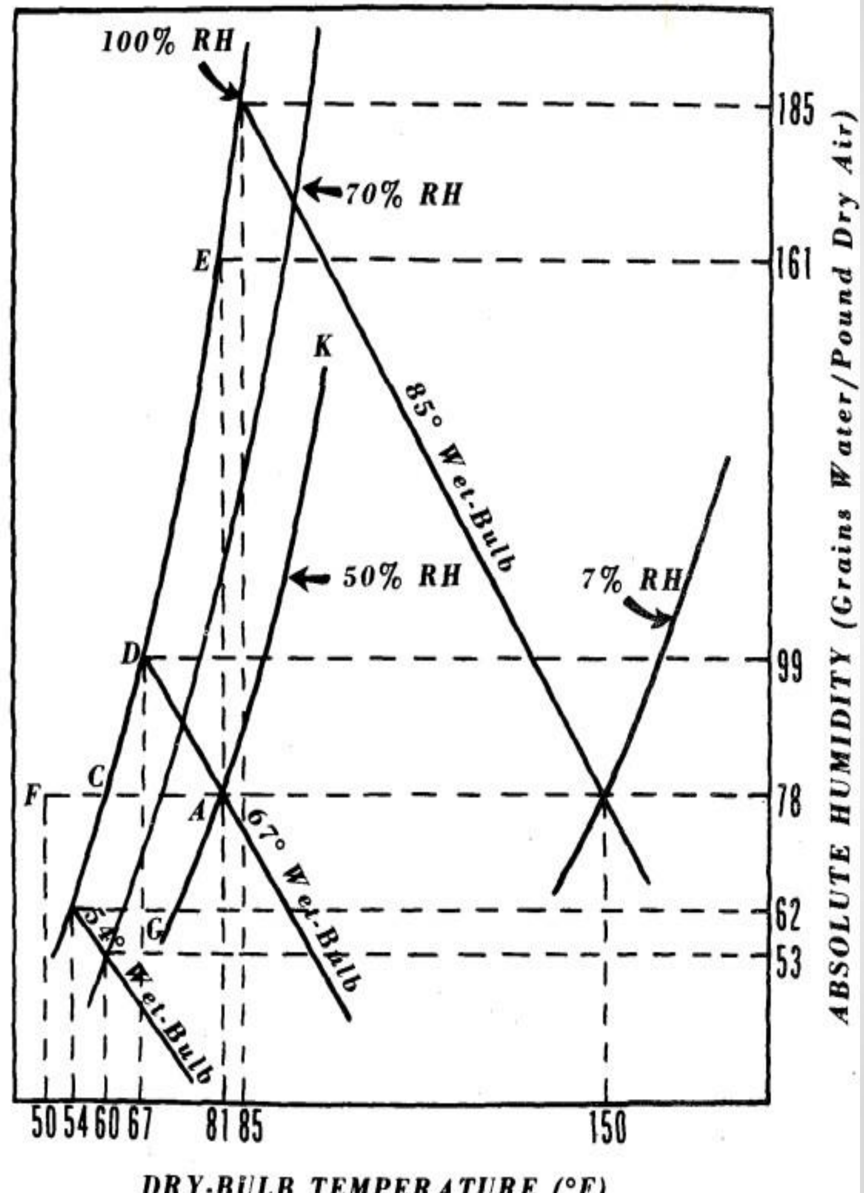


FIG. 3-1. Diagram of psychrometric chart showing the relationship of air temperature to humidity.

Psychrometric or humidity chart

- **CDE curve** → the saturation humidity curve; is the absolute humidity at which the partial pressure of water vapor **in the air** = the vapor pressure of free water at the same temperature.

- Under this condition → The air is completely saturated with moisture, and the humidity does not change when it is in contact with liquid water at the same temperature → that means 100% RH.

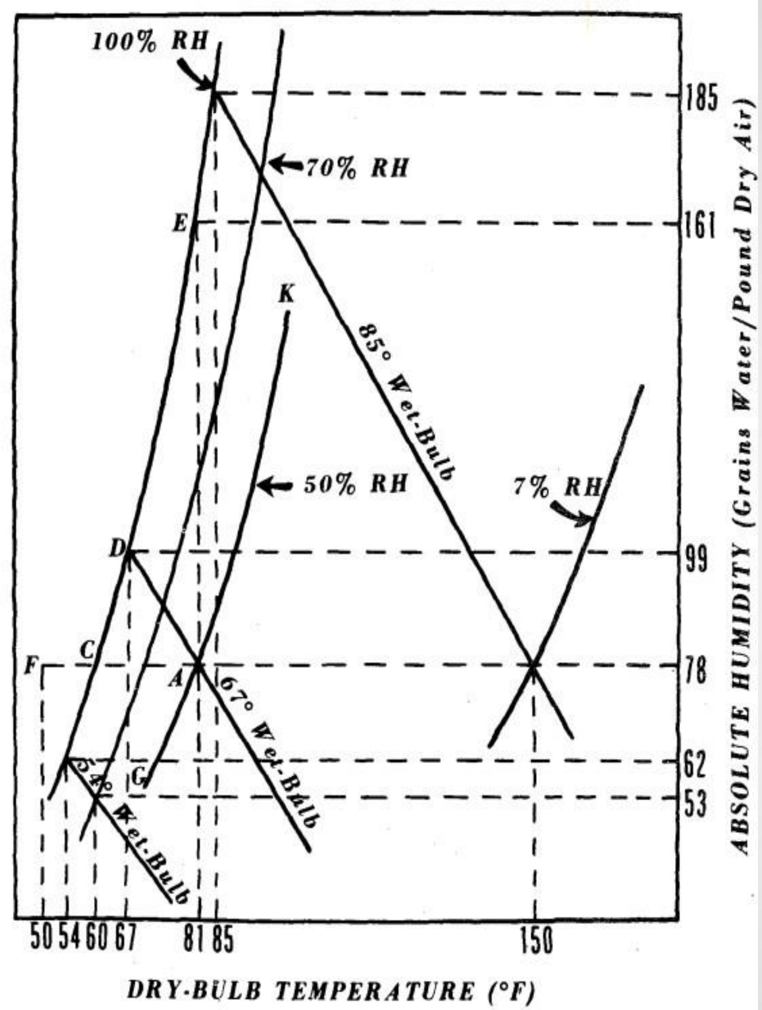


FIG. 3-1. Diagram of psychrometric chart showing the relationship of air temperature to humidity.

– The saturation humidity curve is boundary of a phase diagram, any point on the curve can determine either by the temperature or the absolute humidity.

Psychrometric or humidity chart

– FCA (dotted line) →is the absolute humidity (78 grains water/pound dry air); → which represents relationship between T and P.

– **At point C [Dew point (60 °F)]**→ the air is saturated with water vapor; and its temp., 60 °F (15.6 °C) referred to as *Dew Point.*

○ Dew point: is the temperature to which a given mixture of air and water vapor must be cooled to (at constant pressure) become saturated (i.e., hold max. amount of moisture without condensation).

→ At point F, if Cooling mixture < dew point [50°F ≈ 10°C]→ the water vapor condenses into a 2-phase system of saturated air (Point C) and droplet of free air.

→ To make the air usable for drying purposes **[without changing absolute humidity.** → raise air temp.

- **Raise temp. to 81 °F [27.2 °C, at Point A],** the air is not completely saturated and accept more vapor .

Psychrometric or humidity chart

The relative saturation (RS) is usually measures in term of % RH

- RS (% RH): is the ratio of partial pressure of water vapor in the air to the vapor pressure of free water at the same temperature.
- CDE curve is thus the curve for 100 % RH
- Curves of T vs H at a constant (RH) are plotted on the same axis at a specific intervals of RH such as GK curve with 50% (HR).
- The relative saturation can also express as % H or % AH → the ratio of the absolute humidity (FCA) to saturation humidity (CDE) at the same temperature.

$$\rightarrow \underline{\textbf{Air condition at Point (A)}} = \frac{\text{absolute humidity } (\textbf{FCA})}{\text{AH of saturat}\boldsymbol{d\ air}(\textbf{CDE at point E})}$$

$$= \frac{78}{161} \times \mathbf{100 = 48\%.}$$

Psychrometric or humidity chart

If air in at **point A (81°F), is used to dry a wet material**

↓

The difference in vapor pressure between surface water and the air led to

↓

Some of liquid evaporate

↓

latent heat of vaporization of the water →Cools the evaporated surface below air temp.

↓

The resultant difference in temp. cause

↓

Transfer heat from air to liquid at at rate α to the temp. difference i.e., the rate increased when temp. difference become larger.

↓

Eventually, Heat transfer = Heat of vapor vaporization, →T stabilization

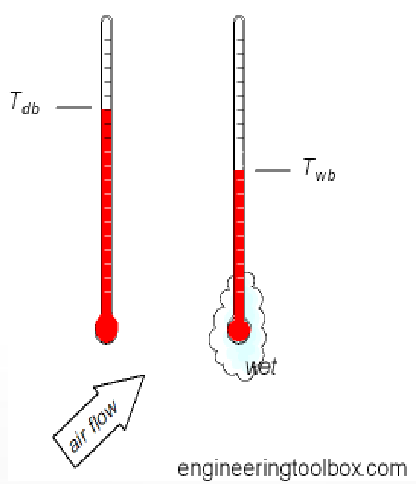
Psychrometric or humidity chart

- **Wet-bulb temperature;** define as equilibrium of heat transfer between air-liquid.
- **Wet-bulb temperature** is measured by thermometer whose bulb بصيلة الترمومتر is covered by wick بفتيل saturated with water).
- **Dry-bulb temperature** is the actual air temperature → measured by ordinary thermometer.

Wet bulb is a function of

T & H of the air used

for evaporation



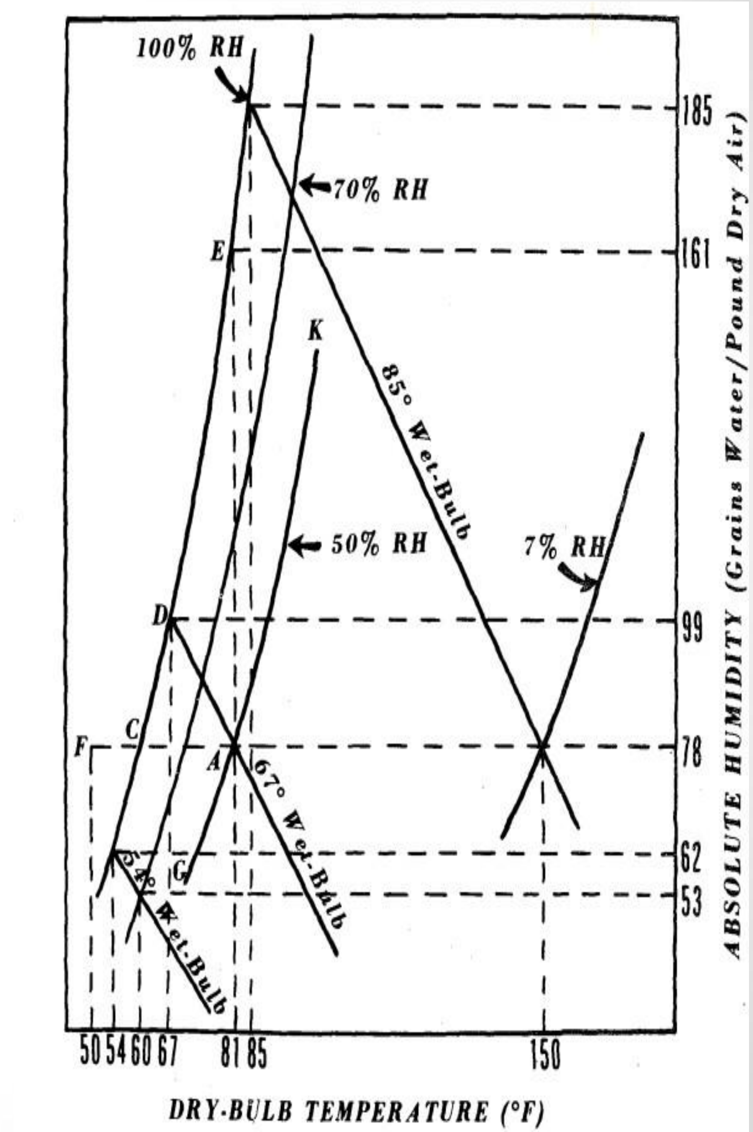


FIG. 3-1. Diagram of psychrometric chart showing the relationship of air temperature to humidity.

Theory of Drying

Drying involves both heat and mass transfer operations.

- **Heat** is transferred into the material to be dried to supply the latent heat required for vaporization of the moisture.
- **Mass transfer** is involved in
  - the diffusion of water through the material to the evaporating surface,
  - then subsequent evaporation of the water from the surface.
  - diffusion of the resultant vapor into the passing air stream.

Theory of Drying

The Drying process focused on the film of liquid at the surface of the material being dried.

- The rate of evaporation of this film is related to the rate of heat transfer by the equation: $\frac{dW}{d\Theta}$ ثيتا $= \frac{q}{\lambda} \rightarrow (1)$
- Where $\frac{dW}{d\Theta} \rightarrow$ is the rate of evaporation expressed as pound of water / hr.
  - $q \rightarrow$ is the overall rate of heat transfer
  - $(\lambda) \rightarrow$ is the latent heat of vaporization of water.
- Driving force for heat transfer is temperature differential.

- The rate of diffusion of moisture into the air stream is expressed by the equation: $\frac{dW}{d\Theta} = k'A\,(H_s - H_g) \rightarrow (2)$
- Where $\frac{dW}{d\Theta} \rightarrow$ is the rate of diffusion expressed as pounds of water per hr.
  - k is coefficient of mass transfer
  - A is the area of evaporating surface ,
  - $(H_s - H_g)$ is humidity differential

Theory of Drying

- The coefficient of mass transfer (k) is not constant but varies with the velocity of the air stream passing over the evaporating surface.
- After initial period of adjustment, the rate of evaporation is equal to the rate of diffusion of vapor mass transfer.

So, the rate of mass transfer $= \frac{dW}{d\Theta} = \frac{q}{\lambda} = k'A(H_s - H_g) \rightarrow (3)$

- If the overall rate of heat transfer (q) is expressed as the sum of the rates of heat transfer by convection, radiation, and conduction. equation is expanded to the form…

$$\frac{dW}{d\Theta} = \frac{q_c + qr + qk}{\lambda} = k'A(H_s - H_g) \rightarrow (4)$$

- From these equations we can conclude that: → The rate of drying may be accelerated by increasing any of the individuals in eq.4 → (The general principle for efficient drying)

Theory of Drying

- The rate of drying may be accelerated by increase of the individual terms in equation 4. $\frac{q_c+qr+qk}{\lambda} = k'A(H_s - H_g) \rightarrow (4)$

1. The rate of convection heat transfer (q_c) increased by increasing the air flow rate with raising the inlet air temperature
2. The rate of radiation heat transfer (q_r) speed up by introducing high temp. radiation heat source into thee drying chamber
3. The rate of conduction heat transfer (q_k) stepped up by reducing thickness of material being dried (↑ SA) and allowing it to come in contact with raised temp. surfaces
4. Increasing air velocity by increasing coefficient of mass transfer (sufficient turbulence to minimize boundary layer thickness)
5. Dehumidifying the inlet air → increasing the humidity differential (Hs – Hg).

Drying of solids

- The moisture in a solid can be expressed on a wet-weight or dry-weight basis.

Wet-weight basis: Loss on Drying (LOS)

- The water content of a material is calculated as % of the weight of the wet solid
- LOD is an expression of moisture content on a wet-weight basis.

$$\%LOD = \frac{\text{wt. of water in sample}}{\text{Total wt. of wet sample}} \times 100$$

- LOD is often determined using moisture balance, which has a heat source for rapid heating and a scale calibrated in % LOD.
- A weighted wet sample is placed on scale and allowed to dry till it is at constant wt. → obtain **%LOD.**

Dry-weight basis: moisture content

- The water content of a material is expressed as % of the weight of the dry solid

$$\%MC = \frac{\text{wt. of water in sample}}{\text{wt. of dry sample}} \times 100$$

Drying of solids

- Example: if exactly 5 g of moist solid is brought to a constant dry weight of 3g

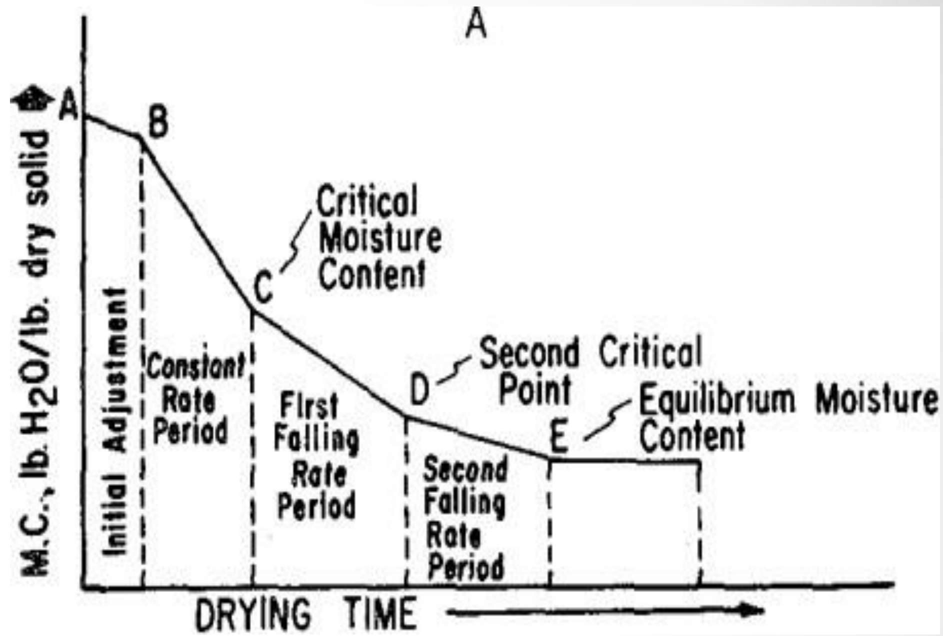
$$\%LOD = \frac{5-3}{5} \times 100 = 40\ \%$$

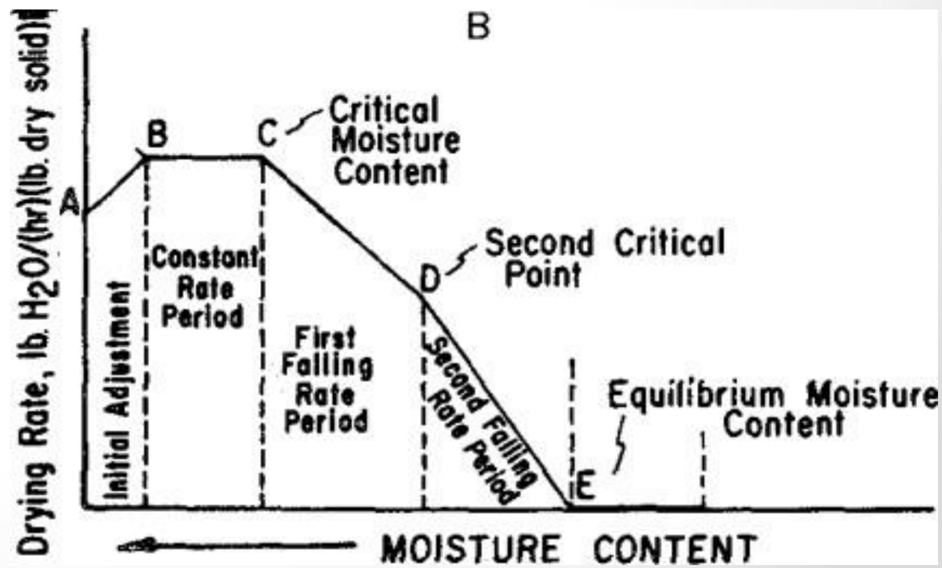
$$\%MC = \frac{5-3}{3} \times 100 = 66\ \%$$

- LOD can vary in any solid-fluid mix from >0 <100
- MC can vary from >0 to approach infinity

Behavior of Solids during Drying/Rate of Drying

- The study of drying rate is crucial to understand the solid behavior during drying.
- Rate of drying could be determined by suspending the wet sample on a scale in a drying cabinet and measuring the weight of dry sample as a function of time.
- The data from drying rate is plotted as moisture content Vs time.
  1. Drying rate vs moisture content
  2. Moisture content vs drying time

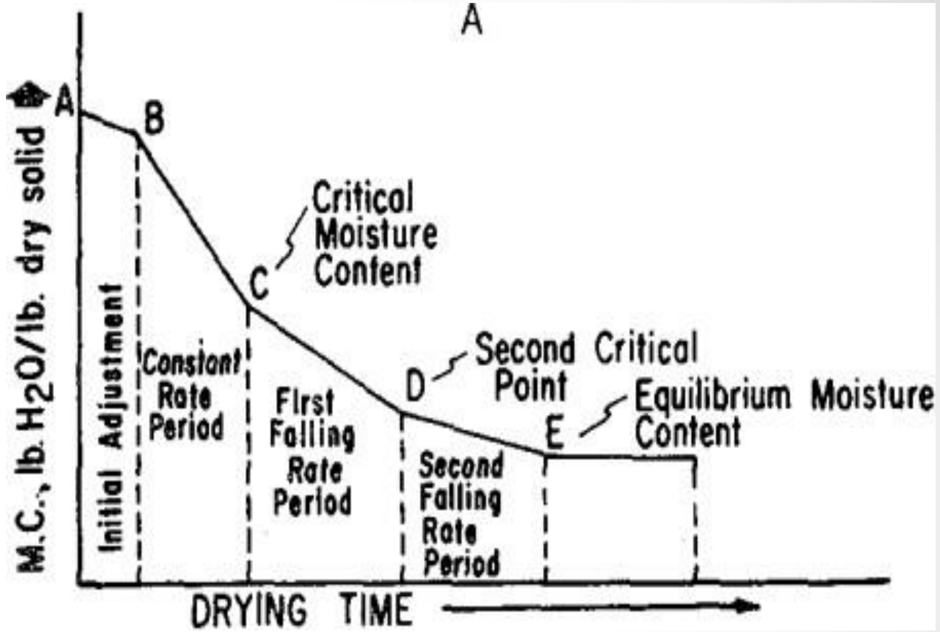


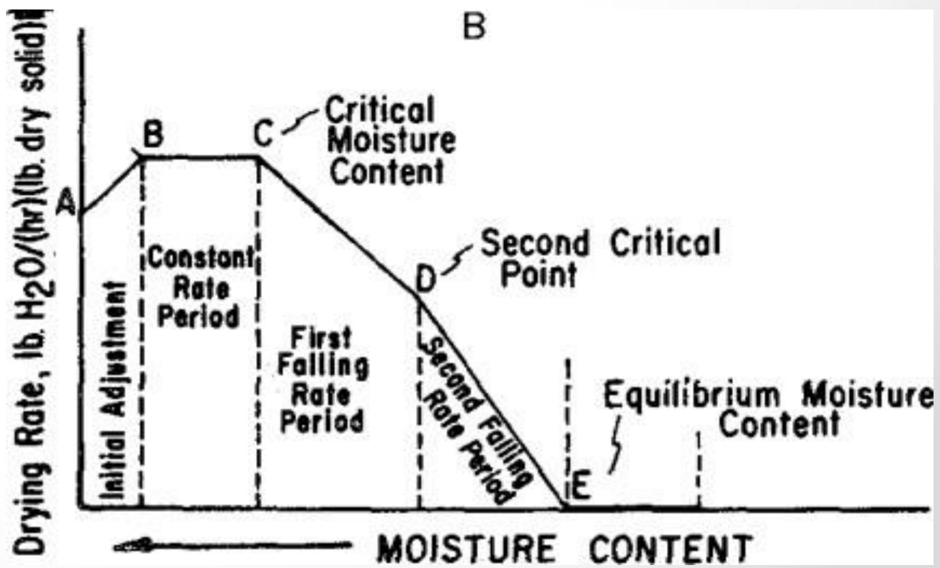


Behavior of Solids during Drying/Rate of Drying

- From A to B→ period of initial adjustment
  - the relationship is linear (that is, the decrease in drying rate is uniform)

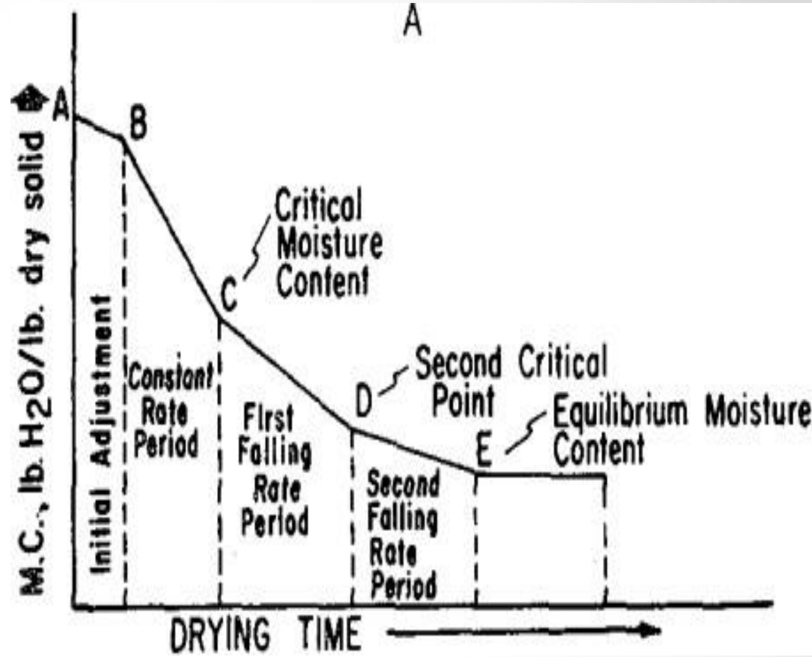
- At point B, After a period of initial adjustment,→ heating rates = cooling rate, → and the drying temperature stabilizes and remain constant.
  - there is a moisture film at the surface of the drying solid.



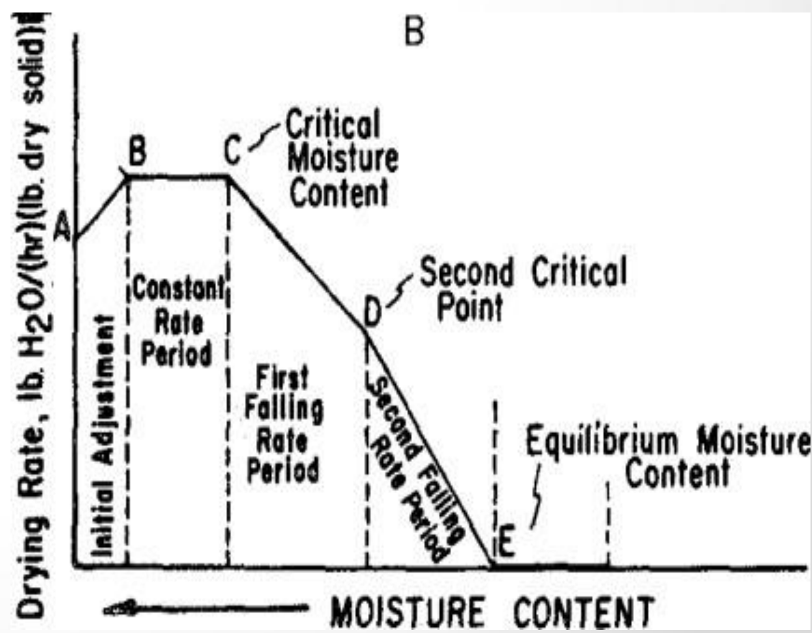


Behavior of Solids during Drying/Rate of Drying

- Between point B and C, the moisture evaporating from the surface is replaced by water diffusing from the interior of the solid at a rate equal to the rate of evaporation.
  - The rate of drying is constant. → known as the constant-rate period.
    (Evaporation rate = Diffusion rate).



- At point C, (critical moisture content) → the surface water is no longer replaced.
  - Drying spots begin to appear, → the rate of drying begins to fall off. يتراجع
- Between point C and D, [1st falling-rate period or unsaturated surface drying)].
  - the number and area of dry spots continue to grow, and the rate of drying falls steadily بثبات.

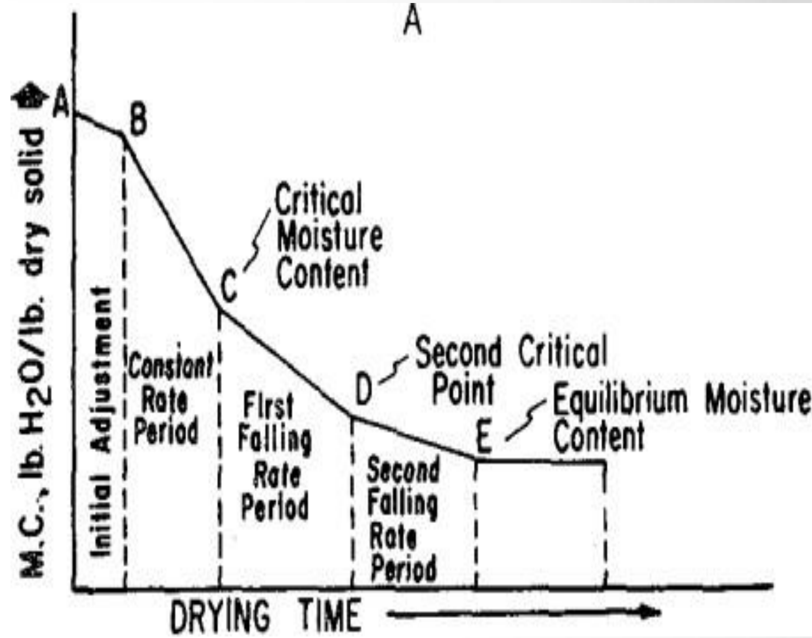


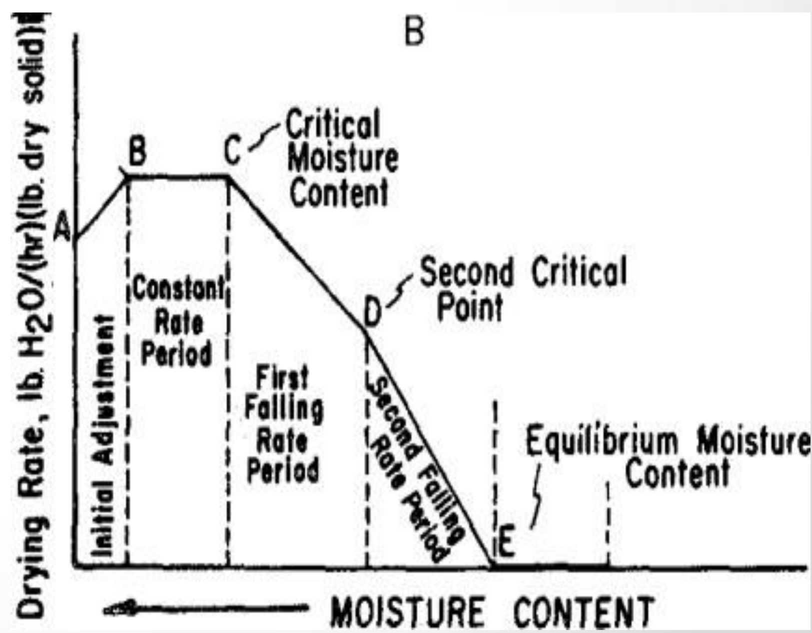
Behavior of Solids during Drying/Rate of Drying

- At point D, (second critical point)
  - the film of continuous water is completely evaporated, and the rate of drying depends on rate of diffusion of moisture to the surface of solid.

- point D and E, (2nd falling rate period)
  - the rate of drying falls rapidly than the first falling rate.

- At point E, (equilibrium moisture content)
  - When dying state is = zero, the equilibrium moisture period begins, and the solid is in equilibrium with its surroundings (i.e., Temp. and moisture content remain constant).
  - Continued drying after this point is a waste of time and energy.





Classification of Solid Based on Drying Behavior

Solids to be dried may be classified based on their drying behavior into:

1- Granular or crystalline solids

water is held in shallow and open surface pores مسام سطحية ضحلة ومفتوحة as well as in interstitial spaces مسافات بينية between particles that are easily accessible to the surface).

Ex: calcium sulfate, zinc oxide, magnesium oxide

2. Amorphous, fibrous or gelatinous solids

moisture is an integral part جزء لا يتجزأ of the molecular structure as well as being physically entrapped in fine capillaries and small interior pores.

Ex: starch, insulin and aluminum hydroxide.

N.B: Amorphous solids are difficult to dry than granular or crystalline solids.

Choice of method of drying

When considering how to dry a material, certain points should be considered:

- material to be dried is sensitive to heat or not.
- Physical nature of the material
- Nature of liquid to be removed
- The scale of operation

- Dryers can be classified based on method of heat transfer or method of sample handling.

- Based on method of heat transfer: → dryer design and energy requirement are important
- Based on sample handling: → we must consider the type of the substance to be dried
  - the presence or absence of agitation is the major criterion:

Types of Dryer

1. Static bed driers (convective driers) e.g., Tray drier.
2. Moving-bed dryers (Dynamic convective driers) e.g., Turbo-shelf drier
2. Fluidized bed drier.
3. Pneumatic dryers (convective drier):

   A)- Spray drier. B) spray congealing C) Flash Dryers.

Specialized Drying Methods

I. Radiation drying of wet solids :

   A)- Infra red heating. B)-Microwave drier

II. Freeze drying.

- ❑ Drier for dilute solution and suspensions

  A). Drum drier B). Spray drier

- ❑ Drier for sticky materials: e.g., Turbo-shelf drier

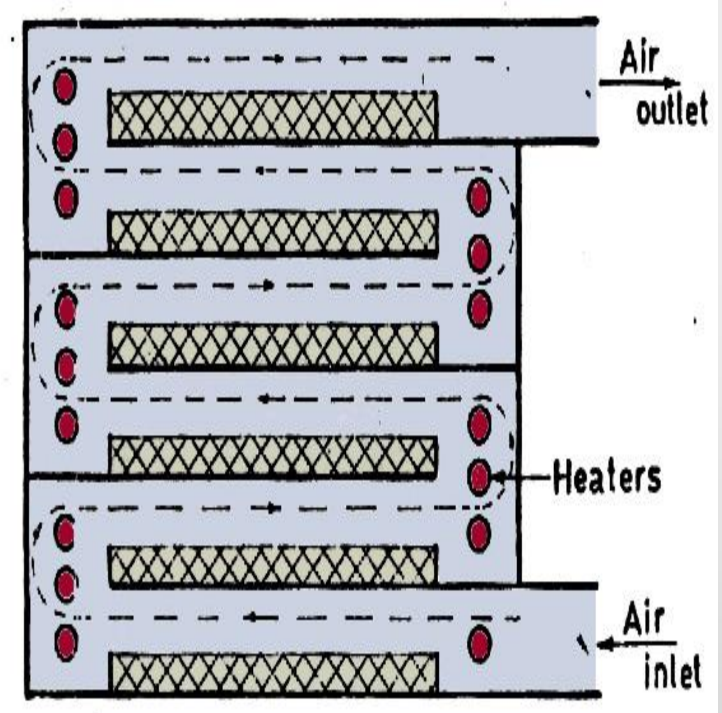
Static bed driers (convective driers) e.g., Tray drier.

- There is no relative movement among the solid particles being dried; only the bulk motion,
- The exposed surface can be increased by decreasing the thickness of the bed and allowing drying air to flow through it.

Construction:

- Tray dryer (Cabinet or shelf) consist of cabinet shelf or compartment in which the material to be dried is spread on trays.
- These dryer fitted with fans to circulate the air at a high speed.

Uses: for drying of tablet granules.



Directed-circulation tray drier

Moving-bed dryers (Dynamic convective driers)

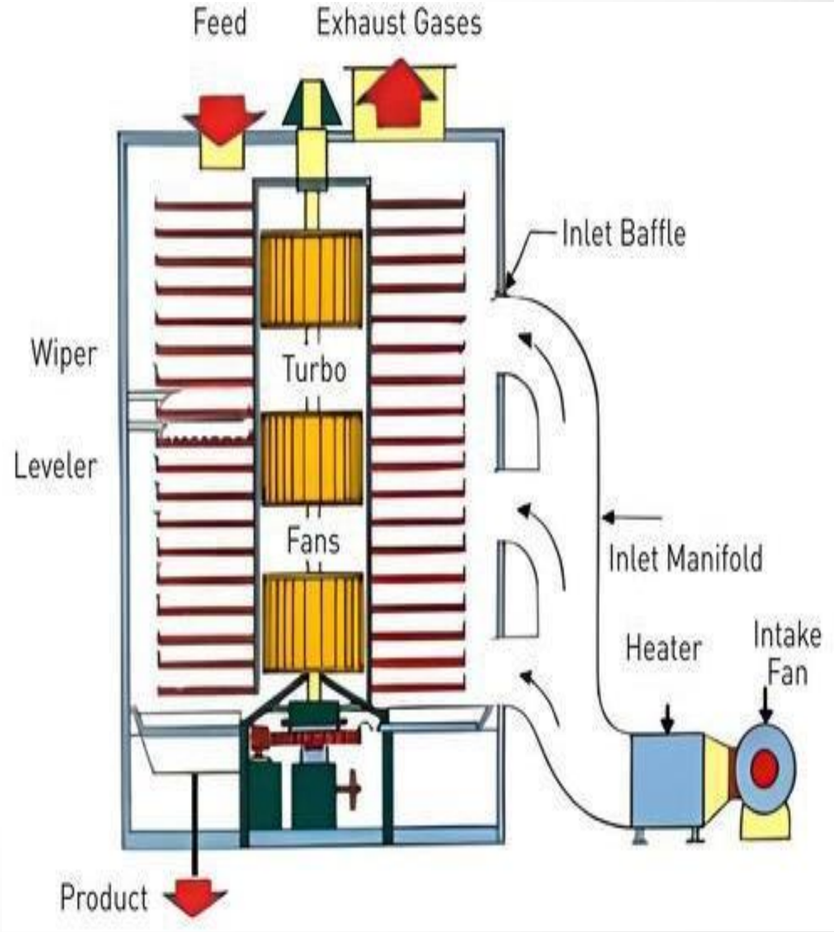
- The drying particles are partially separated so that they flow over each other.
- Motion may be induced by either gravity or mechanical agitation.

Turbo-shelf drier

- This is a continuous shelf, moving-bed dryer.

Construction:

- Consists of series of rotating annular trays arranged in a vertical stack
- Rotating slowly at 0.1 to 1.0 rpm.
- Heated air is circulated by turbo-type fans mounted in the stack center.

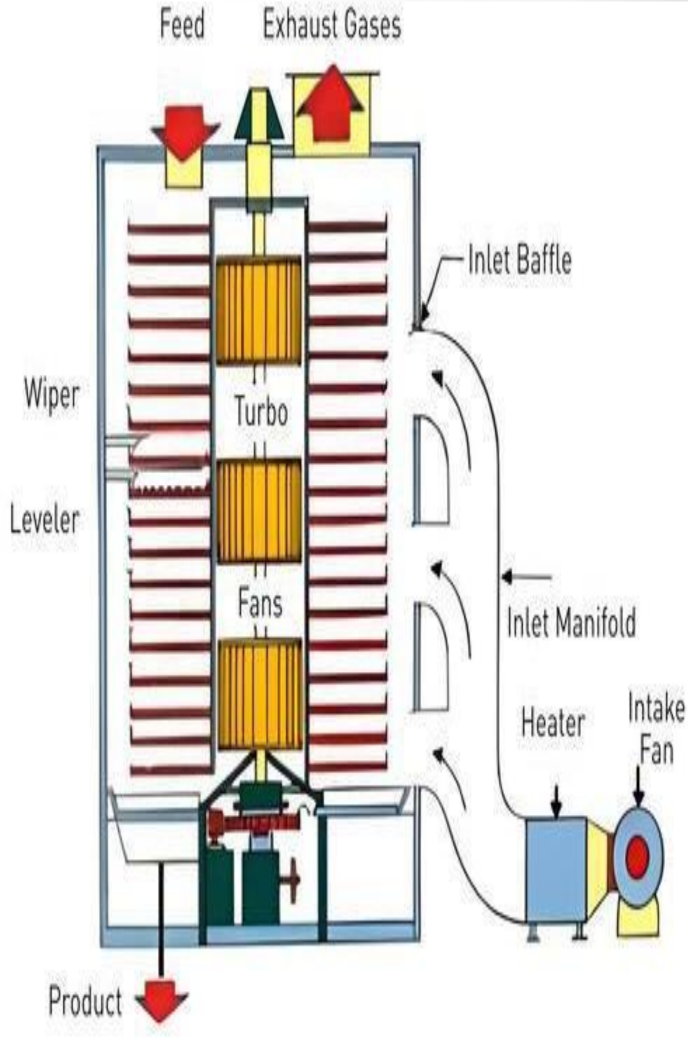


Moving-bed dryers (Dynamic convective driers)

Turbo-shelf drier

How it work?

- Wet mass fed from the roof of dryer; which leveled by a stationary wiper, then the dried material is pushed through radial slots onto the tray below.
- After each cycle the mass transfers to the next shelf until discharge at the bottom.
- Drying rate is faster than tunnel-dryer due to the continuous exposes to the air.



Dynamic convective driers Fluidized bed Drier

Fluidization (or fluidization) is a process like liquefaction whereby a granular material is converted from a static solid- like state to a dynamic fluid-like state.

Principles of fluidization.

- Solid particles are partially suspended in upward moving gas steam (the mixture behaves like a liquid) and the solid is fluidized.
- The intense mixing results in uniform conditions of temperature, and mass transfer (composition, and particle size distribution) throughout the bed than in static and moving bed.

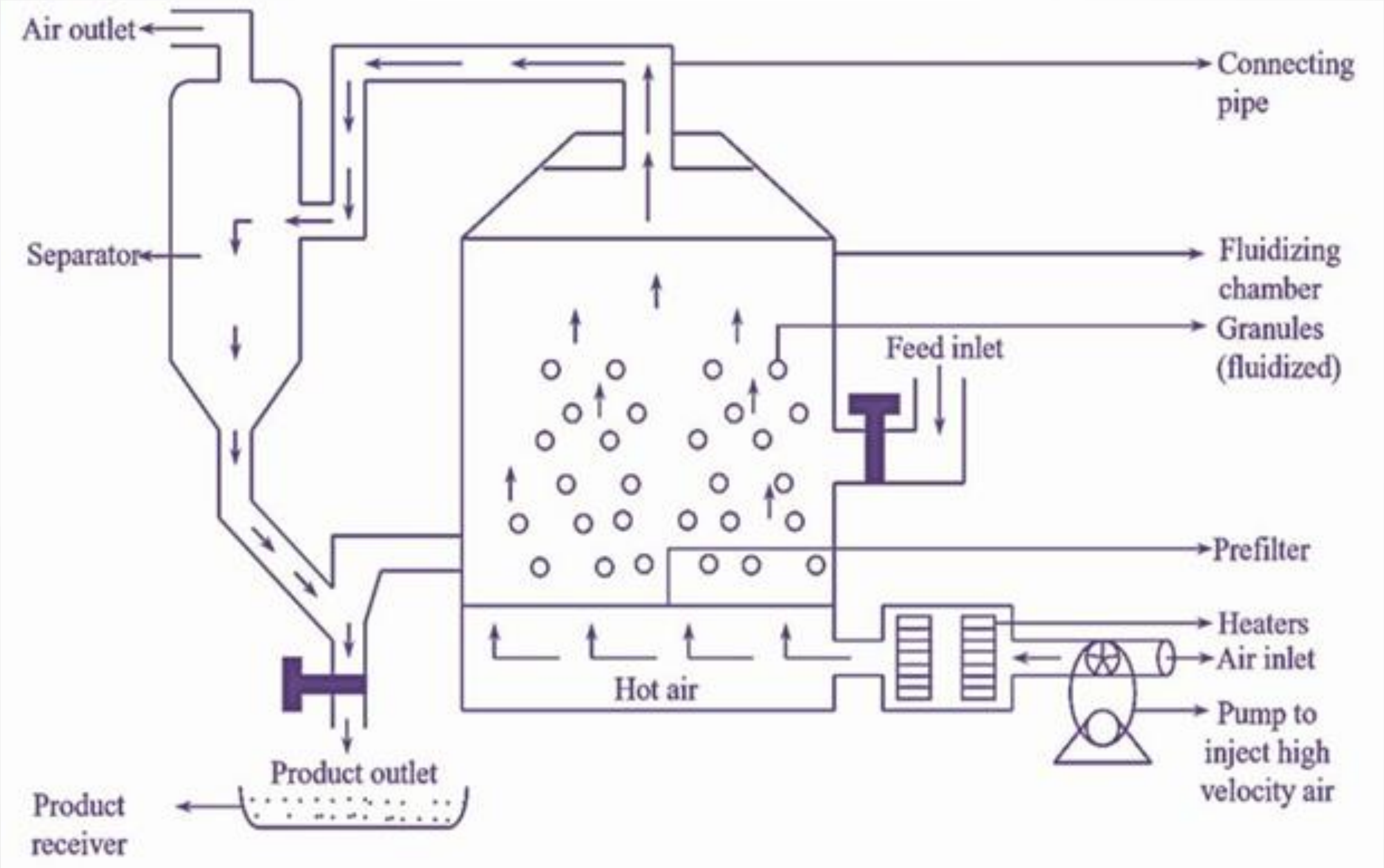
Uses: drying solids for tablet granulation

- The only requirement is that the resultant granules are not so wet that they stick together on drying nor completely dried to avoid the production of an excessive number of fine particles through attrition.

Problem:

- Attrition may occur
- develops static electric charges

Dynamic convective driers Fluidized bed Drier



Dynamic convective driers Fluidized bed Drier

❑ Advantages of fluidized-bed drying

1. High drying rate due to efficient heat and mass transfer (Drying time is short, 20-30 min/ 5-200 kg batch).
2. The drying occur at constant rate because all the surface of particles subjected to heat uniformly (not just from the surface of the bed)
3. More spherical free- flowing particles can be obtained → reduce the problems of aggregation and migration of color.
4. The fluidization technique is efficient for drying of granular solids, as each particle is surrounded by the drying gas.

Spray Drying and Spray Congealing

- Spray drying finds great utility in the pharmaceutical industry because of its rapid drying and the unique form of the final product.
- There are three major uses

1. drying heat sensitive materials
2. changing the physical form of materials for use in tablet and capsules.
3. Coating & encapsulation of both solid & liquid particles

- Both method has the same principle. → The principal difference between the two methods, is the means by which coating solidification is accomplished

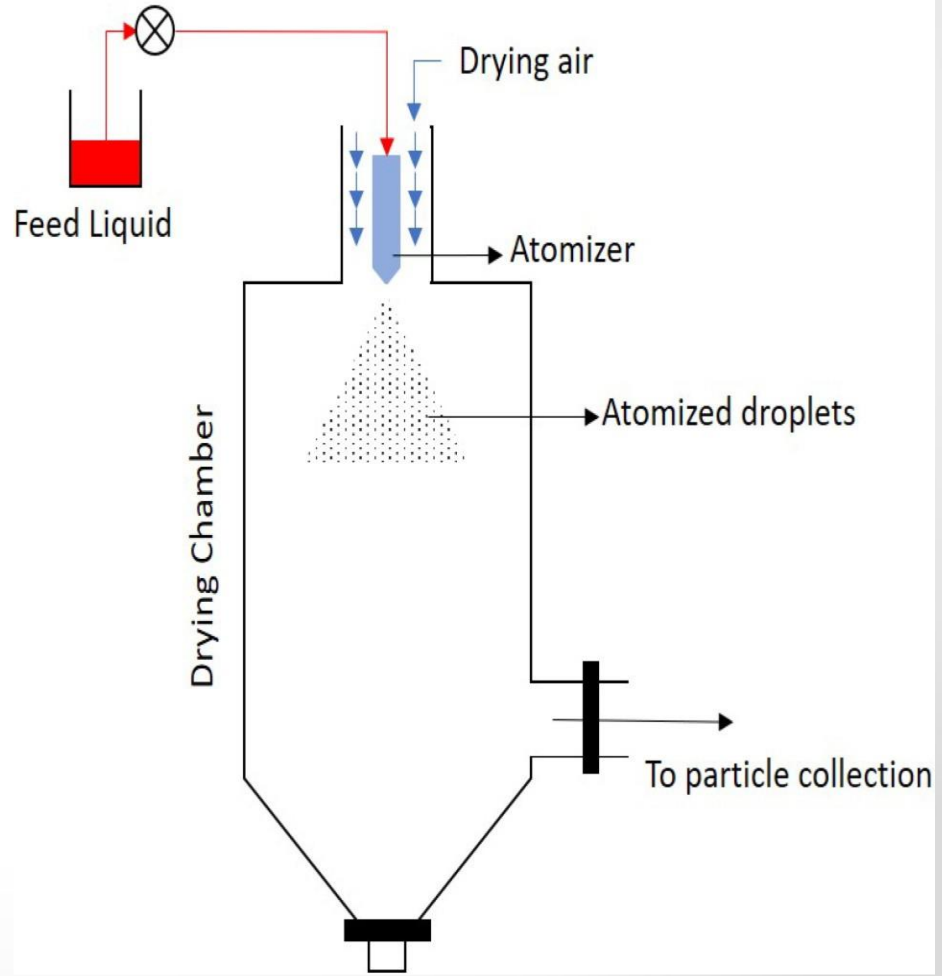
Spray Drying and Spray Congealing

1. Spray Dryers

Uses: It can handle only fluid materials such as solutions, slurries, and thin pastes.

Mechanism:

- Fluid Feeding is dispersed as fine droplets into a moving stream of hot gas,
- they evaporate rapidly before they reach the wall of the drying chamber,
- thus, the product dries into fine powder which is carried by gas current and gravity flow into a collection system.



1. Spray Dryers

- Spray drying is valuable in the material modification for use in tablet and capsule formulations because:
  - Drying process changes the shape, size, and bulk density of the dried product.
  - The spherical particles usually flow better than the same product dried by conventional method due to the particles are uniform in size and shape.
  - Spherical shape has the least SA, → thus ↓ air entrapment between the particles.

Characterization of spray dried products ;

- The spray-dried material are uniform in appearance and have characteristic shape, in the form of intact spheres, spheres with buds, ruptured hollow spheres with a small hole, or sphere fragments.

1. Spray Dryers

Spray-dried products Characterization arises from the drying process,

- When the liquid droplets contact the hot gas, the surface liquid is quickly evaporated, leaving a tough shell of solids.
- As drying proceed the liquid in interior of the droplet must diffuse through this shell
- The diffusion rate of the liquid is slower than heat transfer through the shell of interior of the droplet. The internal pressure causes the droplet to swell, and the shell becomes thinner, allowing faster diffusion.
- If the shell is nonelastic or impermeable, it ruptures, producing either fragments or budlike forms.

2. Spray congealing (chilling):

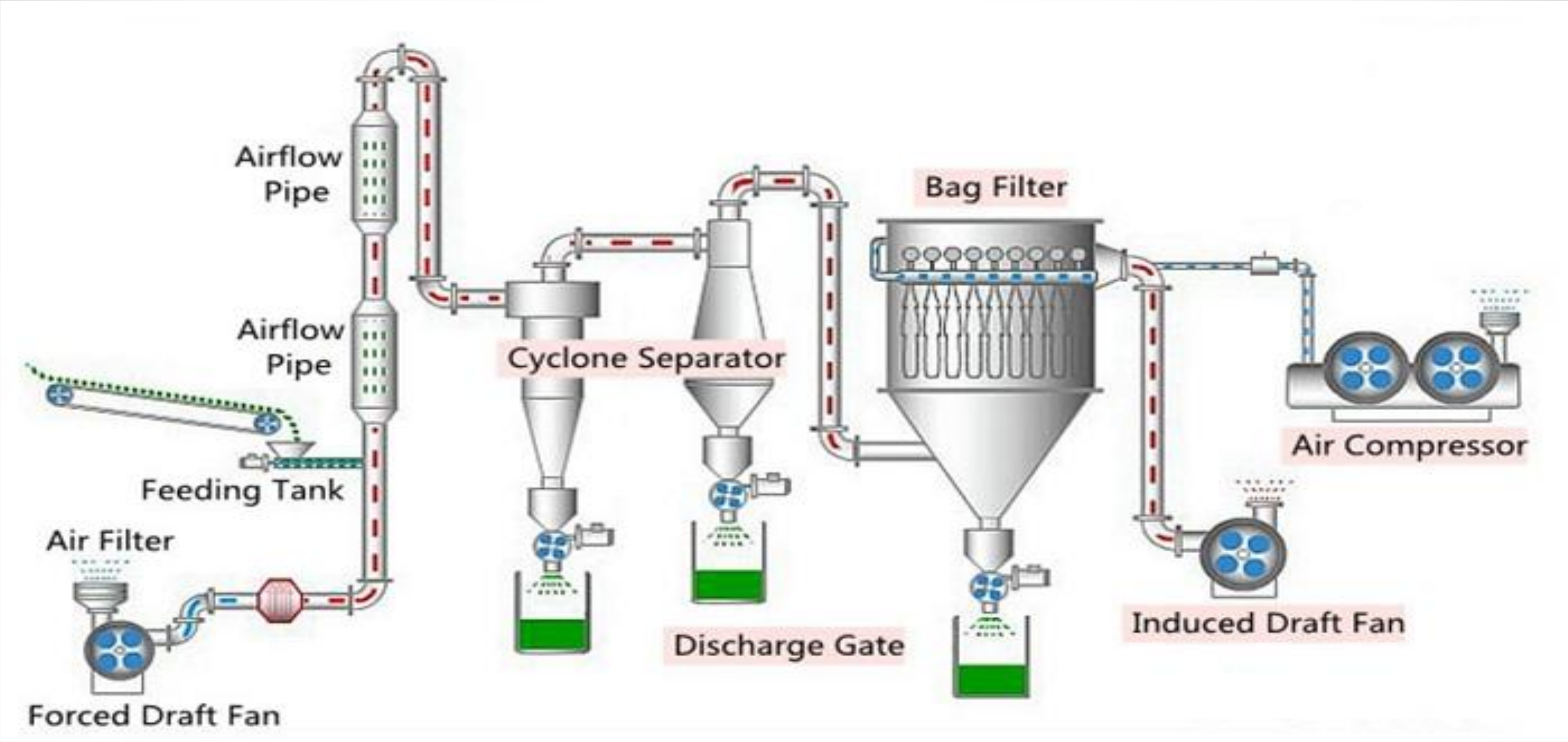
Mechanism:

- This process involve suspending the particles in a molten coating material.
- Pumping the resultant slurry into a spray dryer in which cold air is circulated for the removal of solvent
- The slurry droplet congeals on contact with air; → dried particles are collected.

Uses

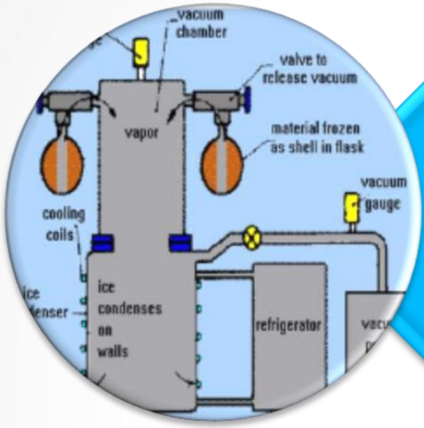
- It is useful in coating and encapsulation → to mask taste and odour and for sustained-release formulations.

3. Flash Dryers



- The moistened solid is suspended in a finely divided state in a high velocity (3000-6000 feet/min) at high temperature (300-1300°F) air stream.
- Drying process called flash drying, because the drying is a short-time process (drying air T can drop from 1300 °F to 600 °F in 2 Sec).

Specialized Drying Methods

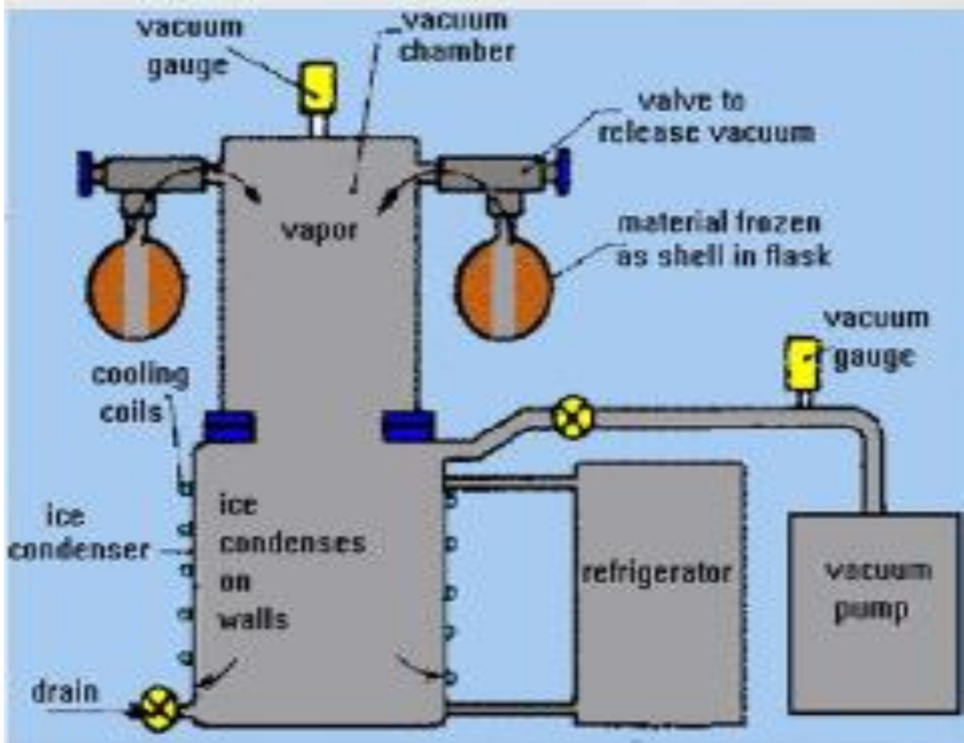


A. Freeze Dryers.

B. Microwave Drying

A. Freeze Drying

- Freeze drying is also referred to lyophilization, or sublimation.
- The drying of heat-sensitive materials must be dehydrated to a solid state to maintain their stability; they are frozen first and then subjected to high vacuum and heat (supplied by conduction, radiation, or both) so the frozen liquid in the dried material sublimes (froths), leaving only the solid.



It consist of 4 basic components:

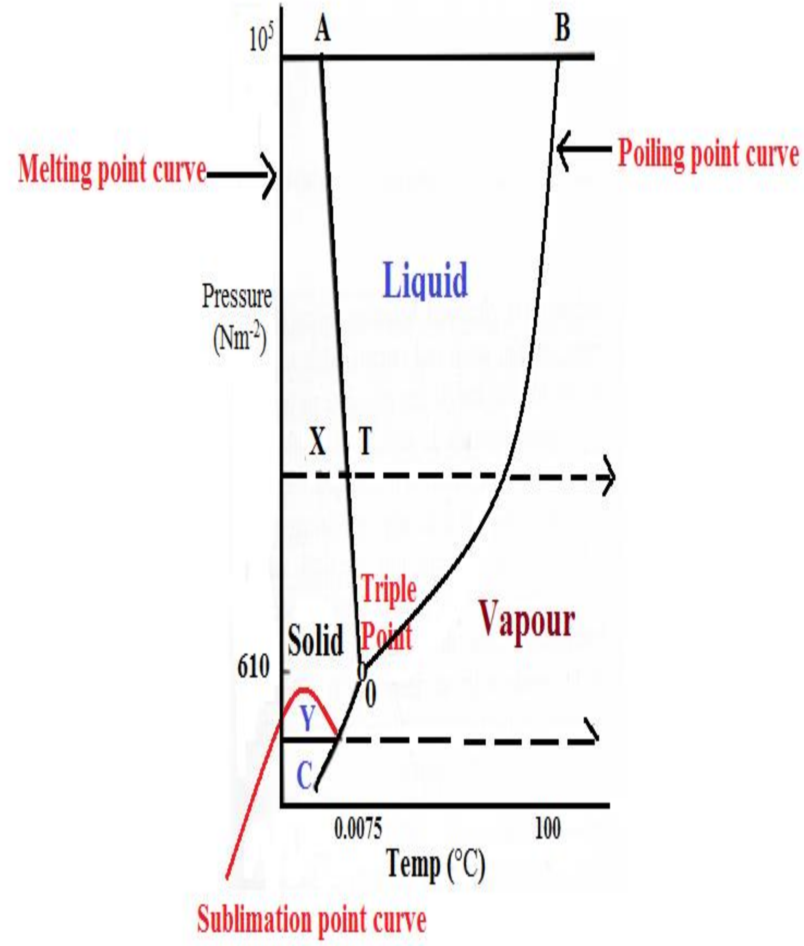
1. chamber for vacuum drying,
2. vacuum source
3. heat source,
4. vapor removal system.

A. Freeze Drying

Mechanism: To stabilize these types of materials we use phenomena of sublimation

- In freeze drying the water containing dissolved solids passes directly from solid state (ice) to vapor state without passing through the liquid state (melting).

- As shown in the schematic pressure-temperature diagram for water → the sublimation can take place at P & T < the triple point [eutectic point; 4.579 mm Hg and 0.0099 °C).
- The diagram consists of 3-separate areas representing the phases of water, solid, liquid, and vapour .
- The point O is the only point where all the three phases can coexist, and is known as the triple point .



A. Freeze Drying

- The method is used for heat sensitive materials (can not be dried by any other heat method) and that react with oxygen. These include biological products, e.g.
  - **Antibiotics.**
  - **blood serum.**
  - **Plasma**
  - **hormones**
  - **vaccines.**
  - **food stuffs.**
  - **bacterial cultures.**
- The dried product is reconstitution (re-dissolved or re-suspended) by the addition of water before use

Stages of the freeze-drying process

1. **Pre-Freezing stage:**

- The liquid material is frozen before the application of vacuum to avoid frothing (foaming).
- Frozen must be below the (TPT) for pure water (below or at −20 °C)

2. **Vacuum:**

- The containers and the frozen material must be connected to a vacuum source to reduce the pressure sufficiently below the triple point and remove the larger volumes of low-pressure vapor formed during drying.
- Rotary pumps are used on small scale, and ejector pumps on large-scale

3. **Sublimation stage (Primary drying):**

- The latent heat of sublimation must be provided, and the vapour removed
- Under these conditions, the ice slowly sublimes; remove the unbound water, leaving a porous solid that still contains about 0.5% moisture after primary drying.

4. **Secondary drying:**

- It is used to remove bound water or traces of water left after primary drying.
- It is performed by raising the temperature as high as (up to 50 °C) or desiccant is used to carry secondary drying.

B. Microwave Drying

- Using microwave energy to dry solids represents an important shift from conventional drying techniques.

Mechanism:

- here instead of applying heat externally to a material, energy in form of microwaves is converted into internal heat by interaction with material itself
- This permits extremely rapid heat transfer throughout the material.

Rapid drying at low temperature

- the moisture is mobilized as a vapor rather than a liquid, and its movement to the surface can be extremely rapid because it does not depend on mass concentration gradients or on slow liquid diffusion rate