THAPAR INSTITUTE
OF ENGINEERING & TECHNOLOGY
(Deemed to be University)

# Mass Transfer-I

## Drying

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# Drying

- Drying is commonly the **last stage** in a manufacture process.
- **Drying** is the final **removal of water** from material (**usually by heat)**

**Non –thermal drying**

**1-** As Squeezing wetted sponge

**2-**Adsorption by desiccant (desiccation)

**3-** Extraction.

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# Purposes of drying

In pharmaceutical technology, drying is carried out for one or more of **the following reasons**:

**1-** To avoid or eliminate moisture which may **lead to corrosion** and decrease the product or **drug stabilit**y.

**2-** To **improve** or keep the **good properties** of a material, e.g. **flowability,** compressibility.

**3-** To r**educe** the cost **of transportation** of large **volume** materials ( liquids)

**4-** To make the **material easy** or more suitable for **handling.**

**5-** Preservative.

**6-** The **final step in**: Evaporation- Filtration- Crystallization.

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## Difference between drying and evaporation

1- In **drying** processes, the main operation usually carried out on **solid materials,** e.g. powders, 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- In most cases, **drying** involves the **removal** of water at **temperatures below** its boiling **point**, whereas **evaporatio**n means 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**.

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# Introduction

- **Drying is the removal of small amounts of water form the wet materials (solid). also used to removed of other organic liquids, such as benzene or organic solvent from solid.**

- **There are two different processes are used in drying:**

1. **Batch processes.**
2. **Continuous process.**

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# Theory

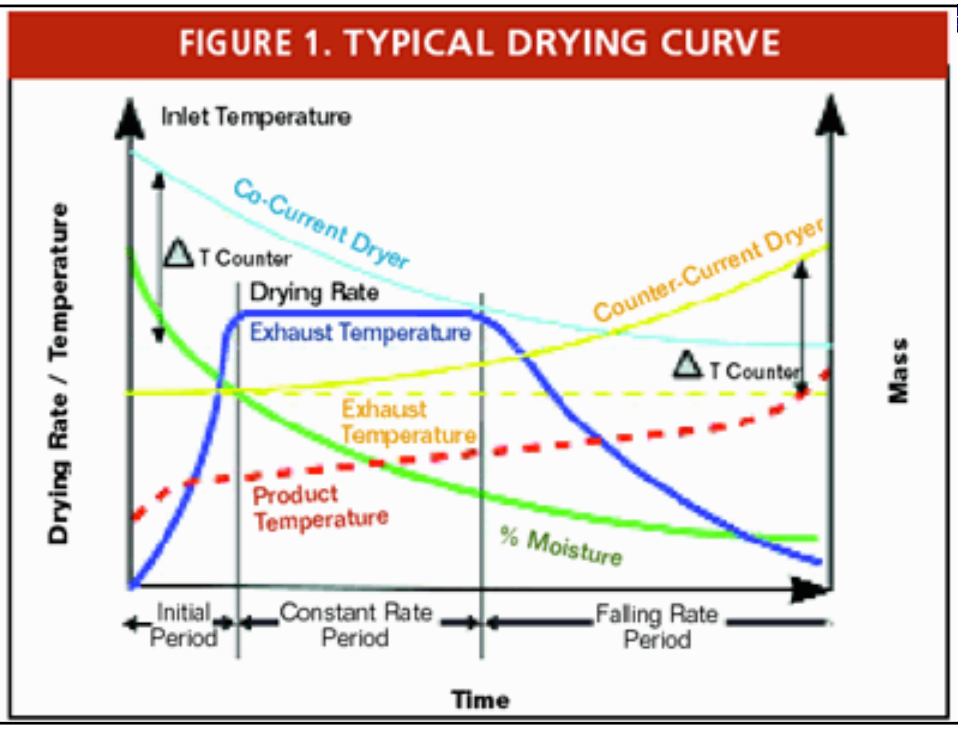
$$R = \frac{-Ls}{A} \cdot \frac{dx}{dt}$$

$$X_t = \frac{W - W_s}{W_s}$$

$$X = X_t - X^*$$

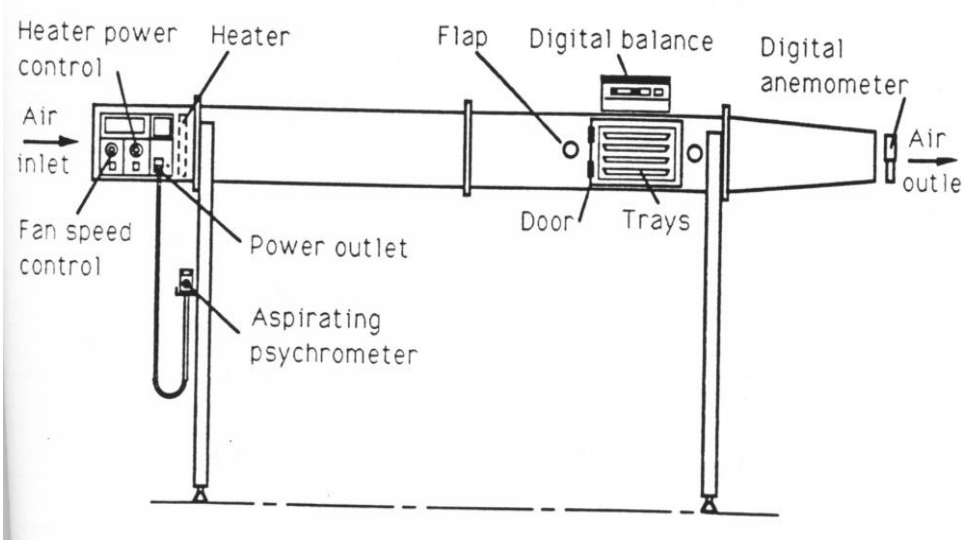
- $X_t$ = W/ LS

**where :**
**R:** drying rate in kg $H_2O$ /h.$m^2$
**Ls :** weight of dry solid in kg
**A :** exposed surface area in $m^2$
**dx :** change in water content in kg $H_2O$/kg dry solid
**dt :** change in time in hour
**x:** water content in kg $H_2O$/kg dry solid
**$X_t$ :** Moisture content at time t (kg water/kg dry solid)
**W :** Weight of water (kg water)
**$X^*$ :** is equilibrium water contents
**$W_s$:** Weight of dry solid (kg)






# Schematic Diagram



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# Equilibrium curve

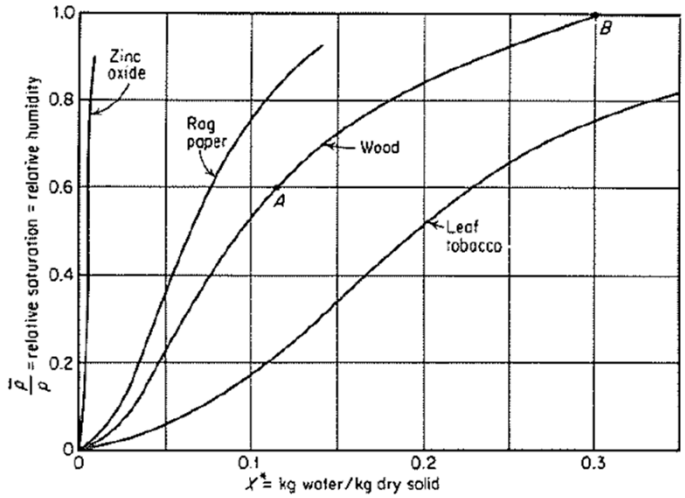


**Figure 12.1** Equilibrium water content of some common solids at about 25°C. *(From "International Critical Tables," vol. II, pp. 322–325. with permission.)*

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# Hysteresis

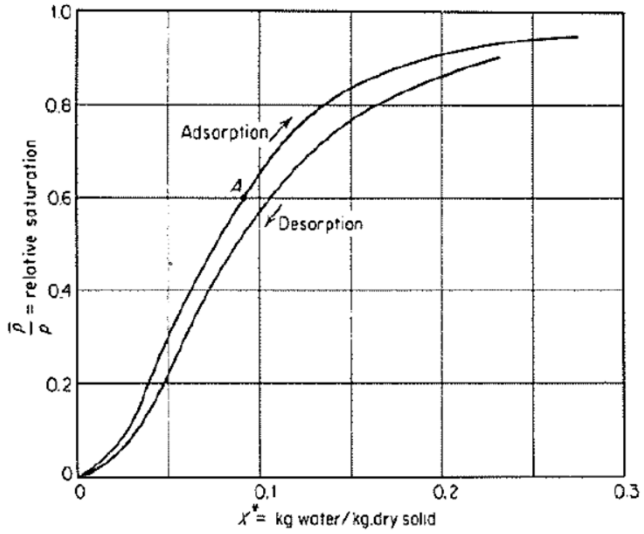


**Figure 12.2** Equilibrium water content of a sulfite pulp, showing hysteresis.

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# Drying of soluble solids

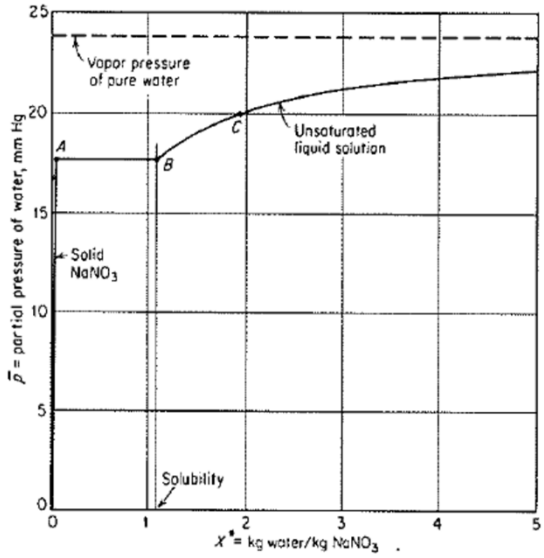


**Figure 12.3** Equilibrium moisture content of sodium nitrate at 25°C.

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# Definition of related terms

*Moisture content, wet basis.* The moisture content of a solid or solution is usually described in terms of weight percent moisture, and unless otherwise qualified this is ordinarily understood to be expressed on the wet basis, i.e., as (kg moisture/kg wet solid)100 = [kg moisture/(kg dry solid + kg moisture)]100 = $100X/(1 + X)$.

*Moisture content, dry basis.* This is expressed as kg moisture/kg dry solid = $X$. Percentage moisture, dry basis = $100X$.

*Equilibrium moisture* $X^*$. This is the moisture content of a substance when at equilibrium with a given partial pressure of the vapor.

*Bound moisture*. This refers to the moisture contained by a substance which exerts an equilibrium vapor pressure less than that of the pure liquid at the same temperature.

*Unbound moisture*. This refers to the moisture contained by a substance which exerts an equilibrium vapor pressure equal to that of the pure liquid at the same temperature.

*Free moisture*. Free moisture is that moisture contained by a substance in excess of the equilibrium moisture: $X - X^*$. Only free moisture can be evaporated, and the free-moisture content of a solid depends upon the vapor concentration in the gas.



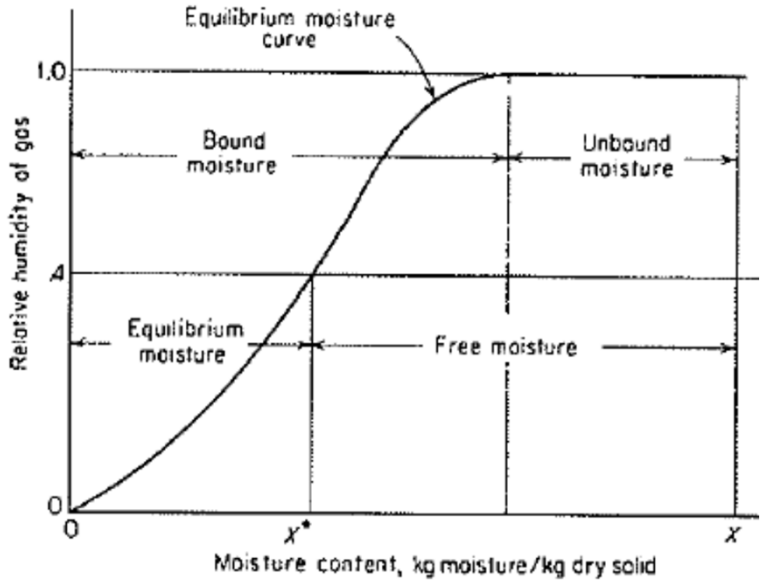



**Figure 12.5** Types of moisture.

**Illustration 12.1** A wet solid is to be dried from 80 to 5% moisture, wet basis. Compute the moisture to be evaporated, per 1000 kg of dried product.

SOLUTION

$$\text{Initial moisture content} = \frac{0.80}{1 - 0.80} = 4.00 \text{ kg water/kg dry solid}$$

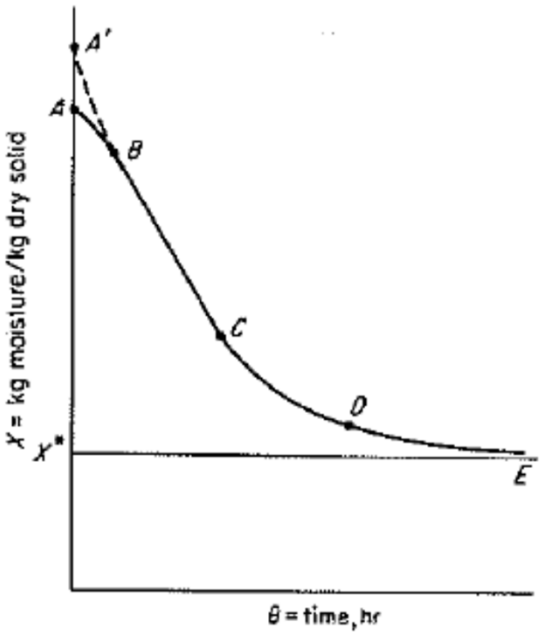
$$\text{Final moisture content} = \frac{0.05}{1 - 0.05} = 0.0527 \text{ kg water/kg dry solid}$$

$$\text{Dry solid in product} = 1000(0.95) = 950 \text{ kg}$$

$$\text{Moisture to be evaporated} = 950(4 - 0.0527) = 3750 \text{ kg}$$



# Batch drying



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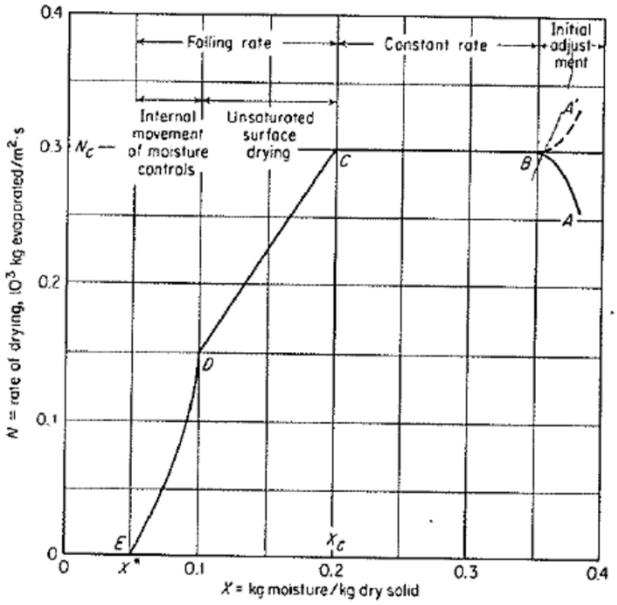


**Figure 12.10** Typical rate-of-drying curve, constant drying conditions.

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# Time of drying

The rate of drying is, by defintion,

$$N = \frac{-S_S\, dX}{A\, d\theta}$$

Rearranging and integrating over the time interval while the moisture content changes from its initial value $X_1$ to its final value $X_2$ gives

$$\theta = \int_0^{\theta} d\theta = \frac{S_S}{A} \int_{X_2}^{X_1} \frac{dX}{N} \qquad (12.3)$$

1. *The constant-rate period.* If the drying takes place entirely within the constant-rate period, so that $X_1$ and $X_2 > X_c$ and $N = N_c$, Eq. (12.3) becomes

$$\theta = \frac{S_S(X_1 - X_2)}{AN_c} \qquad (12.4)$$

2. *The falling-rate period.* If $X_1$ and $X_2$ are both less than $X_c$, so that drying occurs under conditions of changing $N$, we proceed as follows:
   a. *General case.* For any shape of falling-rate curve whatsoever, Eq. (12.3) can be integrated graphically by determining the area under a curve of

$1/N$ as ordinate, $X$ as abscissa, the data for which can be obtained from the rate-of-drying curve.



b. *Special case.* $N$ is linear in $X$, as in the region $BC$ of Fig. 12.10. In this case,

$$N = mX + b \qquad (12.5)$$

where $m$ is the slope of the linear portion of the curve and $b$ is a constant. Substitution in Eq. (12.3) provides

$$\theta = \frac{S_S}{A}\int_{X_2}^{X_1}\frac{dX}{mX + b} = \frac{S_S}{mA}\ln\frac{mX_1 + b}{mX_2 + b} \qquad (12.6)$$

But since $N_1 = mX_1 + b$, $N_2 = mX_2 + b$, and $m = (N_1 - N_2)/(X_1 - X_2)$, Eq. (12.6) becomes

$$\theta = \frac{S_S(X_1 - X_2)}{A(N_1 - N_2)}\ln\frac{N_1}{N_2} = \frac{S_S(X_1 - X_2)}{AN_m} \qquad (12.7)$$

where $N_m$ is the logarithmic average of the rate $N_1$, at moisture content $X_1$, and $N_2$ at $X_2$.

Frequently the entire falling-rate curve can be taken as a straight line between points $C$ and $E$ (Fig. 12.10). It is often assumed to be so for lack of more detailed data. In this case

$$N = m(X - X^*) = \frac{N_c(X - X^*)}{X_c - X^*} \tag{12.8}$$

and Eq. (12.7) becomes

$$\theta = \frac{S_S(X_c - X^*)}{N_c A} \ln \frac{X_1 - X^*}{X_2 - X^*} \tag{12.9}$$



**Illustration 12.3** A batch of the solid for which Fig. 12.10 is the drying curve is to be dried from 25 to 6% moisture under conditions identical to those for which the figure applies. The initial weight of the wet solid is 160 kg, and the drying surface is 1 $m^2$/40 kg dry weight. Determine the time for drying.

SOLUTION The total weight of the batch is unimportant. $S_S/A = 40$. At 25% moisture, $X_1 = 0.25(1 - 0.25) = 0.333$ kg moisture/kg dry solid. At 6% moisture, $X_2 = 0.06/(1 - 0.06) = 0.064$ kg moisture/kg dry solid. Inspection of Fig. 12.10 shows that both constant- and falling-rate periods are involved. The limits of moisture content in the equations for the different periods will be chosen accordingly.

***Constant-rate period*** This is from $X_1 = 0.333$ to $X_c = 0.200$. $N_c = 0.30 \times 10^{-3}$. Eq. (12.4):

$$\theta = \frac{S_S(X_1 - X_c)}{AN_c} = \frac{40(0.333 - 0.200)}{1(0.30 \times 10^{-3})} = 17\,730 \text{ s}$$

***Falling-rate period*** This is from $X_c = 0.200$ to $X_2 = 0.064$. Use Eq. (12.3). The following table is prepared from data of Fig. 12.10:

| $x$ | 0.20 | 0.18 | 0.16 | 0.14 | 0.12 | 0.10 | 0.09 | 0.08 | 0.07 | 0.064 |
|---|---|---|---|---|---|---|---|---|---|---|
| $10^3 N$ | 0.300 | 0.266 | 0.239 | 0.208 | 0.180 | 0.150 | 0.097 | 0.070 | 0.043 | 0.025 |
| $\frac{1}{N} \times 10^{-3}$ | 3.33 | 3.76 | 4.18 | 4.80 | 5.55 | 6.67 | 10.3 | 14.3 | 23.3 | 40.0 |

A curve, not shown, is prepared of $1/N$ as ordinate, $X$ as abscissa, and the area under the curve between $X = 0.20$ and 0.064 is 1060. Eq. (12.3):

$$\theta = \frac{40}{1}(1060) = 42\,400 \text{ s}$$



The total drying time is therefore $17\,730 + 42\,400 = 60\,130$ s $= 16.7$ h.

Alternatively, since the drying curve is straight from $X = 0.20$ to 0.10, Eq. (12.7) can be used in this range of moisture content,

$$\theta = \frac{S_S(X_c - X_D)}{A(N_c - N_D)} \ln \frac{N_c}{N_D} = \frac{40(0.20 - 0.10)}{1(0.30 - 0.15 \times 10^{-3})} \ln \frac{0.30 \times 10^{-3}}{0.15 \times 10^{-3}} = 18\,500 \text{ s}$$

Graphical integration in the range $X = 0.1$ to 0.064, through Eq. (12.3), provides an additional 23 940 s, so that the falling-rate time is $18\,500 + 23\,940 = 42\,440$ s.

As an approximation, the falling rate can be represented by a straight line from $C$ to $E$ (Fig. 12.10). The corresponding falling-rate time is, by Eq. (12.9),

$$\theta = \frac{S_S(X_c - X^*)}{N_c A} \ln \frac{X_c - X^*}{X_2 - X^*} = \frac{40(0.20 - 0.05)}{(0.30 \times 10^{-3})(1)} \ln \frac{0.20 - 0.05}{0.064 - 0.05} = 47\,430 \text{ s}$$

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# Mechanism of batch drying

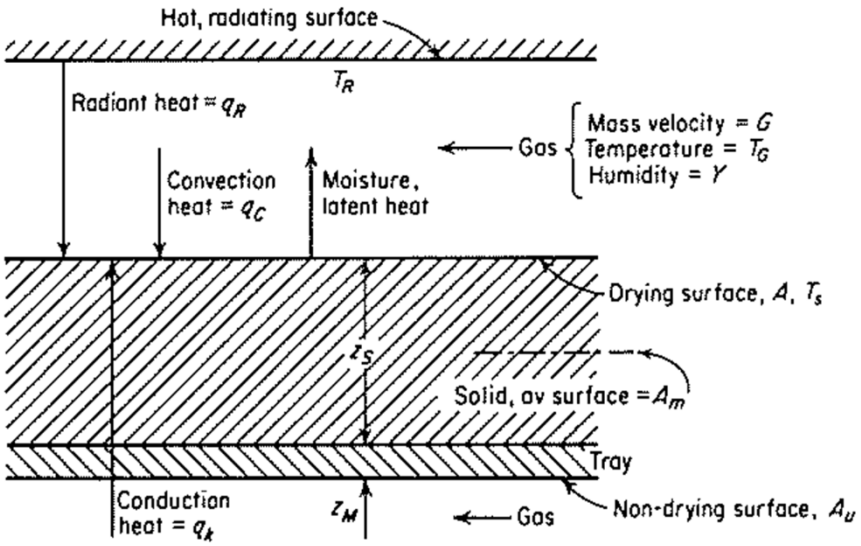


**Figure 12.11** Constant-rate drying.

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The rate of evaporation and the surface temperature can then be obtained by a heat balance [46]. If $q$ represents the total heat arriving at the surface, then

$$q = q_c + q_R + q_k \tag{12.10}$$

If we neglect the heat required to superheat the evaporated moisture to the gas temperature and consider only the latent heat of vaporization $\lambda_s$, the flux of evaporation $N_c$ and the flux of heat flow are related,

$$N_c\lambda_s = q \tag{12.11}$$

The heat received at the surface by convection is controlled by the appropriate convection heat-transfer coefficient $h_c$,

$$q_c = h_c(T_G - T_s) \tag{12.12}$$

The heat received by radiation can be estimated by the usual means [50, 55] and can also be expressed as a heat-transfer coefficient $h_R$,†

$$q_R = \varepsilon(5.729 \times 10^{-8})(T_R^4 - T_s^4) = h_R(T_R - T_s) \tag{12.13}$$

$$h_R = \frac{\varepsilon(5.729 \times 10^{-8})(T_R^4 - T_s^4)}{T_R - T_s} \tag{12.14}$$



where $\varepsilon$ is the emissivity of the drying surface and $T_R$ and $T_s$ are the absolute temperatures of the radiating and drying surfaces. The heat received by convection and conduction through the solid can be computed by the usual methods for heat transfer through a series of resistances,

$$q_k = U_k(T_G - T_s) \tag{12.15}$$

$$U_k = \frac{1}{(1/h_c)(A/A_u) + (z_M/k_M)(A/A_u) + (z_S/k_S)(A/A_m)} \tag{12.16}$$

where $h_c$ = convection coefficient for tray; ordinarily can be taken as same as that for drying surface
$k_M$, $k_s$ = thermal conductivities of tray material and drying solid, respectively
$A_u$, $A_m$ = nondrying surface and average area of the drying solid, respectively

A thermal resistance at the junction of the drying solid and the tray material and an effect of radiation to the tray can be added to the terms of Eq. (12.16) if desired.

Combining Eqs. (12.1) and (12.10) to (12.15) permits calculation of the rate of drying,

$$N_c = \frac{q}{\lambda_s} = \frac{(h_c + U_k)(T_G - T_s) + h_R(T_R - T_s)}{\lambda_s} = k_Y(Y_s - Y) \quad (12.17)$$

The surface temperature must be known in order to use the relationship. It can be obtained by consideration of the left-hand portions of Eq. (12.17), which can be rearranged to read

$$\frac{(Y_s - Y)\lambda_s}{h_c/k_Y} = \left(1 + \frac{U_k}{h_c}\right)(T_G - T_s) + \frac{h_R}{h_c}(T_R - T_s) \quad (12.18)$$



# Classification of dryers

- Based on **solid handling** (static bed dryer-moving bed-fluidized bed).
- Based on **heat transfer** (direct dryers-Indirect-IR or radiant heat).

**Dryers for Dilute Solutions and Suspensions**

- The objective of these **dryers** is to **spread the liquid** to a large **surface area for heat** and mass **transfer** for collecting the **dry solid.**

- **Two main types are used:**
- The first, **spreading** the liquid to a thin film.
- The second, dispersing the liquid **to a spray** of small droplets.

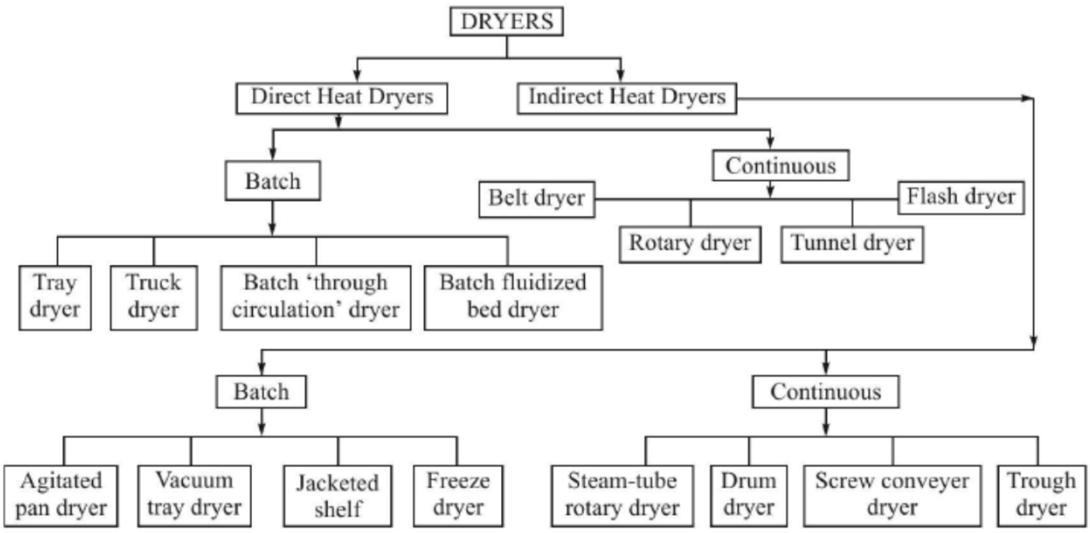


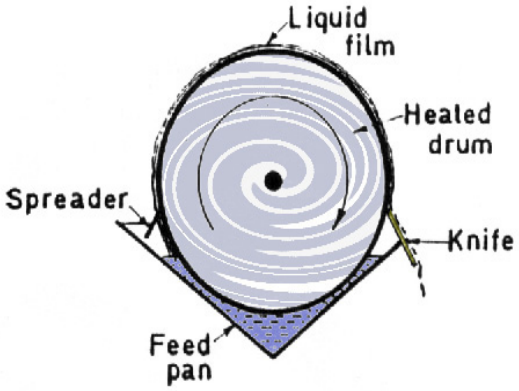

**Figure 11.8** Classification of common dryers.



# Drum Dryer (Film Drying)

- It consists of a drum of about 0, 75-1.5 m in diameter and 2-4 m in length, **heated internally**, usually by **steam**, and **rotated** on its longitudinal axis.

- **Operation:** The li**quid** is applied to the surface and **spread** to a film, this may be done in various ways, but the simplest method ***is*** that shown in the diagram, where the **drum dips** into ***a feed pan***. Drying rate is controlled by using a suitable speed of **rotation** and the **drum temperature**. The product is **scraped** from the surface of the drum by means of a **doctor knife.**



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## Advantages of the drum dryer

1- The method gives rapid drying, the thin film spread over a large area resulting in rapid heat and mass transfer.

2- The equipment is compact, occupying much less space than other dryers.

3- Heating time is short, being only a few seconds.

4- The drum can be enclosed in a vacuum jacket, enabling the temperature of drying to be reduced.

5- The product is obtained in flake form, which is convenient for many purposes.

- **The only disadvantage** : is that operating conditions are critical and it is necessary to introduce careful control on feed rate, film thickness, speed of drum rotation and drum temperature.

- **Uses:** 1- It can handle a variety of materials, either as solutions or as suspensions e.g. starch products, ferrous salts and suspensions of kaolin.

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## Spray Dryer

The spray dryer provides a large surface area for heat and mass transfer by atomizing the liquid to small droplets. These are sprayed into a stream of hot air, so that each droplet dries to a solid particle.

- The drying chamber resembles the cyclone ensuring good circulation of air, to facilitate heat and mass transfer, and that dried particles are separated by the centrifugal action.....

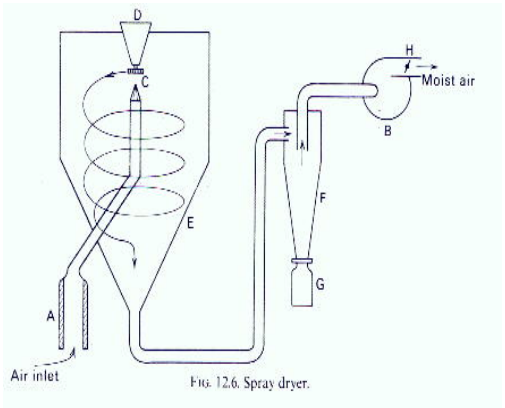

Fig. 12.6. Spray dryer.

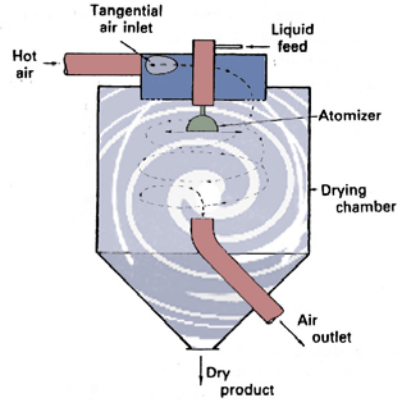

Fig. 38.13 Spray drier

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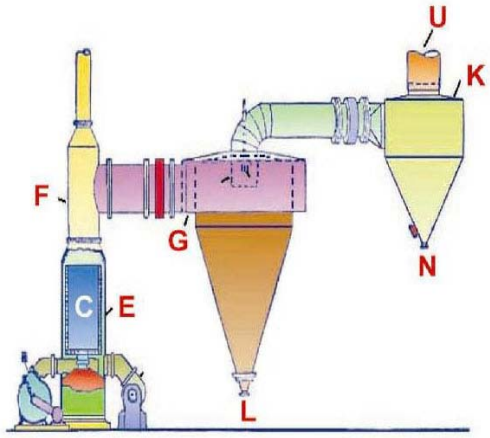


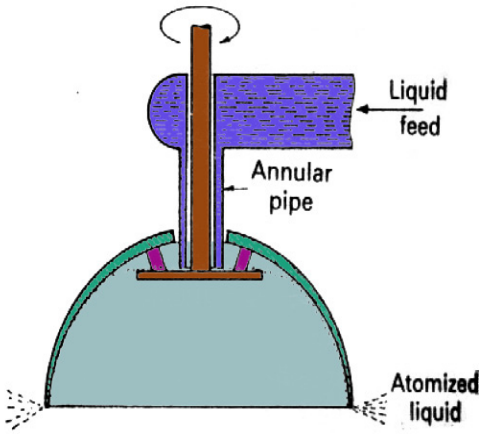


A, control panal; B, coating champer; C, PARTICLIS being treaated;
D, process air flow; E, air distribution plate;
F, nozzle for appling film coatings.

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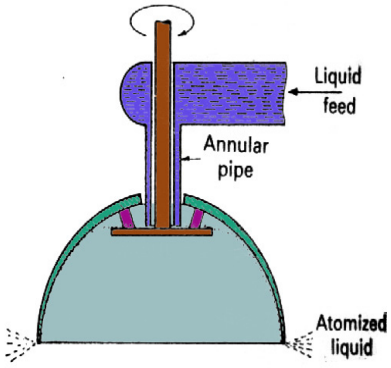
- The character of the particles is controlled by the **droplet form**; hence the type of **atomizer** is im**p**ortant.
- Rotary atomizer is preferable than jet which is easily blocked. Liquid is fed to the disc of the atomizer which is rotated at high speed (up to 20,000 rpm), a film is formed and spread as uniform spray. In addition, the rotary atomizer is effective with suspensions. It can be operated efficiently at various feed rates.




**Rotary atomizer**

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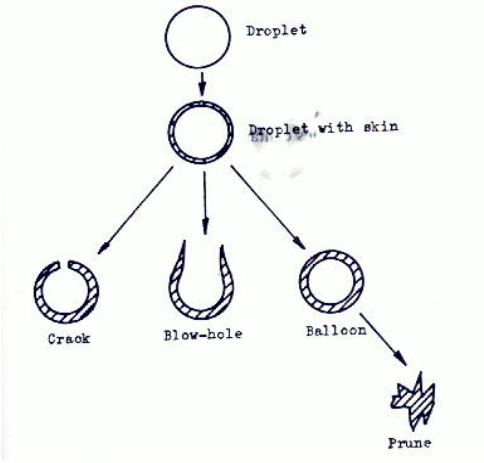
## Characterization of spray dried products ;

The products are uniform in appearance and have characteristic shape, in the form of hollow spheres with a small hole. This arises from the drying process, since the droplet enters the hot air stream, and dries on the outside to form an outer crust with liquid still in the center. This liquid then vaporizes, the vapour escaping by blowing a hole in the sphere.

- This method of drying allows a dry product to retain some properties of feed , e.g., a drop from an emulsion dries with continuous phase on the outside. When reconstituted, the emulsion is easily re- formed.

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## Advantages of the spray drying process

1- The droplets are small, giving a **large surface area** for heat transfer, so that **evaporation** is very **rapid**. The actual **drying time** of a droplet is only a **fraction of a second**, and the **overall time** in the dryer is only a few **seconds**.

2- Because **evaporation** is very **rapid,** the droplets **do not** attain a **high** temperature, most of the **heat** being used as **latent heat** of vaporization.

3- The characteristic **particle** form gives the product a **high bulk density** and, in turn, **ready solubility.**

**4- T**he powder will have a uniform and controllable particle size.

5- The product is free-flowing, with almost spherical particles, and is especially convenient for tablet manufacture.

6- Labour costs are low, the process yielding a dry, free-flowing powder from a dilute solution, in a single operation with no handling.

7- It is possible to operate spray driers aseptically using **heated filtered air** to dry products such **as serum** hydrolysate.

8- Some spray driers **operate in a closed-circuit** mode with **an inert gas** to **minimize oxidation** of the product. **Volatile solvents** can be recovered from such systems.



## Disadvantages & Uses

The equipment is very bulky, connected to accessories, fans, heaters,)

That is make it expensive.

**Uses**:

1- Drying of any substance in **solution** or in **suspension** form.

2- It is most useful for drying of **thermolabile materials e.g. antibiotics.**

3- Suitable for **large quantities** solution.

4- Suitable for both **soluble** and **insoluble** substances e.g. citric acid, gelatin, starch.

5- It can produce spherical particles in the **respiratory range** e.g. dry powder **inhalers.**

6- Drying of **milk, soap and detergents** which is pharmaceutically related compounds.

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## Freeze Drying

- Freeze drying is a process **used to dry extremely heat – sensitive** materials**.** It allows the drying , without excessive damage, of **proteins, blood products** and even microorganisms, which retain a small but significant viability.
- In this process the initial liquid solution or suspension is frozen, the pressure above the frozen state is reduced and the water removed by sublimation.
- Thus a liquid –to-vapour transition takes place, but here three states of matter involved: liquid to solid, then solid to vapour

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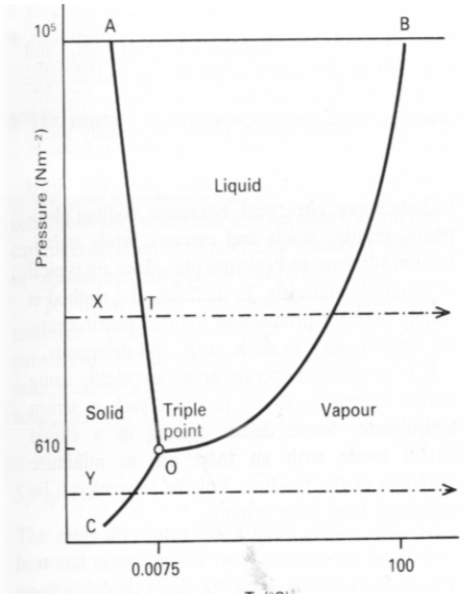
## The phase diagram for water



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## The phase diagram for water

1. The diagram consists of three separate areas representing the phases of water, solid, liquid, and vapour .
2. The point O is the only point where all the three phases can coexist, and is known as **the triple point .**
3. On heating at constant **atmospheric pressure** ice will melt when the temperature rises to 0 C . At this constant temperature and pressure it will then change to water. Continued heating will raise the temperature of the water to 100 C where, if heat addition is continued, the liquid water will be converted into water vapour at 100 C.
4. If , however, solid ice is maintained at a pressure below the triple point then on heating the ice will sublime and pass directly to water vapour without passing through the liquid phase.
5. This sublimation, and therefore drying, can occur at a temperature below 0 C.
6. This will only happen if the pressure is prevented from rising above the triple point pressure .
7. It may be thought that as the process takes place at a low temperature the heat required to sublime the ice will be **small.**

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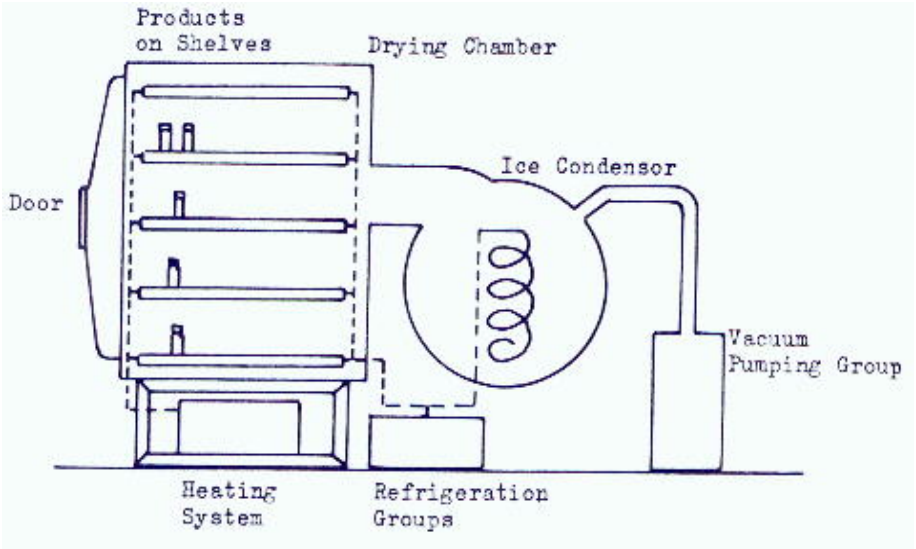
## Freeze Dryer



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## Limitations of the freeze drying process.

The freeze drying of products such **as blood plasma,** although simple in theory, presents a number of practical problems:-

1- The depression of the **freezing point** caused by the presence of **dissolved solutes** means that the solution must be **cooled below** the normal freezing temperature for pure water (-10-30).

2- Sublimation can only occur at the **frozen surface** and is **slow** process (1mm thickness of ice per hour). So, the **surface area** must therefore be **increased** and

3- the **liquid thickness** prior to freezing be **reduced** in order to reduce the thickness of ice to be **sublimated.**

4- At low pressure **large** volumes of **water vapour** are produced which must be removed to **prevent** the **pressure rising** above the **triple point** pressure.

5- The dry material often needs to be **sterile,** and it must also be **prevented** from **regaining** moisture prior to the final packaging.

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## Stages of the freeze drying process

***1- Freezing stage*:**

The liquid material is frozen before the application of vacuum to avoid frothing, and several methods are used to produce a large frozen surface.

a- Shell freezing : This is employed for large **volumes** such as blood products. The **bottles** are **rotated** slowly and **almost horizontally** in a **refrigerated bath**. The liquid freezes in a **thin shell** around the inner surface of the bottle.

Freezing is **slow** and **large ice** crystals form, which is a **drawback** of this method.

In **vertical** spin **freezing the bottles** are spun individually in a **vertical position ,** centrifuged and **cooled** by a blast of cold air. The solution super cools and freezes rapidly, with the formation of **small ice** crystals.

b- Centrifugal evaporative freezing: The solution is spun in small containers within a centrifuge. This prevents the foaming when a vacuum is applied. The vacuum causes boiling at room temperature. About 20% of the water is removed prior to freeze drying and there is no need for refrigeration. Ampoules are usually frozen in this way

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***2 - Vacuum application stage:*** The containers and the frozen material must be connected to a vacuum source sufficient to drop the **pressure below the triple point** and remove the larger volumes of low – pressure vapour formed during drying.

***3 - Sublimation stage:***

Heat of sublimation must be supplied. Under these conditions the ice slowly sublimes, leaving a porous solid which still contains about 0.5% moisture after primary drying .

**Primary drying**: It can reduce the moisture content of a freeze-dried solid to around 0.5%. Further reduction can be affected by secondary drying .

**Heat transfer:** Insufficient heat input prolongs the process, which is already slow, and excess heat will cause melting.

**Vapour removal:** The vapour formed must be continually removed to avoid a pressure rise that would stop sublimation.

**Rate of drying**: The rate of drying in freeze drying is very slow, the ice being removed at a rate of about only 1mm depth per hour.

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## Advantages of freeze drying

***4- Secondary drying:***

The removal of residual moisture at the end of primary drying is performed by raising the temperature of the solid to as high as 50 or 60 C .

***5- Packaging:***

Attention must be paid to packaging freeze-dried products to ensure protection from moisture. Containers should be closed without contacting the atmosphere.

**Advantages of freeze drying**

1- Drying takes place at very low temperatures, so the chemical decomposition, particularly hydrolysis is minimized.

2- The solution is frozen occupying the same volume as the original solution, thus , the product is light and porous.

3- The porous form of the product gives ready solubility.

4- There is no concentration of solution prior to drying. Hence, salts do not concentrate and denature proteins, as occurs with other drying methods.

5- As the process takes place under high vacuum there is little contact with air, and oxidation is minimized.

## Disadvantages & Uses of freeze drying

**Disadvantages:**

-There are two main disadvantages:

1-The porosity, ready solubility and complete dryness yield a very hygroscopic product. Unless products are dried in their final container and sealed in situ, packing require special conditions.

2-The process is very slow and uses complicated plant, which is very expensive .It is not a general method of drying but limited to certain types of valuable products.

**Uses of freeze drying**

The method is used for products that can not be dried by any other heat method. These include biological products, e.g. antibiotics, blood products, vaccines, enzyme preparations and microbiological cultures.



## Dryers for Solid Materials

- **Moisture content of wet solids**
- The moisture content of a wet solid is expressed as kilograms of moisture associated with 1 kg of the moisture – free solid. Thus a moisture content of 0.4 means that 0.4 kg of removable water is present per kg of the solid. It is sometimes calculated as percentage moisture content.
- Total moisture content: This is the total amount of liquid associated with a wet solid. The easily removable water is known as the **free moisture content**, and the moisture which is more difficult to remove is the **equilibrium moisture content.** The easily removable water is known as **unbound water**.

- *Unbound water:* This water exists as a liquid and exerts its fully vapour pressure, it can be removed readily by evaporation. During a drying process this water is easily lost but the resulting solid is not completely free from water molecules.

- Equilibrium moisture content:
- The moisture content present in a solid under steady-state ambient conditions is termed the eq. moisture content. Its value changes with temperature, humidity and the nature of the solid.
- *Bound water :*
- Part of the moisture present in a wet solid may be adsorbed on surfaces of the solid or be adsorbed within its structure to such an extent to prevent it from developing its full vapour pressure and from being easily removed by evaporation. Such moisture is described as "bound" and is more difficult to remove than unbound water.



## Relative humidity (RH) of air

- Air at a given temperature is capable of taking up water vapour until it is saturated (at 100% RH ). If the **temperature is raised** then the air will be able to take up **more** moisture and the **relative humidity falls**.
- The RH of air is dependent not only on the amount of moisture in the air , but also on its temperature, as the amount of water required to saturate air is itself dependent on temperature.
- It should be noted that in convective drying, where warm air is passed over the surface of a wet solid, the relative humidity may rise during the drying process as a result of **two separate factors:-**
- 1- Uptake of evaporated water vapour from the wet solid,
- 2- The cooling of the supply air as it transfers heat to the wet solid (evaporative cooling).
- If the cooling is **excessive** the temperature of the air may fall to a value known as the **dew point,** when liquid water will condense and be deposited.

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## Relationship between equilibrium moisture content (EMC) and relative humidity

- The EMC of a solid exposed to moist air **varies** with the relative humidity (Fig 26.2, Aulton p.382). **Ordinary atmospheric conditions** are of the order of 20 C and 70-75 RH
- , so that if exposed to atmosphere a material such as **kaolin** will contain about **1%** moisture, whereas **a starch** –based product may have as much **as 30%** or more. Materials exposed to humid conditions will regain moisture, and so there **is no advantage** in drying to moisture content **lower than** that which the material will have under the conditions of use

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## Loss of water from wet solids

- Unbound water is easily lost by evaporation until the equilibrium moisture content of the solid is reached (Fig 26,3, Aul) , Once the solid reaches its EMC , extending the time of drying will not change the moisture content as an equilibrium situation has been reached. The only way **to reduce** the moisture content is to **reduce the RH** of the ambient air. This can be done mechanically with **an air-conditioning** system.
- On small scale, **desiccators** are used.

- **..... Fig 26.2 .....Fig.26.3.....(Aul**

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## Different techniques of drying of solids ( Drying methods)

- The following points should be considered before the **selection of the suitable** drying method:

- 1- Heat sensitivity the material being dried.
- 2- Physical characteristics of the material.

- 3- Nature of the liquid to be removed.
- 4- The scale of the operation.
- 5- Available **sources** of heat (steam, electrical).

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## The general principles for efficient drying can be summarized as follows

- 1- Large surface area for heat transfer.

- 2- Efficient heat transfer per unit area (to supply sufficient latent heat of vaporization or heat of sublimation in case of freeze-drying)

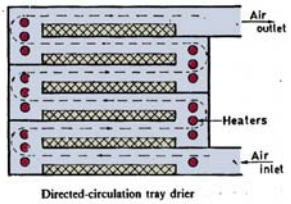
- 3- Efficient mass transfer of evaporated water through any surrounding boundary layers, i.e. sufficient turbulence to minimize boundary layer thickness.

- 4- Efficient vapour removal , i.e. low relative humidity air at adequate velocity.

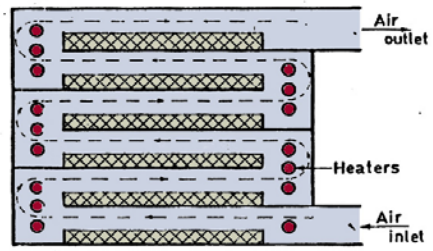
- It is convenient to categorize pharmaceutical driers according to the heat transfer method they use, i.e. convective, conductive or radiant.

## Convective Drying of Wet Solids

- **I-Fixed (or static) bed convective drying e.g. tray drier**
- **Tray drier:**



Directed-circulation tray drier



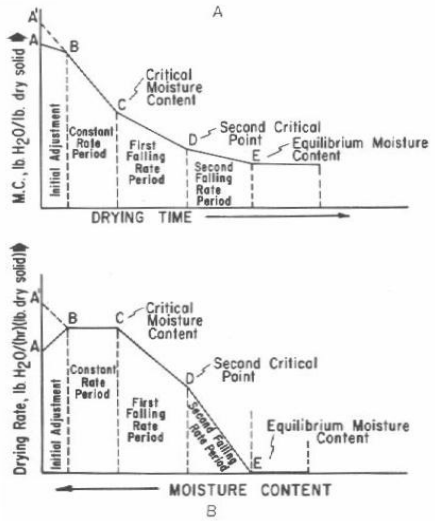
Directed-circulation tray drier

- In Fig.26.4. tray drier. Air flows in direction of the arrows over each shelf in turn. The wet material is spread on shallow trays resting on the shelves. Electrical elements or steam-heated pipes are positioned as shown, so that the air is periodically reheated after it has cooled by passage over the wet material on one shelf before it passes on the next.
- .**Fig.26..4.....Directed- circulation tray drier**.,Aul,p.383.



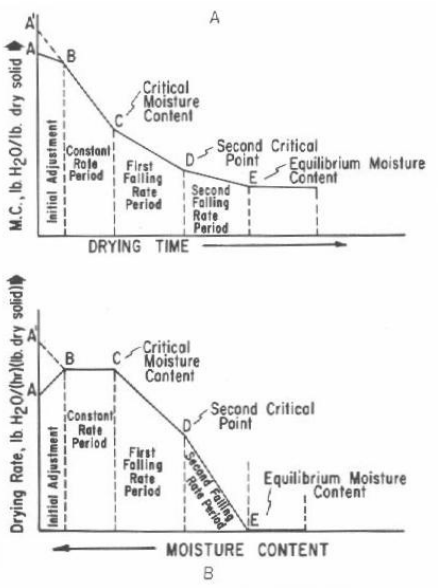
## Rate of drying in a bed of powder:

- The rate at which drying occurs has been found to show certain phases (fig.26.5, Aul) in which the change in moisture content is plotted against time. From A to B the relationship is linear, which is known as **the constant-rate period,** whereas from B to C the rate of loss of moisture decreases and is known as **the falling-rate period.** The end of the constant rate period, B,is referred to as **the critical moisture content.**



- The **first falling- rate period** has a linear relationship, that is , the decrease in drying rate is uniform, whereas in **the second falling-rate period** there is a continuous decrease in the rate of drying until the EMC is reached.

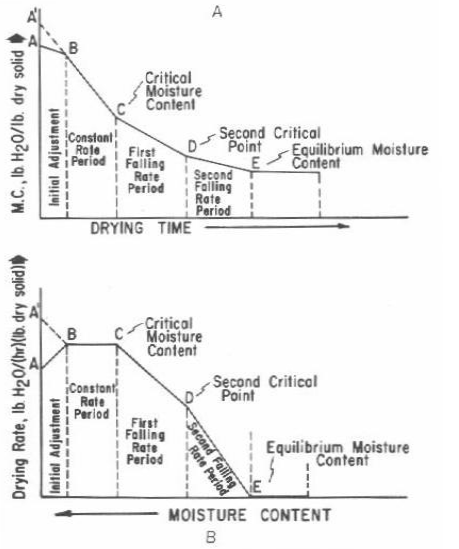
Each of these periods will be considered in more details.



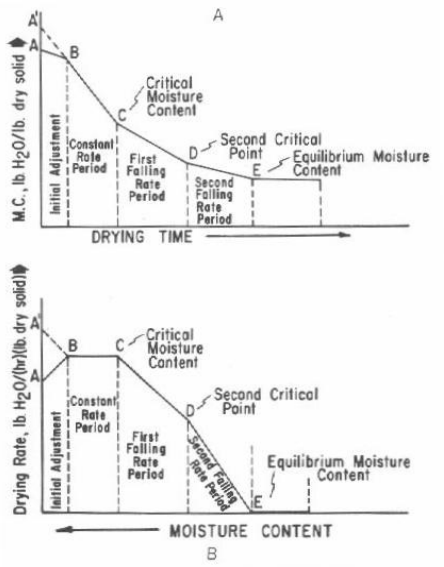



Constant-rate period: It is found that the evaporation rate from the drying bed is similar to that of the solvent alone from a free liquid surface under the same conditions, indicating that the evaporation takes place from the wet surface of the solid, and that the surface remains wet in this period as a result of the liquid being replaced from below as fast as it is vaporized

Controlling factors in this period are the rate at which heat can be transferred and the rate of removal of the vapour.

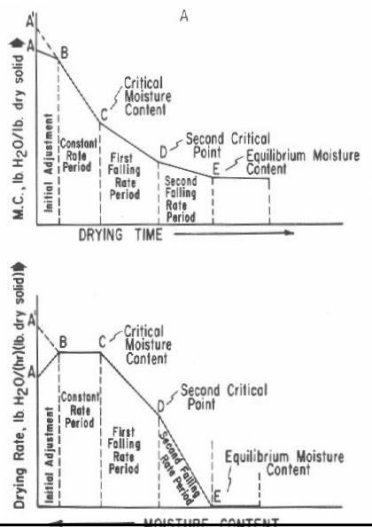


- First falling-rate period: As moisture is removed from the surface, a point will be reached when the **rate of vaporization is insufficient** to saturate the air in contact with the surface. Under these conditions, **the rate of drying** will be limited by the rate of **capillary transfer** of the liquid to the **surface** of the **wet bed**, and this becomes increasingly difficult as the bed dries, the **solvent level decreases** and thus has further to travel to the point of **evaporation**. Consequently, the **rate** of drying **decreases** continuously.






- Second falling-rate period: **A**ny moisture that remains within the drying bed at the end of the first falling –rate period is unable to move, so that drying cannot take place on the surface. i.e. the drying rate depends on the movement of the vapour through the pores of the bed to the surface, in general by molecular diffusion



- Minimal atmospheric humidity above the solid , in addition to the thermal conductivity of the solid decreases as it becomes dry, if the solid is thermostable it is safe to allow temperature gradients to increase to maintain the rate of heat transfer, but if the material is thermolabile the heating must be decreased.
- In the operation of a tray drier it is usual to remove the dry material on the trays near the air inlet and replace them with the trays with partially dry material from further away.

- .**Fig 26.5 Drying curve..,** Critical moisture content (**CMC),** Equilibrium moisture content ( **EMC)**...Ault, P.384.



## II- Dynamic convective driers

Example, fluidized – bed drier.

- Good contact between the warm drying air and wet particles is found in the fluidized – bed drier.
- Principles of fluidization. The particulate matter is contained in a vessel, of which is perforated, enabling a fluid to pass through the bed of solids from below.
- If the air velocity through the bed is increased gradually and the pressure drop through the bed is measured, a graph of the operation shows several distinct regions, as indicated in **the fig.**
- **Fig.26.6..Effect of air velocity on pressure drop through a fluidized bed.....**

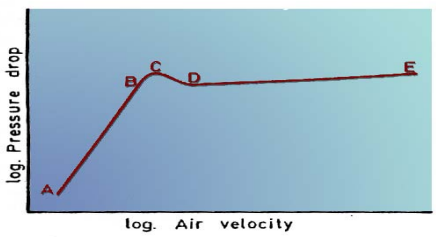
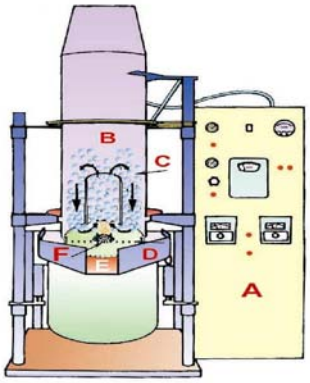
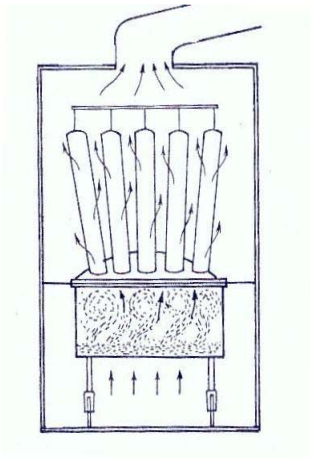


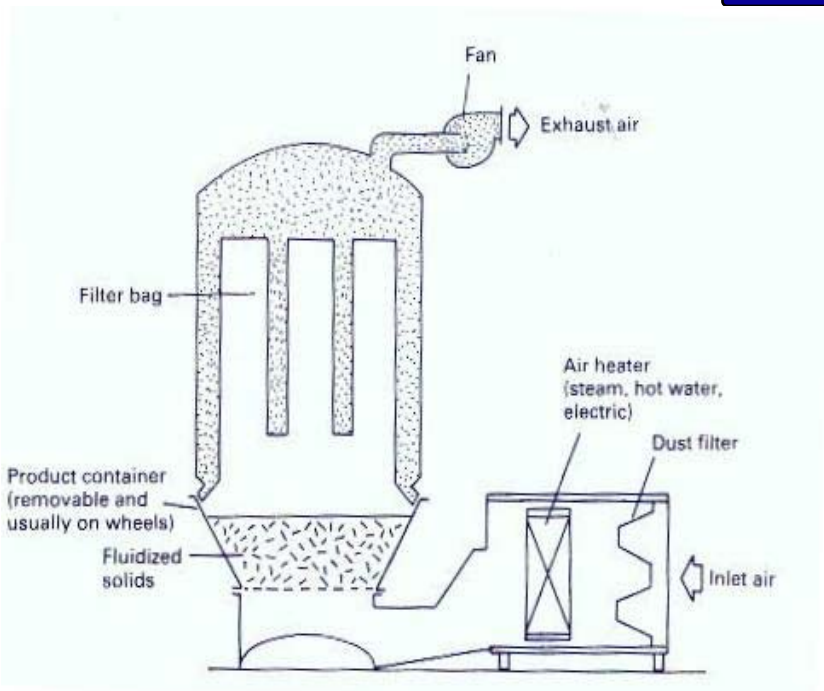

**Fig. 38.7** Effect of air velocity on pressure drop through fluidized bed

# Wurster Air Dryer







- At **first,** when the air velocity is **low, (point A) ,** flow takes place between the particles **without** casing disturbance, but as the **velocity is increased** e **point (B**), is reached (**gravity = force of fluidization** on that particle). Rearrangement of the particles occurs to offer least **resistance (C),** and they are **suspended** in the air and can **move.**

- At **point (D),** pressure **drop** through the bed **decreases slightly** because of the greater **porosity.** Further increase in the air velocity causes the particles to separate and move freely and the bed is **fully fluidized.**

Any additional increase in velocity separates the particles further, that is, the expands without appreciable change in the pressure drop, until (**E),** when the air velocity is sufficient to transport the particles to the top of the bed.

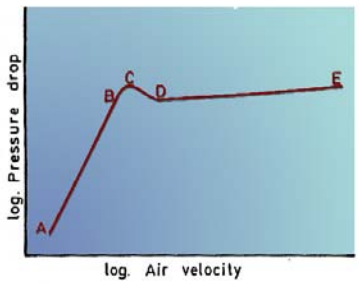



**Fig. 38.7** Effect of air velocity on pressure drop through fluidized bed

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- In the **region D---E** fluidization is irregular , much of the air flowing through in bubbles, the **term boiling bed** being commonly used to describe it .

- The important fact is that to produces conditions of great turbulence, the particles mixing with good contact between them and the air. Hence if hot air is used the **turbulent** conditions lead to **high heat** and **mass transfer** rates, the fluidized – bed technique therefore offers a mean of **rapid drying.**

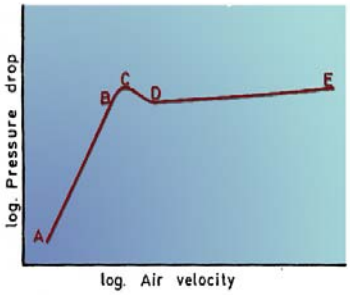


**Fig. 38.7** Effect of air velocity on pressure drop through fluidized bed

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## Advantages of fluidized-**bed** drying

- 1- Efficient heat and mass transfer give **high drying rates,** so that drying times are shorter than with static-bed convection driers. Economic, heat challenge to **thermolabile** materials is minimized.
- 2-The fluidized state of the bed ensures that drying occurs from the surface of all the individual particles and not just from the surface of the bed. Hence, most of the drying will be at constant rate and the falling –rate period is very short.
- 3-The temperature of a fluidized bed is uniform and can be controlled precisely.
- 3-The turbulence in a fluidized bed causes some attrition to the surface of the granule. This produces a more spherical free-flowing product.
- 5-The free movement of individual particles eliminates the risk of soluble materials migrating, as may occur in static beds.

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## Disadvantages of fluidized-bed drying

- 1-The turbulence of the fluidized state may cause excessive attrition of some materials, with damage to some granules and the production of too much dust.
- 2- Fine particles may become entrained in the fluidizing air and must be collected by bag filters, leading to segregation and loss of fines.
- 3-The vigorous movement of particles in hot dry air can lead to the generation of static electricity charges. The danger is increased if the fluidized material contains a volatile solvent such as isopropanol. Adequate electrical earthing is essential.

## Conductive Drying of Wet Solids

- Mechanism of heat transfer:
- **1- Conduction**:
- The transfer of heat from **one part** of a body to **another**, without appreciable displacement of the particle is **referred as conduction**. This mode of heat transfer is called **molecular heat transfer**, because it involves the transfer of kinetic energy from one molecule to the **one adjacent** to it, e.g. conduction of heat along the length of a **metal rod** when one end is heated.
- **2- Convection**:
- The transfer of heat **from one point to another** in a body of **fluid**, such as a liquid or a gas, by a mixing process, is referred to as **convection heat** transfer .In most cases convection involves the transfer of heat **from a solid** surface to the **bulk of the fluid,** the change in heat induce a **change in the density** of the **liquid** e.g. the conventional **currents** observed when water is heated in a glass beaker.



- **3- Radiation**:
- The transfer of heat by radiant energy in the form **of electromagnetic** waves, which **travel in straight** lines at the speed of light, is **referred to** as radiation heat transfer. As a body is heated, it emits radiant energy, e.g. sun and infrared heat lamps. When this radiation strikes another body, portions may be reflected

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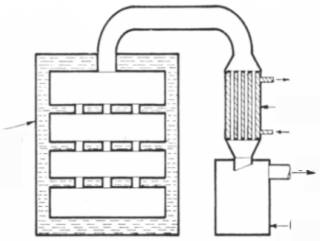
## Vacuum oven

- This equipment is a good example of **conduction drier**. The vacuum oven consists of a **jacketed vessel** to withstand **vacuum** within the oven. There are supports for the **shelves** giving a larger area for **conduction** heat transfer. The oven can be closed by a door. The oven is connected through a condenser and liquid receiver to a vacuum pump.
  Operating **pressure** can be as **low as 0.03-0.03 bar**, at which **pressures** water boils **at 25-35 C.**

- Advantages of vacuum oven:
- Drying takes place at a low temperature.
- There is little air present, so, there is minimum risk of oxidation.



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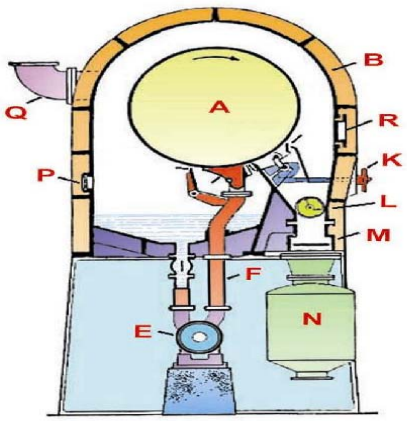
## VACUM DRUM DRYER




A, drying roll; B, casing; C, trunnions; D, rotary joint; E, feed pump;
F, feed-inletpipe; G, drumfeeder; H, spreader; J, doctor knife;
K, doctor-knife-adjusting handwheels; L, product conveyor;
M, product-receiver shut-off valve; N, product receivers;
P, sight glasse; Q, vapor outlet; R, manhole

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## Radiation Drying of Wet Solids

- **Radiant heat transmission:**

- Heat transmission by **radiation** differs from heat transfer by c**onduction** or **convection** in that **no transfer medium** (solid, liquid or gaseous) need be present. Heat energy in the form of radiation can **cross empty space** or **travel** through the atmosphere virtually without **loss**. If it falls on a body capable of absorbing it then **it appears as heat,** although a proportion may be reflected or transmitted.

- **Use of infrared radiation :**

- Infrared heating has been used to **dry wet granules**, but it suffers from the **disadvantage** that it is **absorbed very quickly** and **does not penetrate** far into the **wet mass**.
- The **surface** layers dry quickly and the **absorption** of further energy then **raises** the temperature of the **dry material** to a high value. For this reason infrared radiation is now seldom used as a heat source in pharmaceutical manufacture.

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## The use of microwave radiation

- Although energy in the **IR region** is more easily generated there are other, **longer,** wavelengths that can generate heat when the radiation is absorbed by a wet solid. Microwave radiation in the wavelength range 10 mm to 1m penetrates much better than IR radiation. Microwave driers are used now in pharmaceutical industry.

- Generation and action of microwaves

- Microwaves are **produced** by an electronic **device** known as a **magnetron.** Microwave energy can be reflected down through **a window** into a drying **chambe**r.
- The **penetration** of microwaves into the **wet product** is so good that **heat** is generated **uniformly** within the solid.

- When microwaves **fall** on substances of **small polar** molecules such as **water**, the **electrons** in the molecule **try to resonate** in sympathy with the **radiation** and the resulting molecular "**Friction"** results in the **generation of heat**. Dry solids **do not** resonate as well as water, so **further heating** may be avoided once the water is removed.

- The **absorption** of the microwave energy is far **greater** for **small polar** molecules than for **larger and less polar** molecules (methanol-**ethanol-water** are **larger than starch** and **lactos**e which is **larger and less polar** molecules). This is indicated by the value of the loss factors of each substance. The **loss factor** is a measure of the ratio of the microwave **energy absorbed** by individual molecules, the **higher the number** the **greater** the absorption of microwave energy**.**



## A microwave drier for granulates

- Figure 26.9 (Aul, p.388) is a sketch of **microwave drier.** It is designed to operate under a slight **vacuum.** This in itself is **not essential** for the use of microwaves, but the air flow allows the **continuous removal** of evaporated **solvent**. The **radiation is generated** by multiple **magnetrons.** The radiation passes through a **window** into the **drying chamber,** where it is **absorbed by the liquid** in the **wet granules** contained on a tray. The **heat** generated in the mass **drives off the moisture** and the evolved **vapour** is **drawn away** in the **air** flow as it is **formed.** When d**rying** is nearly **complete** the **radiation field** intensity will **rise**, as the dry solids **do not absorb** as readily as water. This rise is detected and the magnetrons are progressively turned off automatically, to give an accurate control of the final moisture content and minimize the danger of over-heating.

**Fig.26.9 Microwave drier**

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## Advantages of microwave drying:

- 1- It provides rapid drying at low temperature.
- 2- The thermal efficiency is high, as the drier casing and the air remain cool. Most of the microwave energy is absorbed by the liquid in the wet material.
- 3- The bed is stationary, avoiding the problems of dust and attrition.
- 4- Solute migration is reduced as there is uniform heating of the wet mass.
- 5- Equipment is highly efficient; all the requirements of product and operator safety follow the GMP considerations.
- 6- Granulation end-point can be detected by measuring the residual microwave energy, ( it is rises sharply when there is little solvent left to evaporate).

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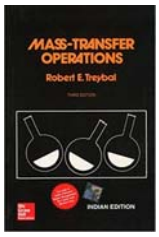
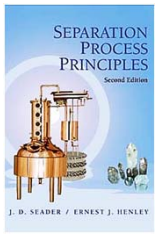
## Disadvantages of microwave drying:

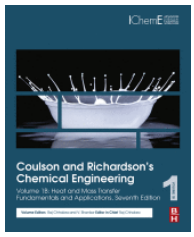
- 1- The batch size of microwave driers **is smaller** than those available for fluidized-bed driers.
- 2- Care must be taken to shield operators from the microwave radiation, which can cause damage to organs such as the eyes and testes. This is ensured by "**Failsafe"** devices preventing the generation of microwaves until the drying chamber is sealed.

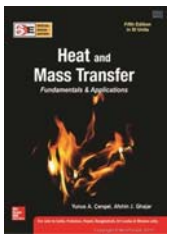
## References

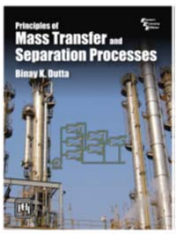


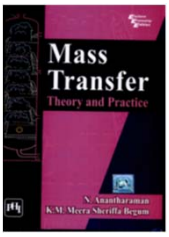





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Mass Transfer

Theories for Mass Transfer Coefficients

Lecture 9, 15.11.2017, Dr. K. Wegner

- Lecture notes/ppt of Dr. Yahya Banat (**ybanat@qu.edu.qa**)

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**MASS TRANSFER OPERATIONS: ABSORPTION AND EXTRACTION**

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