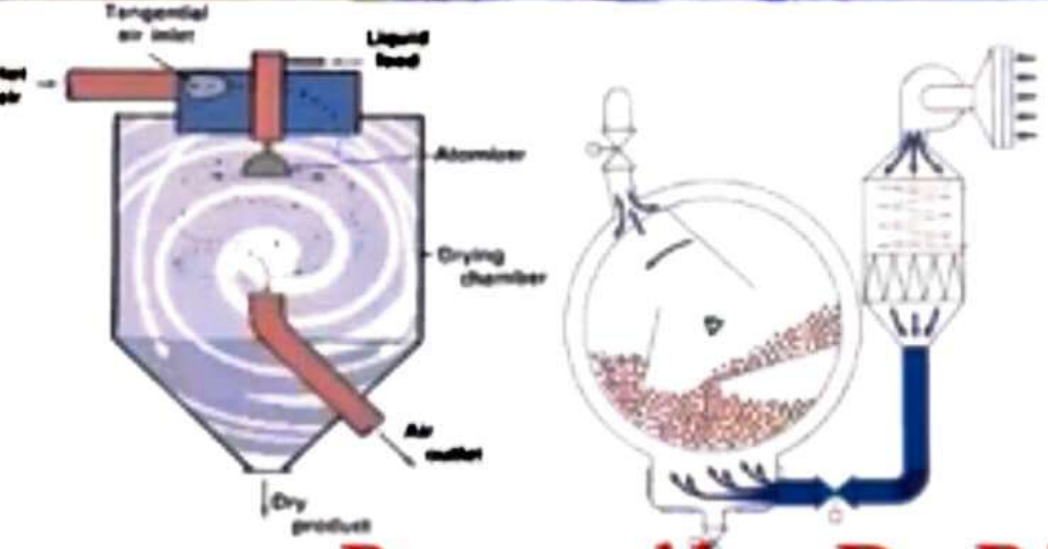
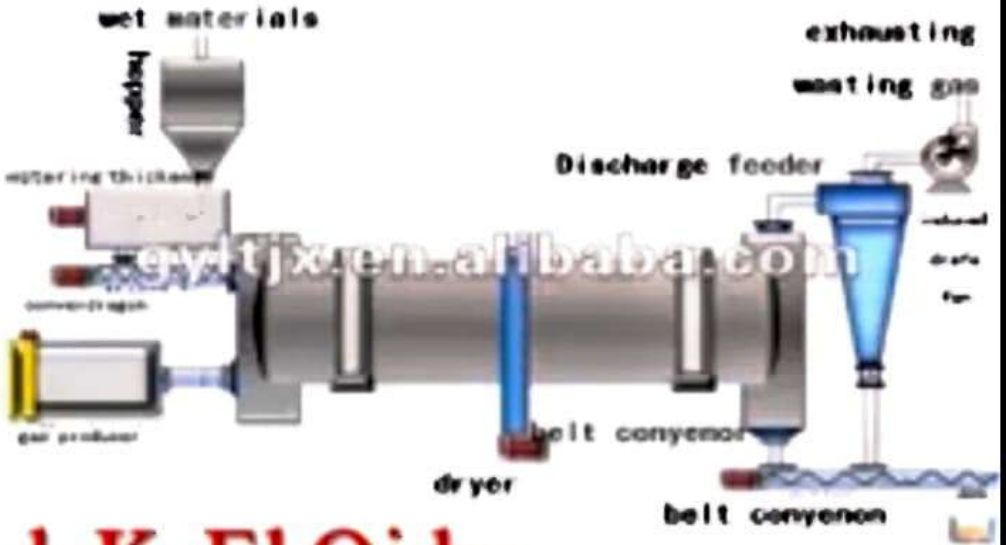
Drying





Prepared by: Dr. Riad K. El Qidra

Drying

- Most pharmaceutical materials are not completely free from moisture but contain some residual water, which may vary with the temperature and humidity.

- The Final Process in the manufacturing process is refers to as

  " the final removal of water"

  is known as **Drying**

Drying Process

- **Drying** is an operation in which a liquid usually water is removed from material (wet solid) in equipment termed **a dryer** **(usually by heat).**

- **Drying** is reduction in moisture content from an initial amount to acceptable form (EMC equilibrium moisture content).

  - **EMC:** If any material exposed to air it will either lose or gain water until equilibrium this point of equilibrium is called **EMC**.

    And it depends on the material nature (wool, leather, cotton or soap).

- The moisture content present in a solid under steady-state ambient conditions is termed the equilibrium moisture content

Applications and 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 stability**.
2. To **improve** or keep the **good properties** of a material, e.g. flowability, compressibility.
3. To **reduce** the cost **of transportation** of large **volume** materials (liquids).
4. To make the **material easy** or more suitable for **handling.**
5. Preservative.
6. **Drying makes materials more convenient in packaging,**
7. The **final step in**:

   Evaporation- Filtration- Crystallization.

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 **evaporation** 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**

Terminologies

Moisture

- **Total moisture content:** the total amount of liquid associated with a wet solid.
- In the context of drying, not all of it can be easily removed by the simple evaporative processes employed by most pharmaceutical driers.
- The easily removable water is known as the free moisture content (**unbound water**)

1- **Bounded moisture:** its vapor pressure less than that of free water and it is retained in small capillaries and adsorbed on the surface or as a solution in cell wall.

2- **Free moisture:** water in excess of EMC

Drying processes

Drying can be described by three processes operating simultaneously:

1- Energy transfer from an external source to the water or organic solvent

Direct or Indirect Heat Transfer

2- Phase transformation of water/solvent from a liquid-like state to a vapour state

Mass Transfer (solid characteristics)

3- Transfer vapour generated away from the API and out of the drying equipment

Factors affecting drying process:

1. Temp. ↑ → drying ↑.
2. S.A. of dryer.
3. Type of dryer. (static or moving)
4. Type of material. (porous or non porous)

The drying bed (dryer) is classified into:

1. **Static bed drying (dryer)**: the material to be dried is static while the hot air is moving above it.
2. **Moving bed drying (dryer)**: the material to be dried is moving inside the hot air so the drying occurs from all surfaces so it has fast drying rate.

✓ **Disadvantages of static bed:**

1. size reduction is needed by cutting.
2. conc. will differ from bottom to surface → water soluble substances will be at the surface.

✓ **Advantages of moving bed:**

1. ↑ rate.
2. each granule acts as a single bed.

✓ Examples of moving bed dryer:

1. FBD (fluidized bed dryer) : directly heated by hot air.
2. agitated dryer : indirectly heated by steam.

Types of solid Materials to be dried

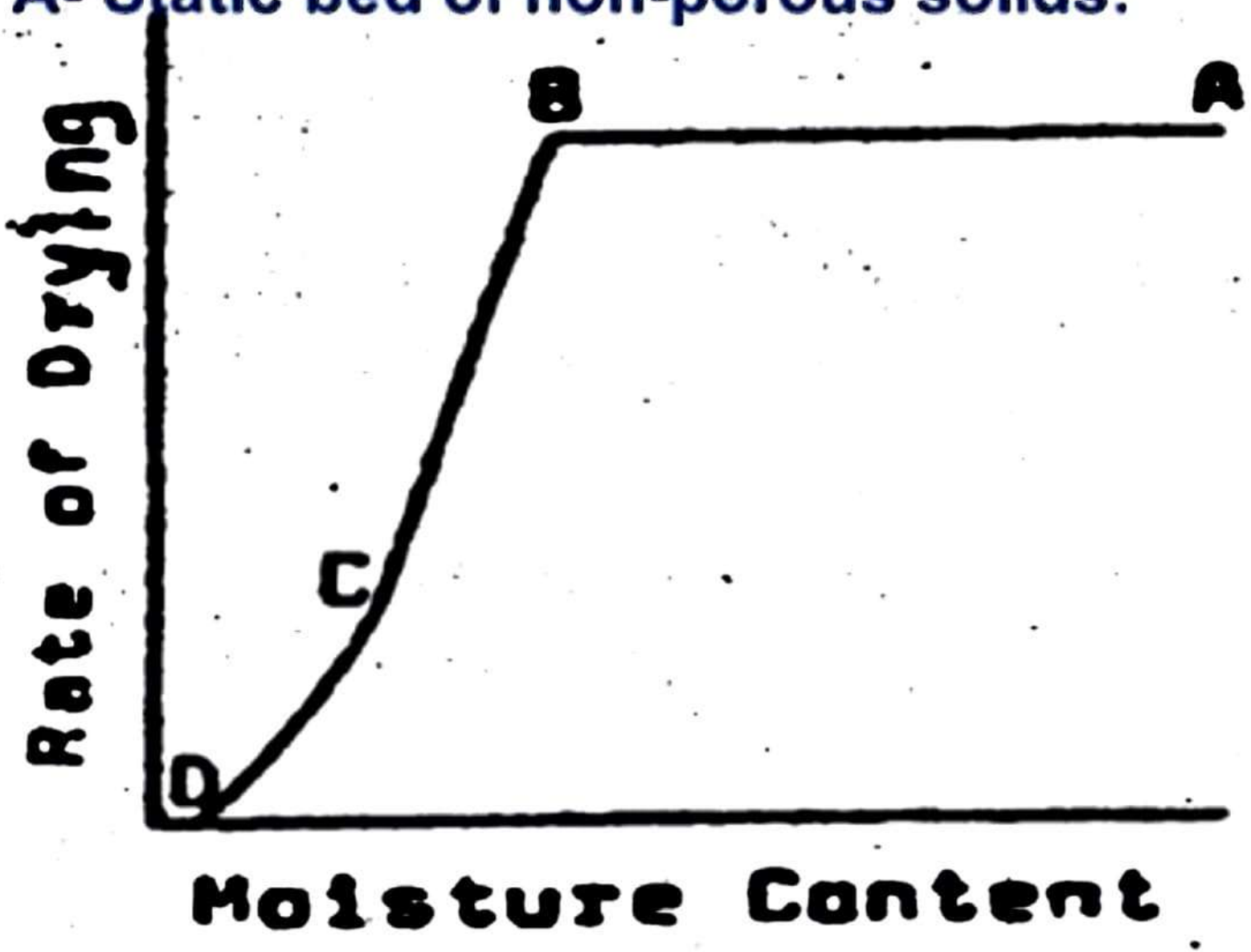
1- Porous

2- Non–porous skin forming materials
eg. (soap , gelatin).

3- Non porous

1- Rate of drying in static beds

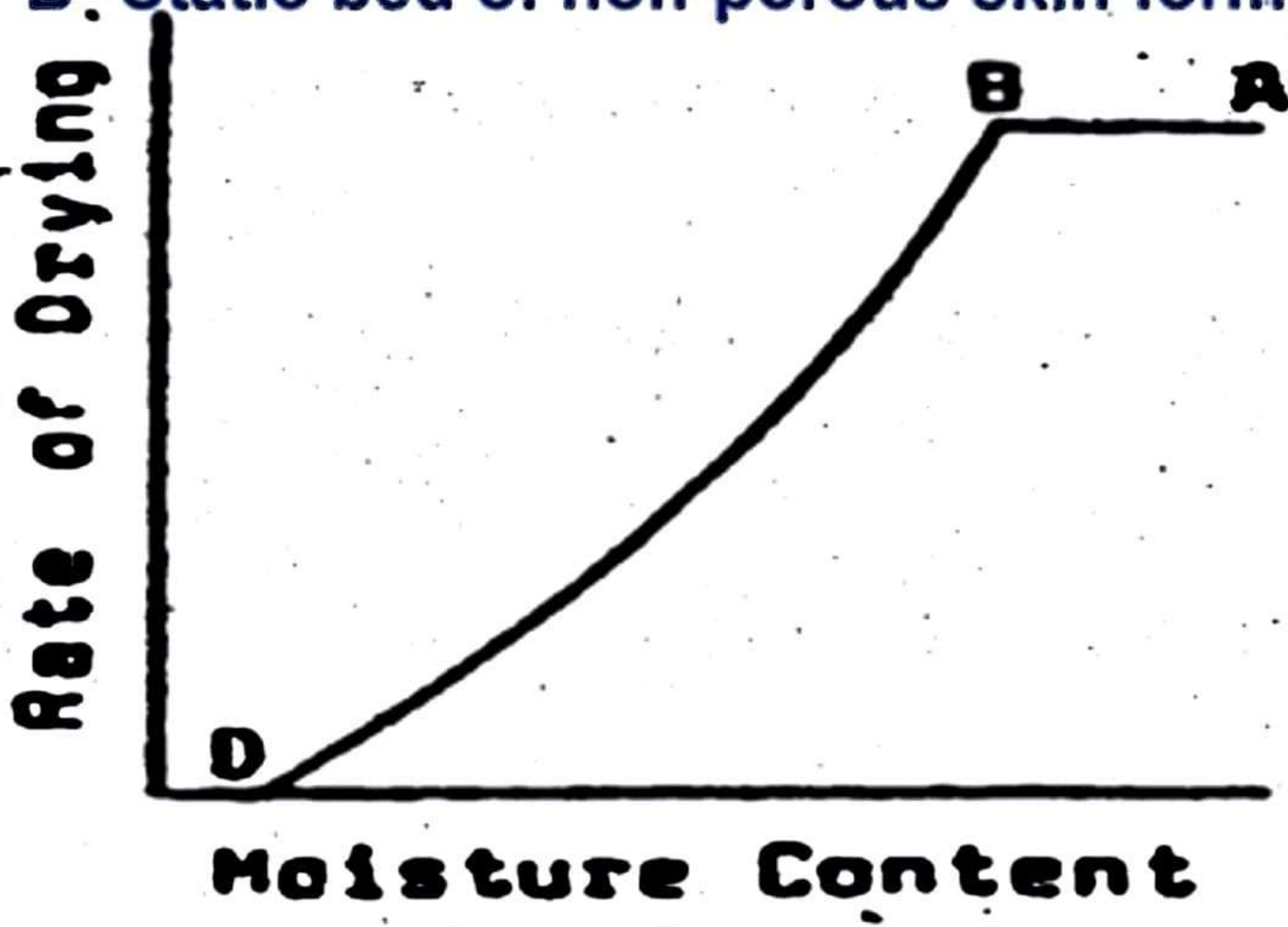
A- Static bed of non-porous solids:



typical non-porous solid

1- Rate of drying in static beds

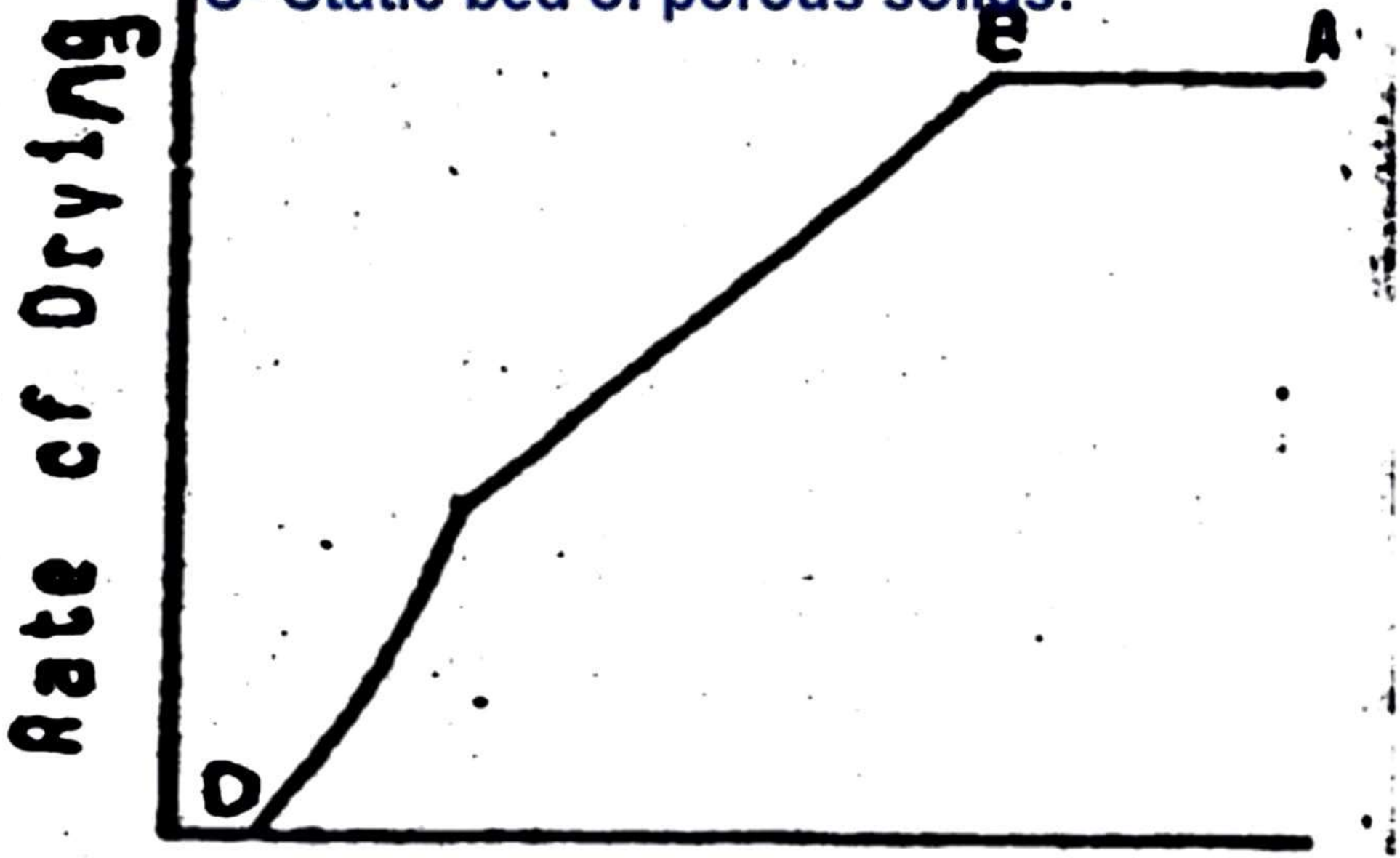
B- Static bed of non-porous skin forming solids:



b- skin forming material

1- Rate of drying in static beds

C- Static bed of porous solids:



porous materials

II – Rate of drying in Moving beds

- It is 10-20 times faster than static bed drying.
- Each particle behaves as a drying bed.

Choice of Method of Drying

- Product is sensitive to heat or not.
- Nature of solvent to be removed.
- Process is to be carried out under aseptic condition.
- Quantity of products to be dried.
- Available source of heat.
- Cost involved.

Dryers

Dryer is an equipment or machine that removes liquid usually water from material (wet solid) to obtain dry material (dry solid) by usually heating or another process

Types of Dryers

Batch Dryer	Continuous Dryer
1. Tray or Shelf Dryer	1. Turbo Shelf Dryer
2. Vacuum Tray Dryer	2. Drum Dryer
3. Tumbling Dryer	3. Spray Dryer
4. Freeze Dryer	4. Flash Dryer
5. Fluidized Bed Dryer	5. Rotary Dryer
6. I.R Dryer	6. Tunnel Dryer

I- Batch Dryers

I - Tray or shelf dryer (hot air oven)

- ❖ It operates by passing hot air over the surface of wet Solids in the trays.
- ❖ Thermal efficiency of air is improved by recirculating the air over the heater (Temperature / humidity changes):

1-The incoming air at point A is heated to B

2-When passes over the wet solids on tray1and 2 its temperature falls and the humidity increase to point C.

3- It then re circulated to the heater to raise its temperature to D and so on.

Advantages: simple, cheap.

Disadvantage:1- It requires large floor space

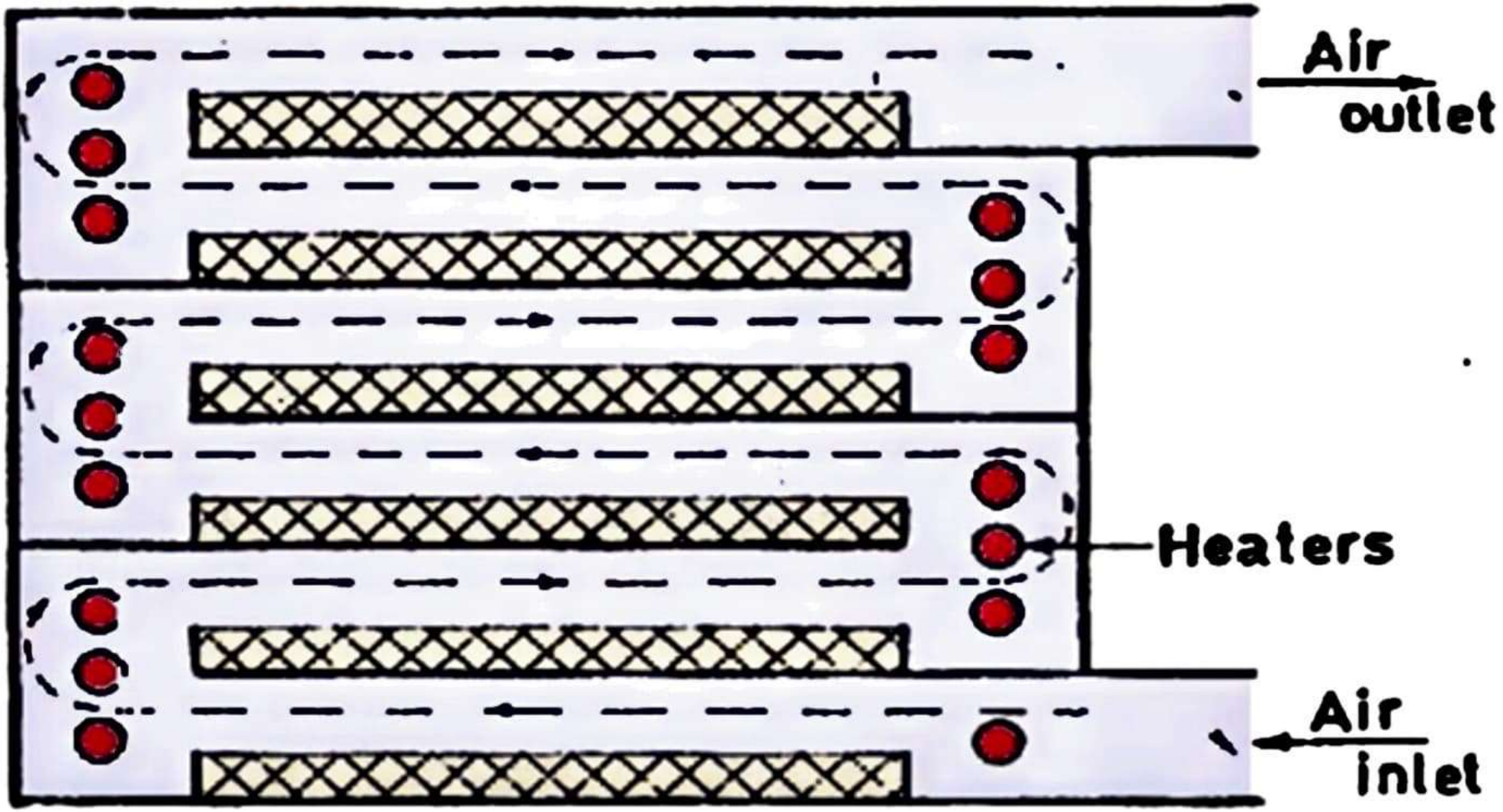
2-High labor cost (Batch)

3-Long drying time (24 hours)

4- Solvents can not be recovered from the dry air

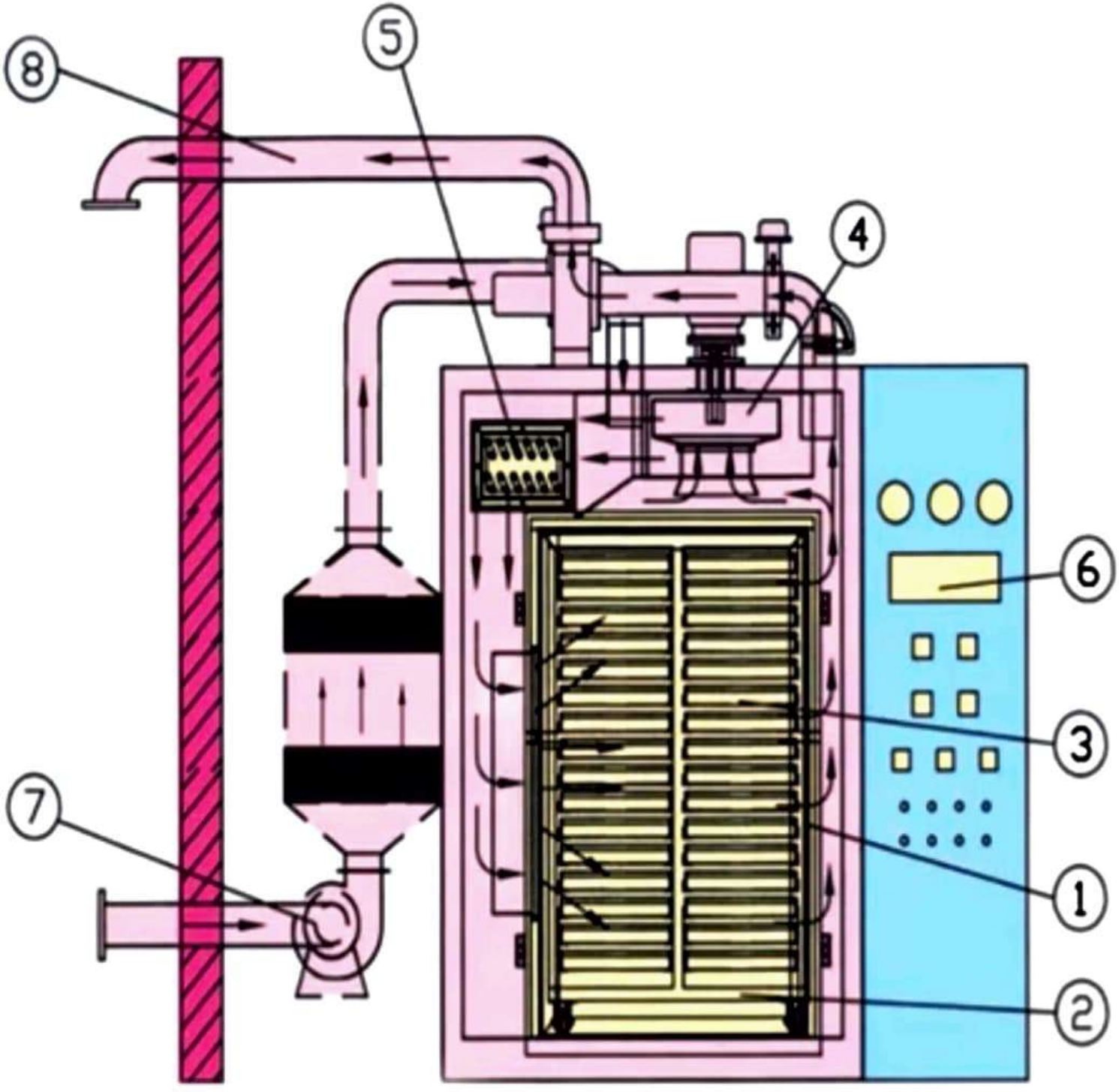
5-Variable temp. from location to anther $\pm$7 C

6-Used for thermostable only.

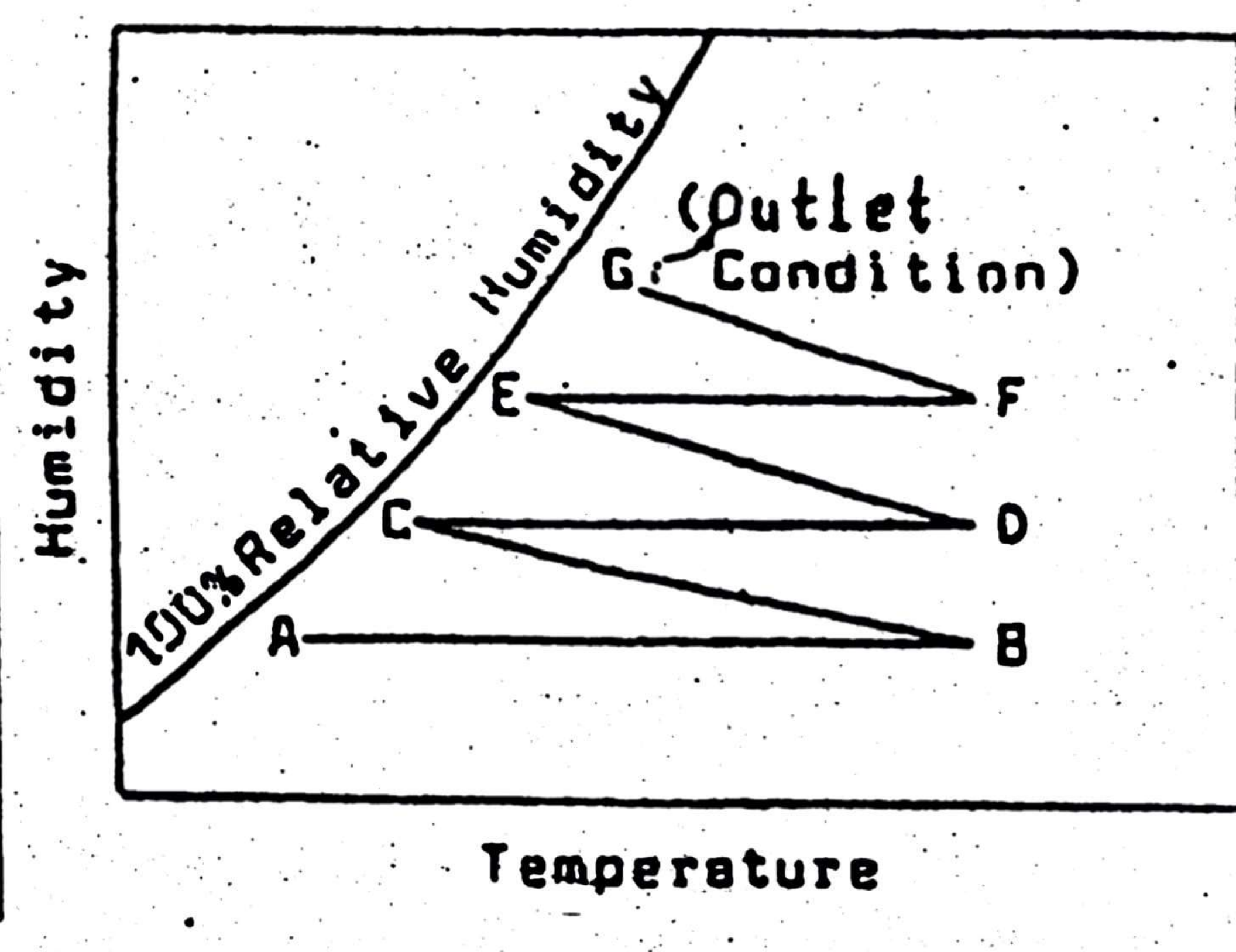


Directed-circulation tray drier

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.



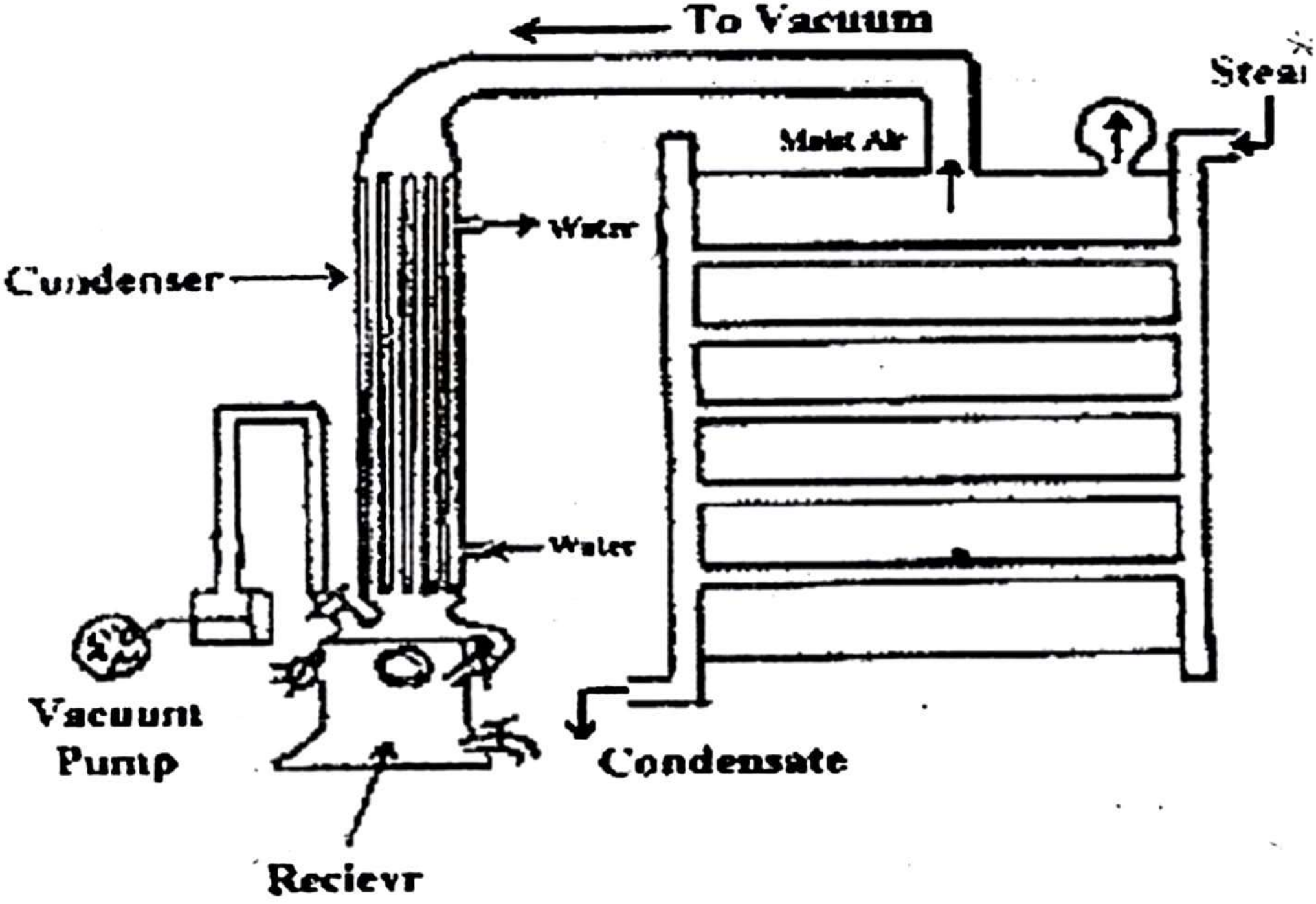
1. Drying Chamber
2. Drying Trolley
3. Drying Tray
4. Circulation Fan
5. Heat Exchanger
6. Control Panel
7. Fresh Air Inlet
8. Exhaust Damper


Humidity
100% Relative Humidity
(Outlet
G Condition)
E
F
C
D
A
B
Temperature

2- Vacuum Tray Dryer

- 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 0.03-0.04 bar**, at which **pressures** water boils **at 25-35 C.**
- The condenser is placed between oven and pump to
  1- to protect vacuum pump from condensate .
  2- to recover the solvent.

2- Vacuum Dryer



Advantages of vacuum oven:

1. Drying takes place at a low temperature.

2. There is little air present, so there is minimum risk of oxidation.

3. In addition, the low level of oxygen in the atmosphere diminishes oxidation reactions during drying. In general, colour, texture, and flavour of vacuum-dried products are improved compared with air-dried products

Vacuum Dryer

3-Tumbling Dryer

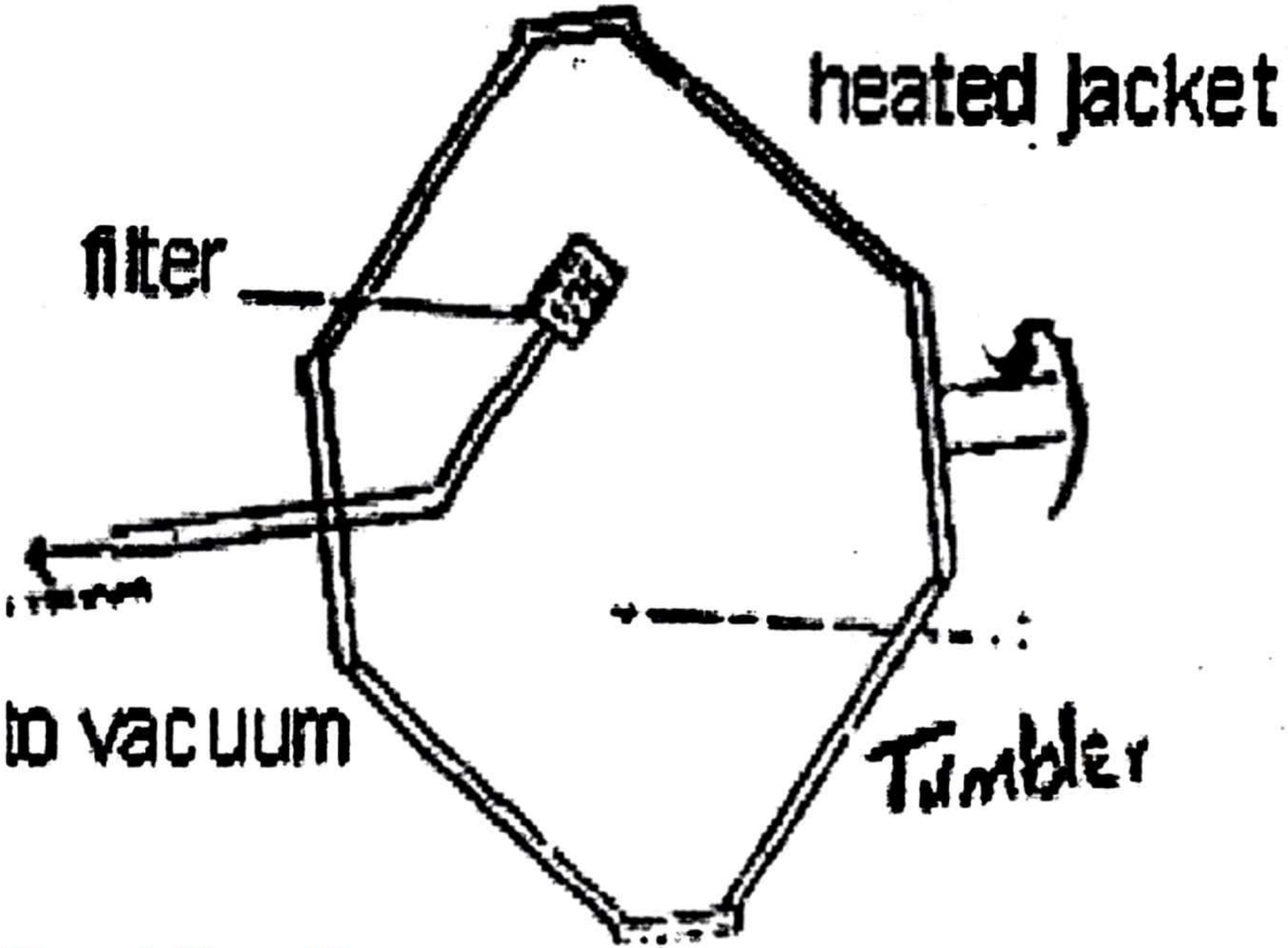
It consists of double cone with double jacket heated by steam working under reduced pressure at variable speeds.

Advantages

- ❖ It is a moving bed dryer so:
  1- It has short drying time 2-3 hours.
  2- The final product has granular shape
- ❖ normal charge is 60% of total volume
- ❖ can be used for mixing as well as drying **(MixoDryer)**

Disadvantage:

Not used for waxy materials due to aggregation



Tumbling Dryer

4- Fluidized Bed Dryer (moving Bed)

Good contact between the warm drying air and wet particles is found in the fluidized–bed drier.

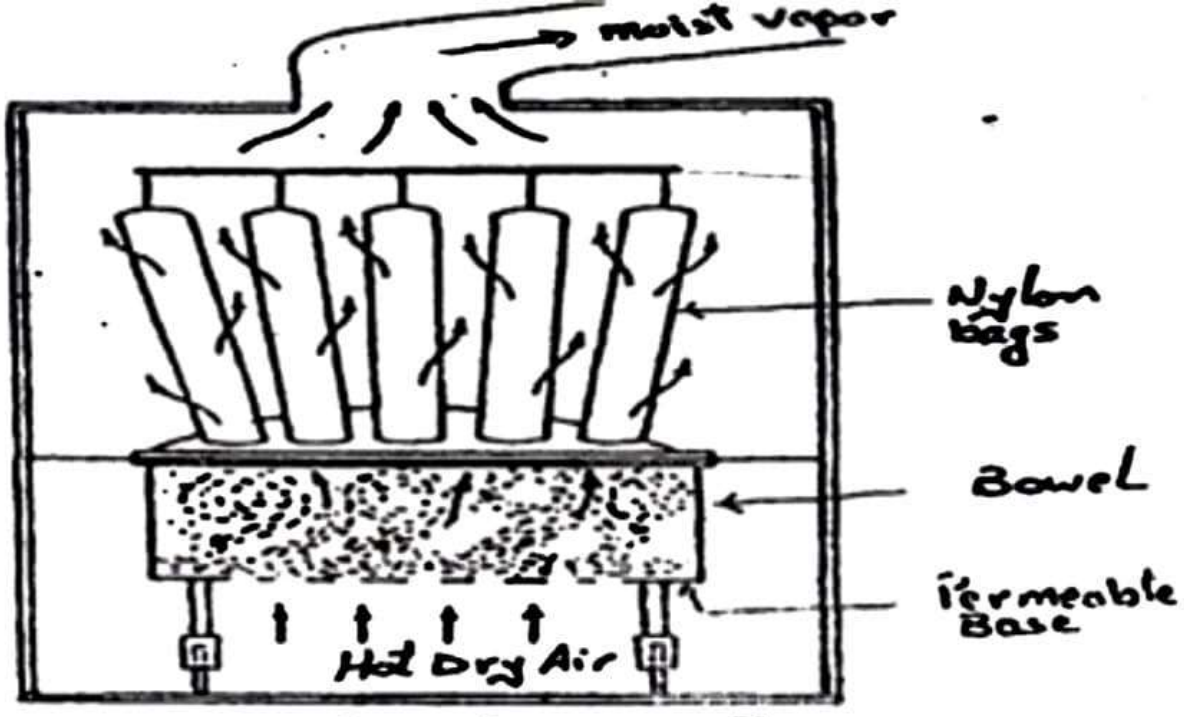
Principle:

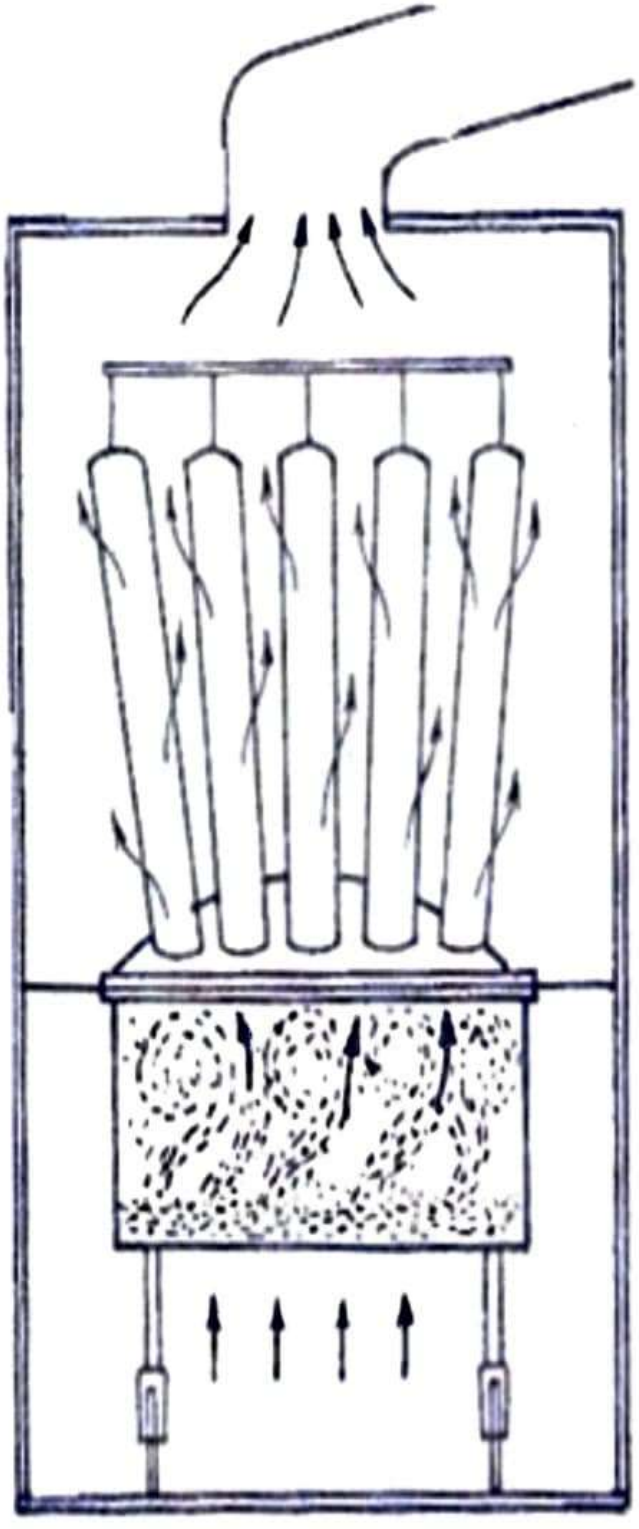
- If the hot air is allowed to flow upward at sufficient velocity through a bed of solid particles to render the solids suspended in the air.

- The resultant mixture behaves like a liquid and

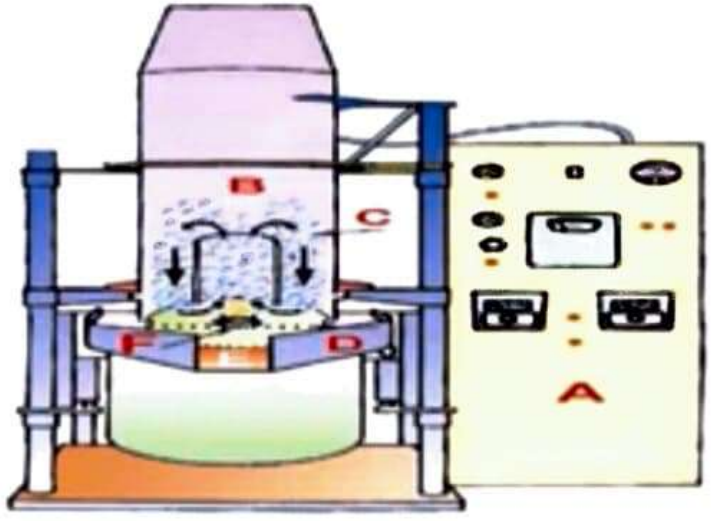
- The solids are said to be fluidized.

- It is very efficient for granular solids drying
- Not used for solids with high water content that they stick together on drying.





Wurster Air Dryer



Advantages:

1- Short drying time 20-40 min.

2- Less floor space

3- Uniform temperature through the oven

4- Produce free flowing particles improve (**flowability**)

5- Mixing may be done during dryer so we can add lubricant, disintegrate in tablet granulation **(MixoDryer).**

6-The free movement of the particles eliminates the risk soluble materials to surface layers as in case of tray.

- The final dried product is free flowing spherical in shape.

5- Freeze Dryer (Lyophilization)

- **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.

- A stabilizing process in which the initial liquid solution or suspension is frozen, the pressure above the frozen state is reduced and the water removed by sublimation.

- The word **lyophilized** is derived from the Greek "made solvent-loving".

Theory:

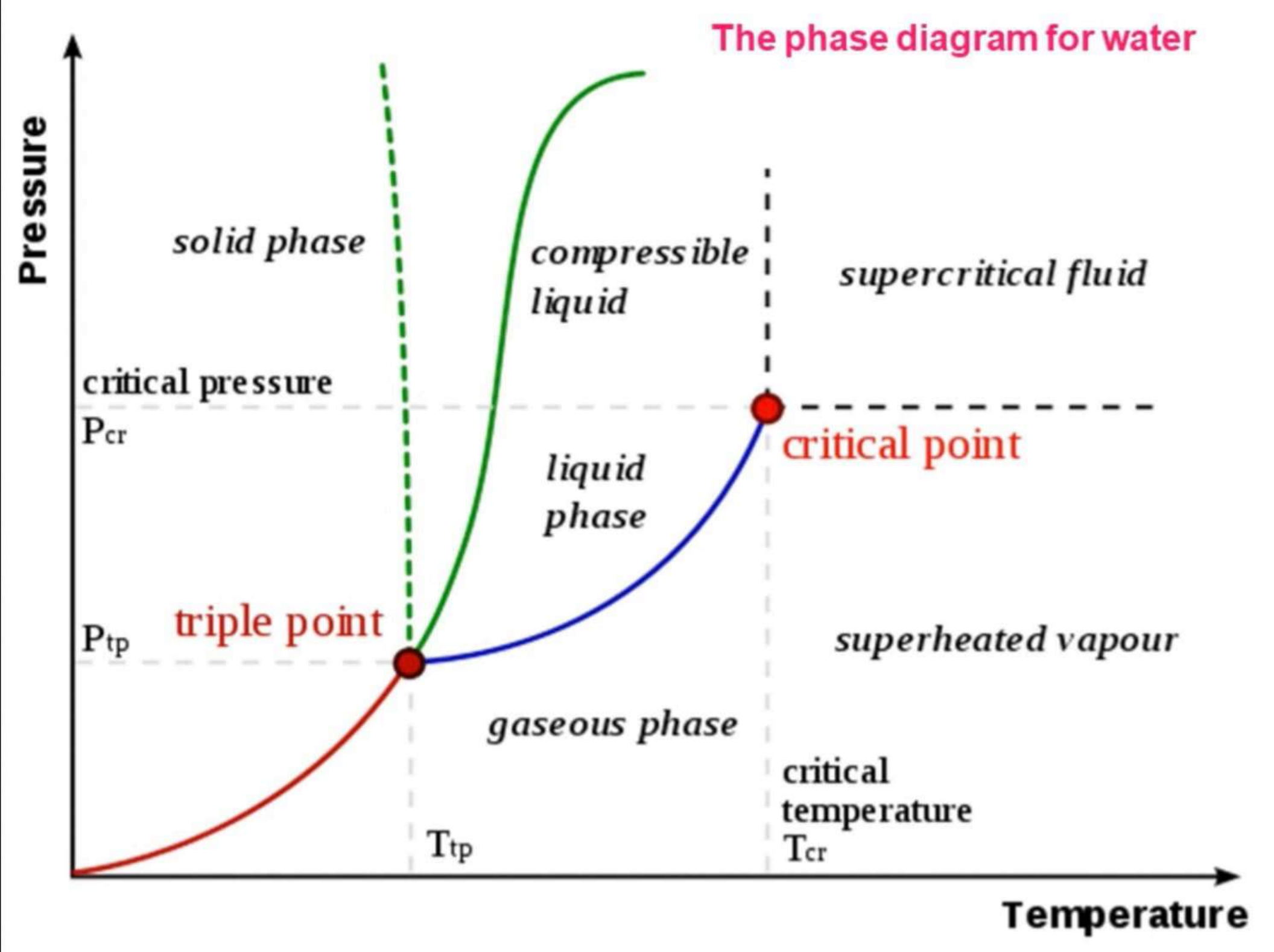
- when the external pressure around ice is less than its vapor pressure sublimation occurs.

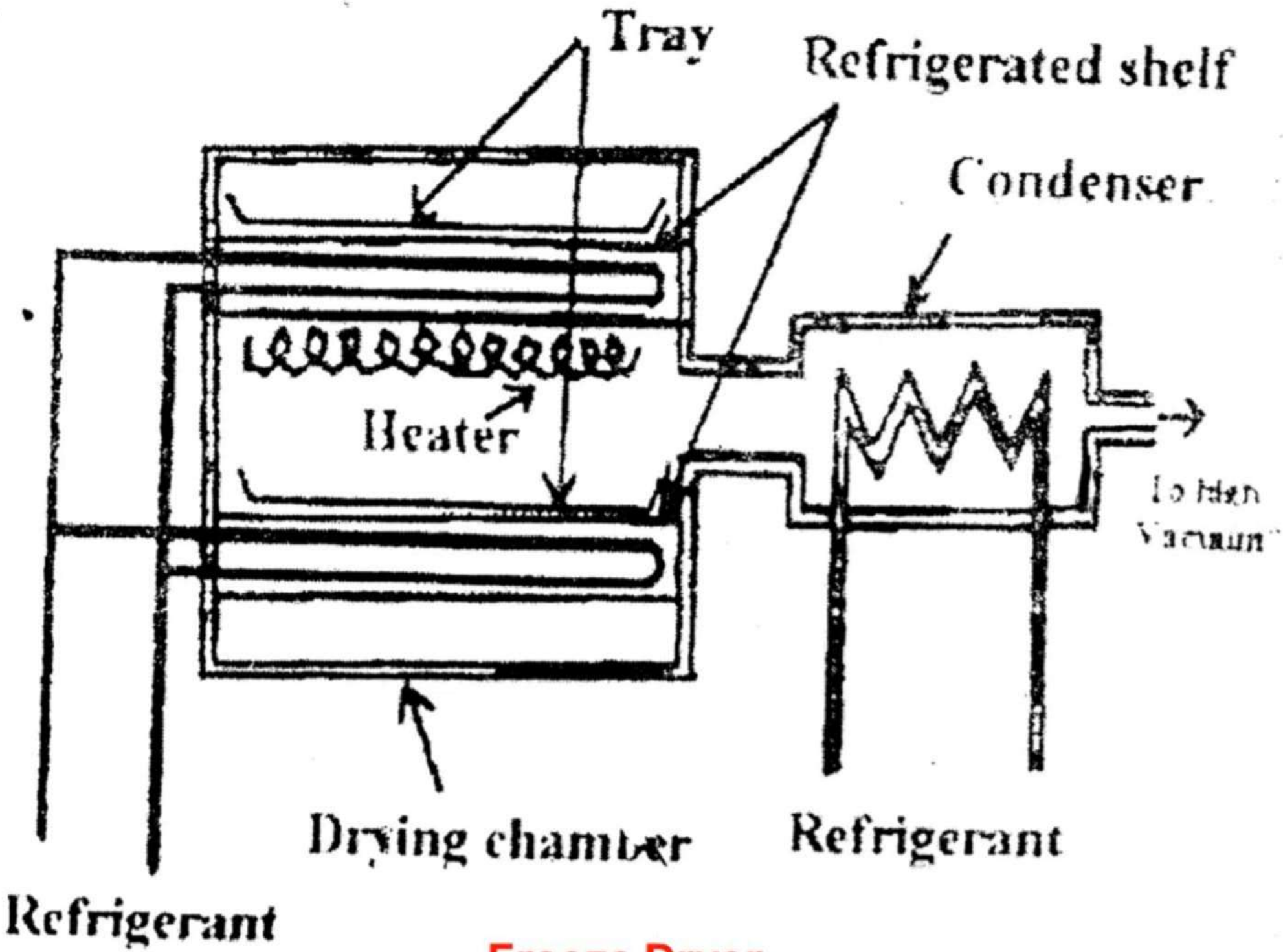
- i.e water is transferred from the solid form to gas form without melting.

- The most suitable working conditions for **F.D.** is

I. Temperature is **–10 C** at which vapor pressure is **0.3** mmHg.

II. By time temperature decreases to **–30 C** at vapor pressure **0.1** mmHg


The phase diagram for water
Pressure
solid phase
compressible liquid
supercritical fluid
critical pressure
Pcr
critical point
liquid phase
triple point
Ptp
superheated vapour
gaseous phase
critical temperature
Tcr
Ttp
Temperature



Freeze Dryer

Operation:

Thermolabile materials is placed over the trays and refrigerant decreases.

The vacuum is operated to reduce chamber pressure.

When the chamber pressure is less than ice vapor pressure sublimation occurs.

By time temperature is reduced to -30 and the vapor pressure decreased to 0.1 mmHg.

At this point heater is operated to

1- increase the temperature to -10^0C with pressure 0.3 mmHg

2- provide the ice with the required latent of sublimation

{

The **final dried** product is: sponge like residue and can be dissolved rapidly.

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.

Freeze Dryer

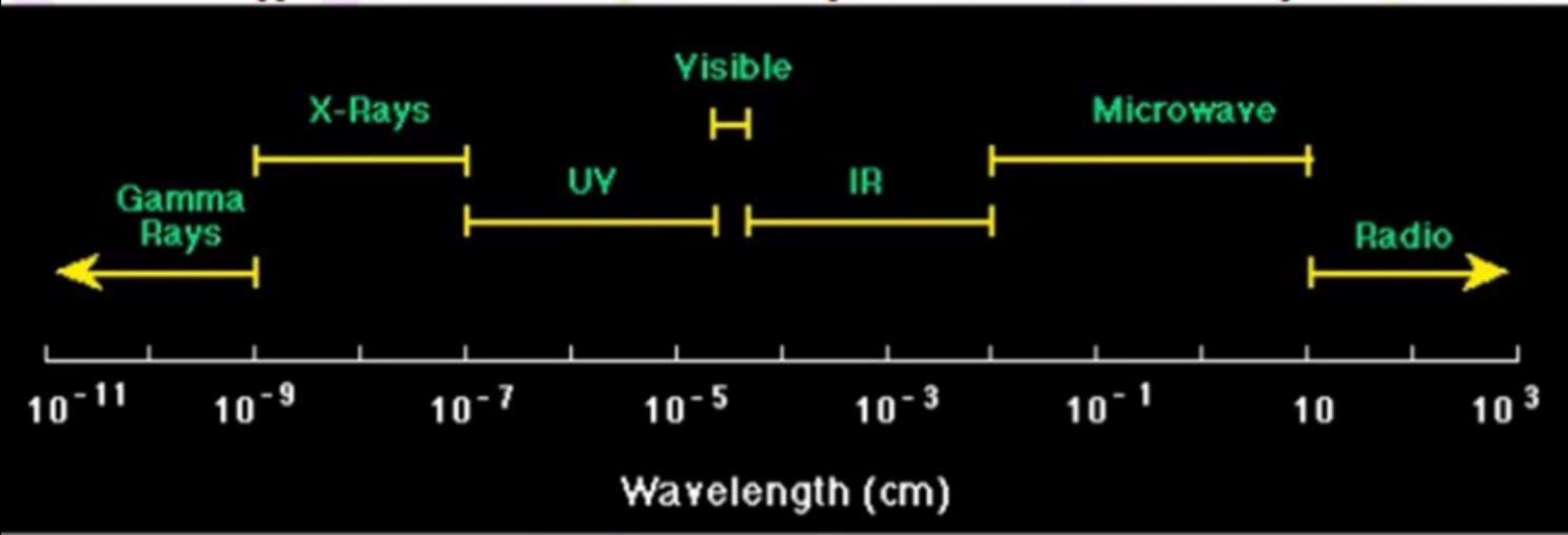
6- Infra-Red Dryer

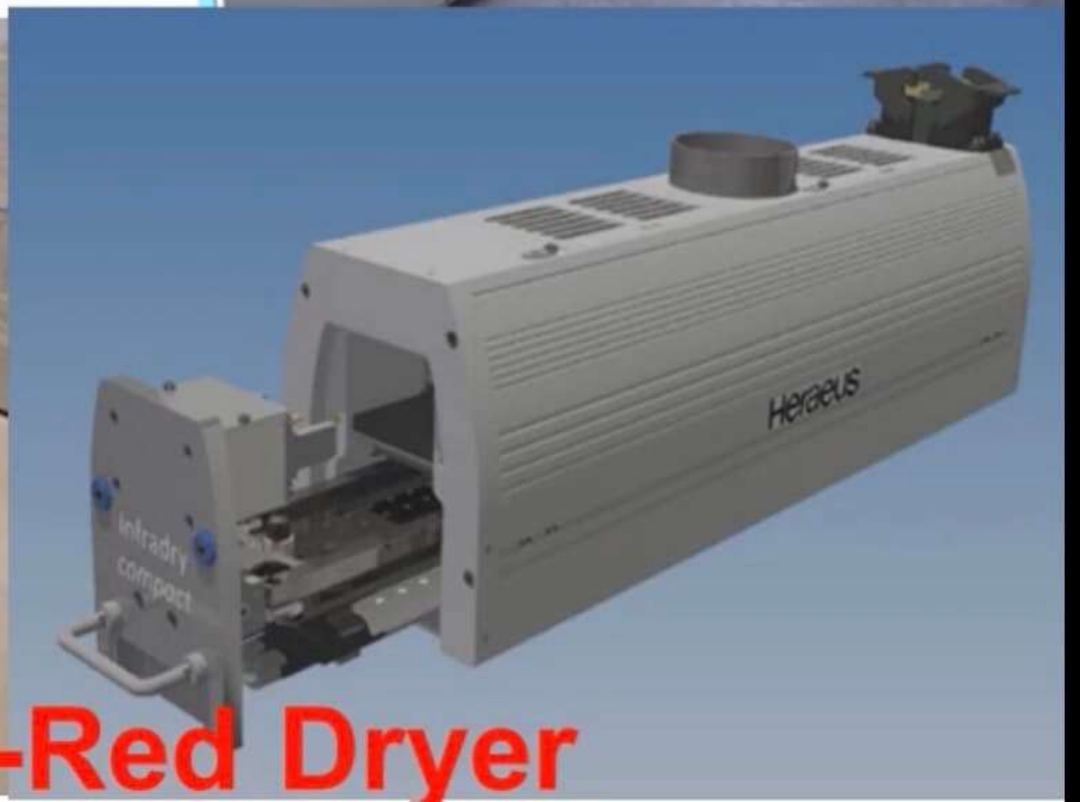
- Infrared (IR) dryer uses the infrared radiations, and
- Infrared radiations are invisible electromagnetic radiation whose wavelength is longer than the visible light wave range between 0.75 and 1000 μm.
- This type of radiation has characteristic to transfer thermal energy from warmer object to cooler object.
- The desired heat is produced at the surface of the targeted material, heat is produced on surface by matching the infrared emission spectrum of radiator to the absorption capacity of material

Infrared Definition and Classification

Infrared radiations are part of an electromagnetic spectrum, with wavelength of 750nm-1mm, and frequency of $4x10^{14}$ and $7.4x\ 10^{11}$, and located between microwave and visible light.

Infrared is superficial Heating modality (penetration depth 1- 10mm).





Infra-Red Dryer

Advantages:

- Highly efficient in converting electrical energy into heat for electrical IR.
- Greater rate of heat transfer.
- Compact in size .
- Heats only the object without heating the environment.
- Easy to zone for uniform heating of the product.
- Faster response to changing process conditions.
- Quick start up and shut down.
- Easy to control and automate.
- Lower capital and installation cost .
- Infrared Air dryers suitable for solvent based coating

Disadvantage:

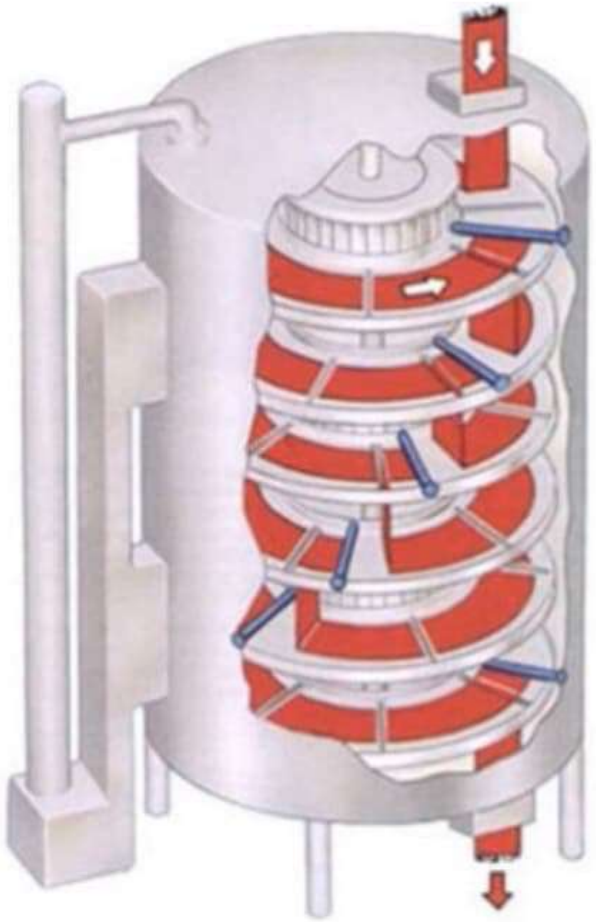
- Expensive

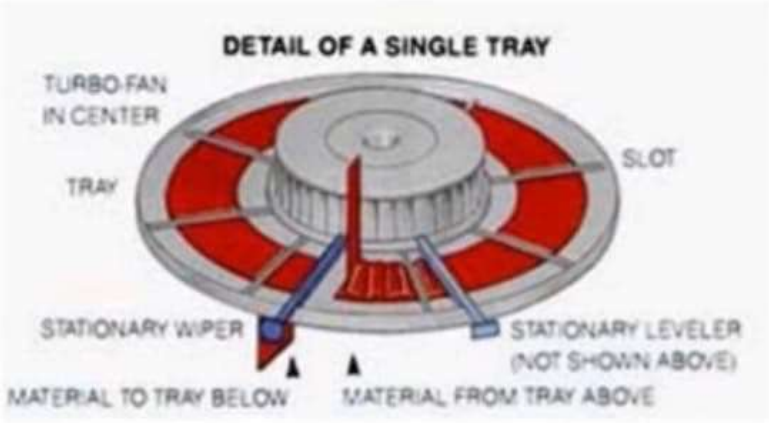
II– Continuous Dryer

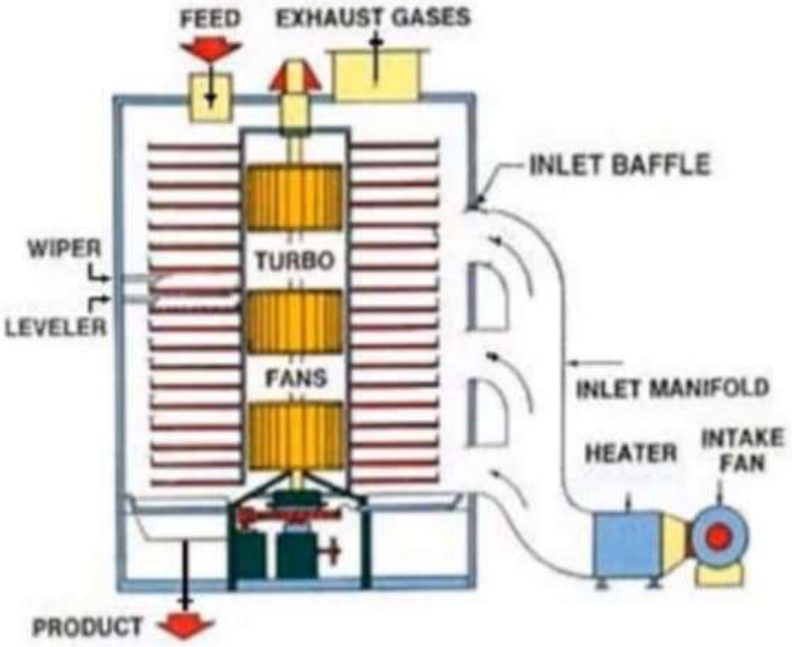
1-Turbo Shelf Dryer

- It is used for too sticky substances or viscous.
- It rotates at 0.1 to 1 r.p.m.
- The product is transferred from one shelf to another after one revolution

Turbo-Tray Dryers





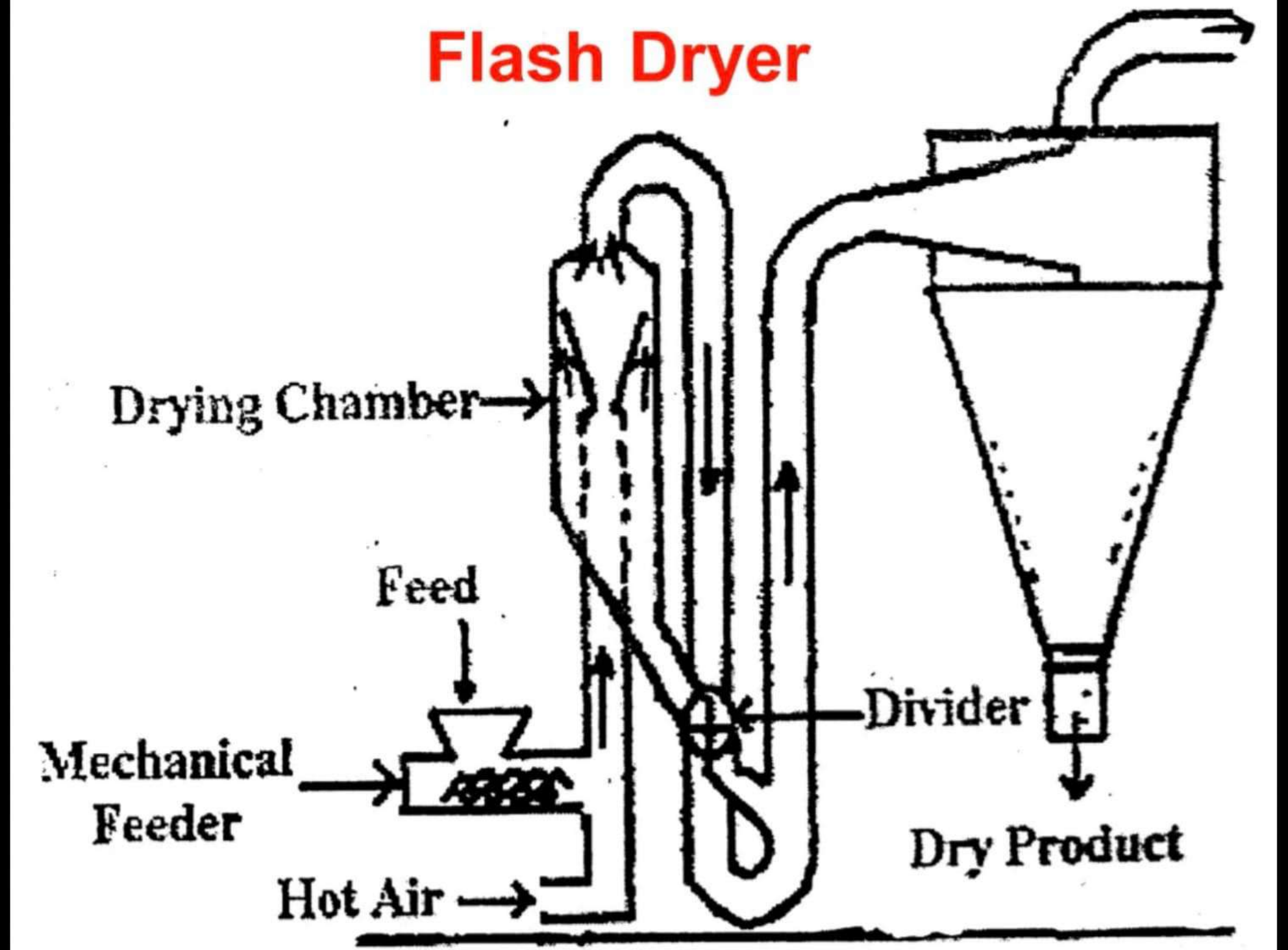


2- Pneumatic Dryer (Flash Dryer)

- The drying time is 3-4 seconds only so it is called flash dryer

- The drying is achieved using hot air with **650°C** so it is suitable for thermolabile due to very short drying time .

Flash Dryer


Drying Chamber
Feed
Mechanical Feeder
Hot Air
Divider
Dry Product

Drum Dryer

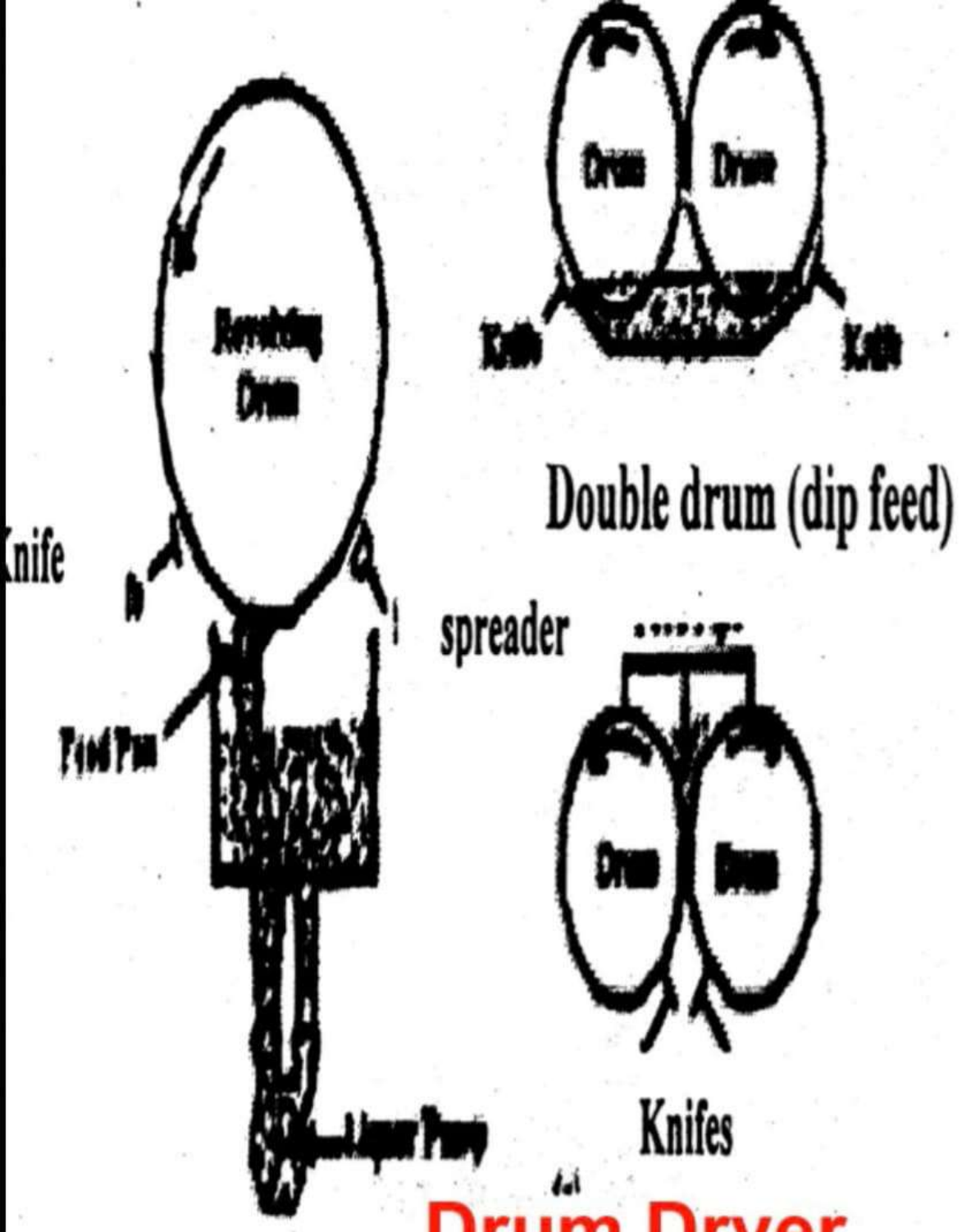
❖ Operation:

1. The li**quid** is applied to the surface and **spread** to a film,

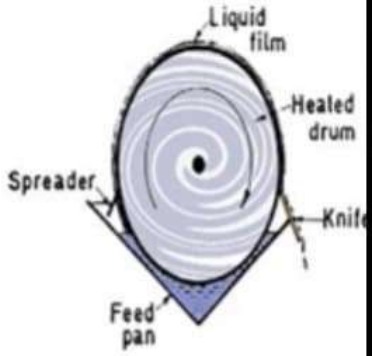
2. This may be done in various ways, but the simplest method **is** that shown in the diagram, where the **drum dips** into **a feed pan**.

3. Drying rate is controlled by using a suitable speed of **rotation** and the **drum temperature**.

4. The product is **scraped** from the surface of the drum by means of a **doctor knife.**



Double drum (dip feed)



Drum Dryer

• Single Drum Vacuum Dryer

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.

Disadvantage

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

4- 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.

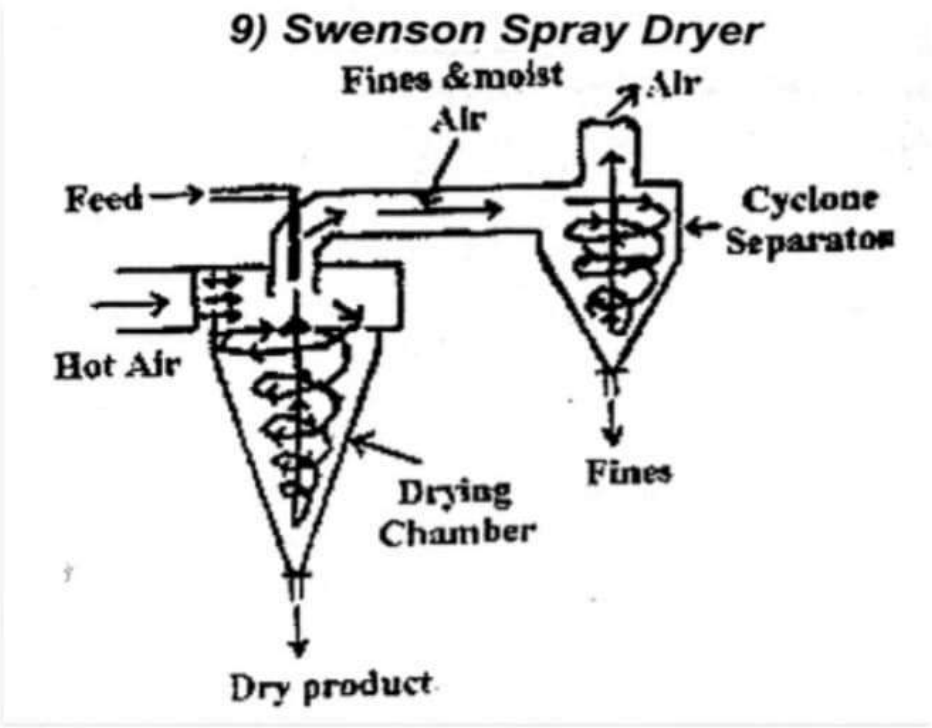
- The drying chamber resembles the cyclone ensuring good circulation of air, to facilitate heat and mass transfer, and that dried particles are separated by the centrifugal action.

Types of atomizer:

A-Pressure atomizer (SPRAY NOZZLE)	B- Disc atomizer
• Used for solutions and thin paste • May be clogged not for suspension or thick paste. • Must be operate at fixed conditions to give the specified P.s. depending on the applied pressure • Used in Swenson Spray Dreyer	• Used even for thick paste • Operated under variable conditions. • Used in instant Spray Dryer.

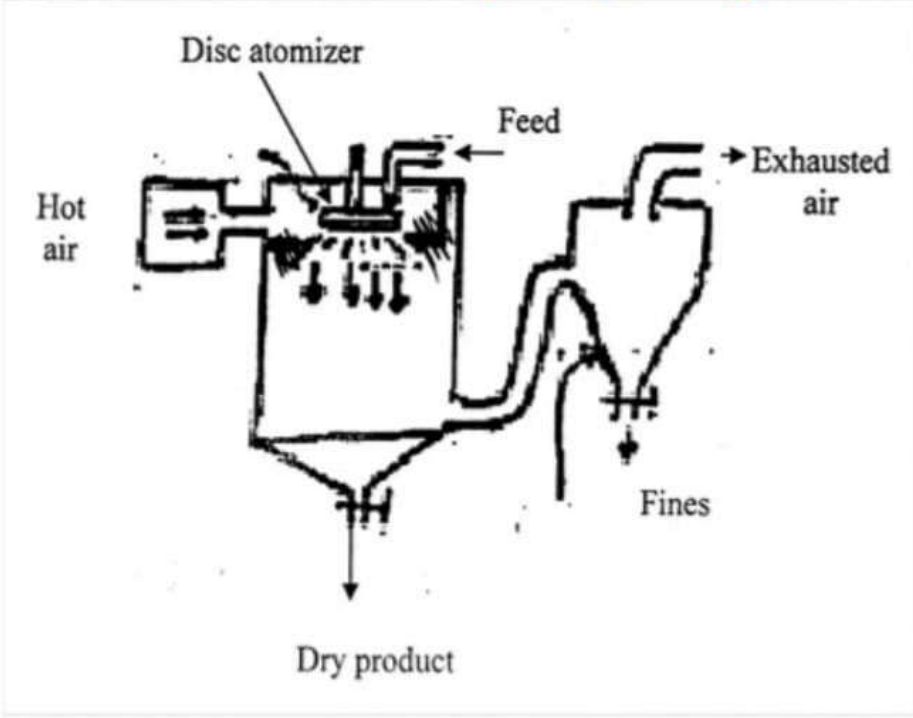
Spray dryers may be classified into 3 types:

A- Swenson Spray Dryer



- Pressure atomizer
- Air flow is parallel & countercurrent
- Cyclonic chamber.
- Nozzle is changed according to the required P.S.
- Used as classifier of the dried powder according to their P.S.

B- Instant Spray Dryer

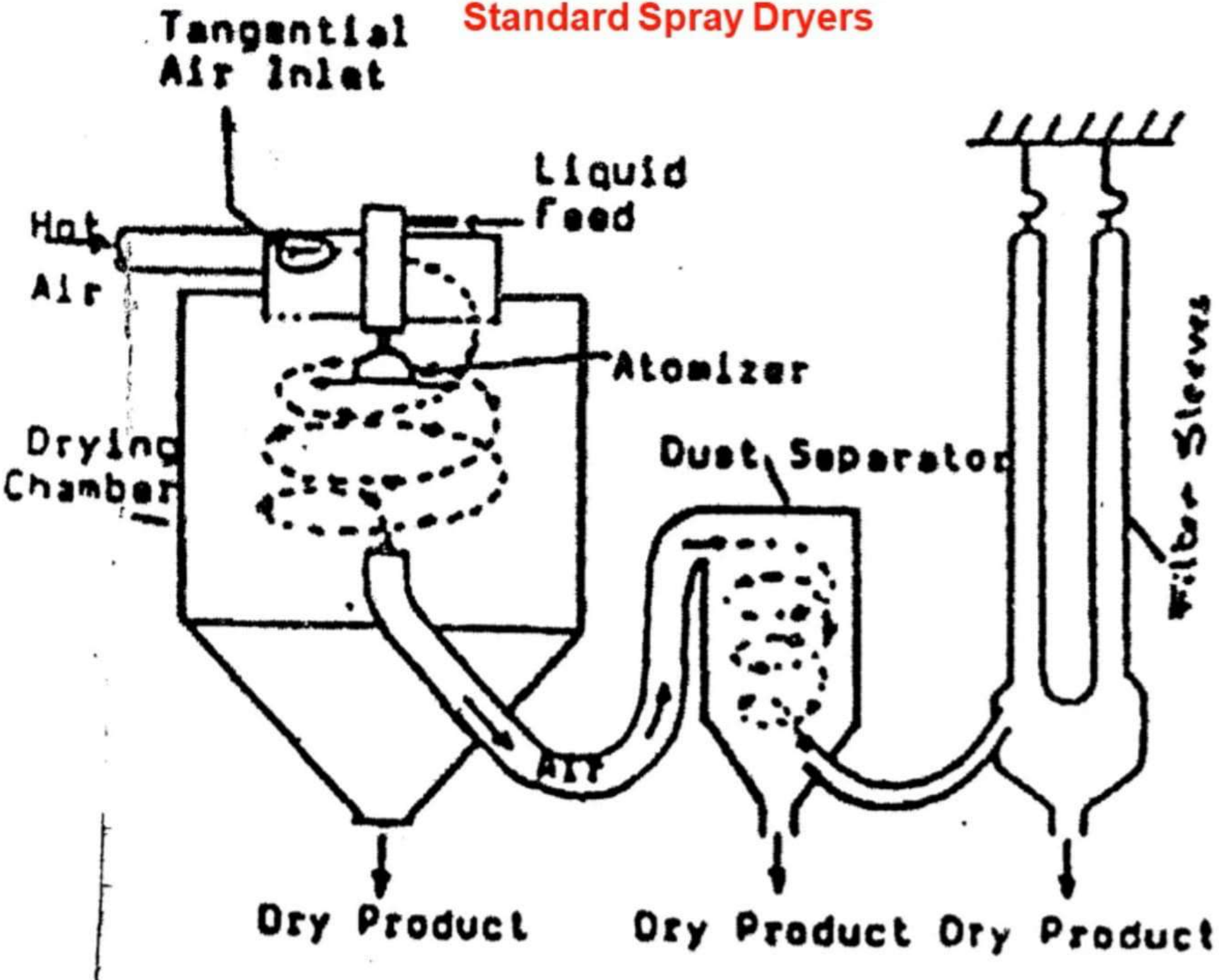


- Disc atomizer (5000-10000rpm).
- Air flow is parallel only (concurrent)
- Cylindrical chamber.
- Used in FOOD product drying as Nescafe.

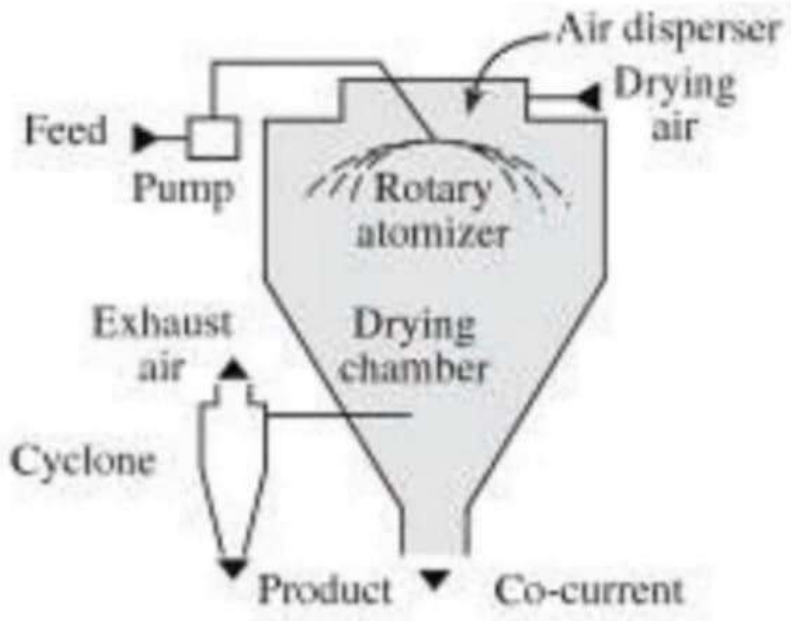
C- Standard Spray Dryers

- Using pressure and disc atomizer
- Cylindrical chamber
- Parallel air flow.
- Shaker → to prevent accumulation of dry product.
- The product is of high solubility, potency, stability and flowability.
- Used as classifier also →(coarse, fine and very fine).

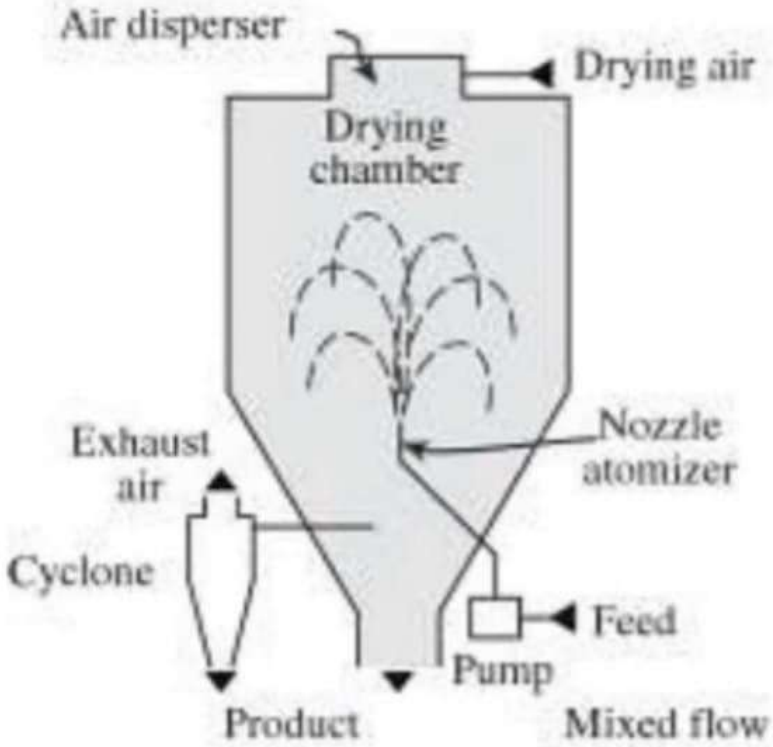
Standard Spray Dryers



Spray Dryer

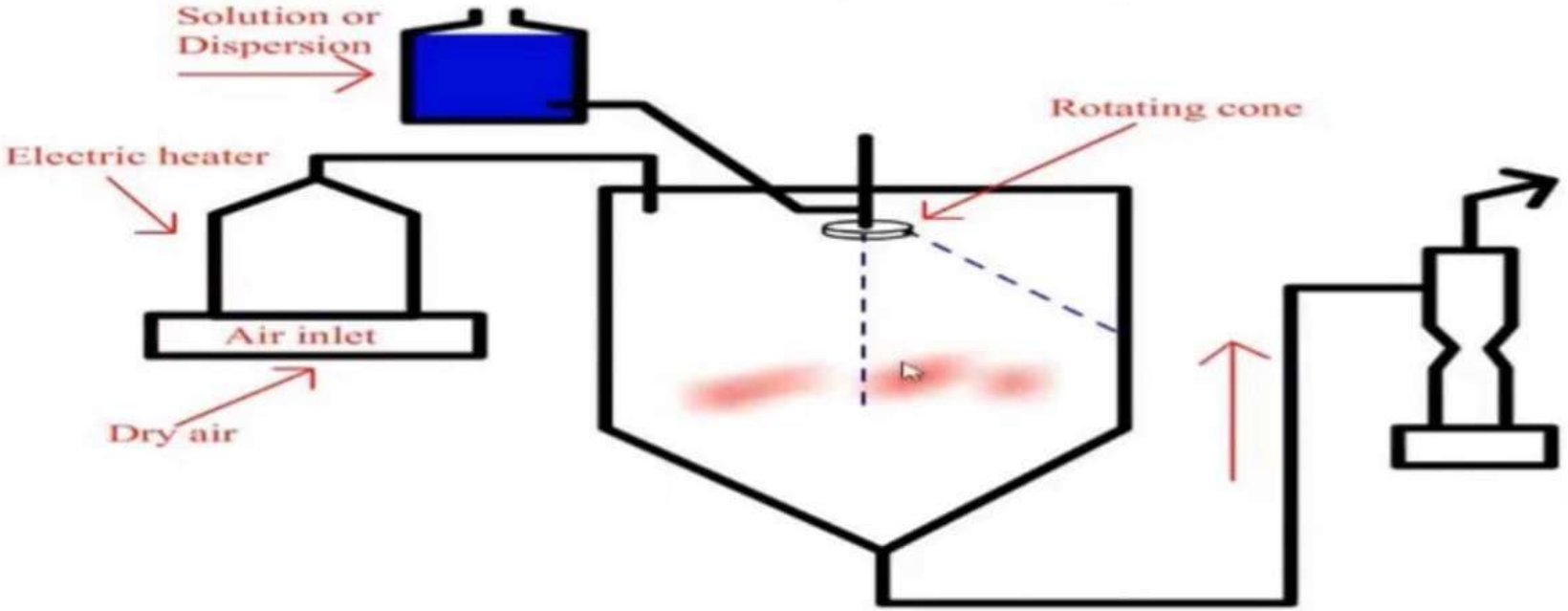


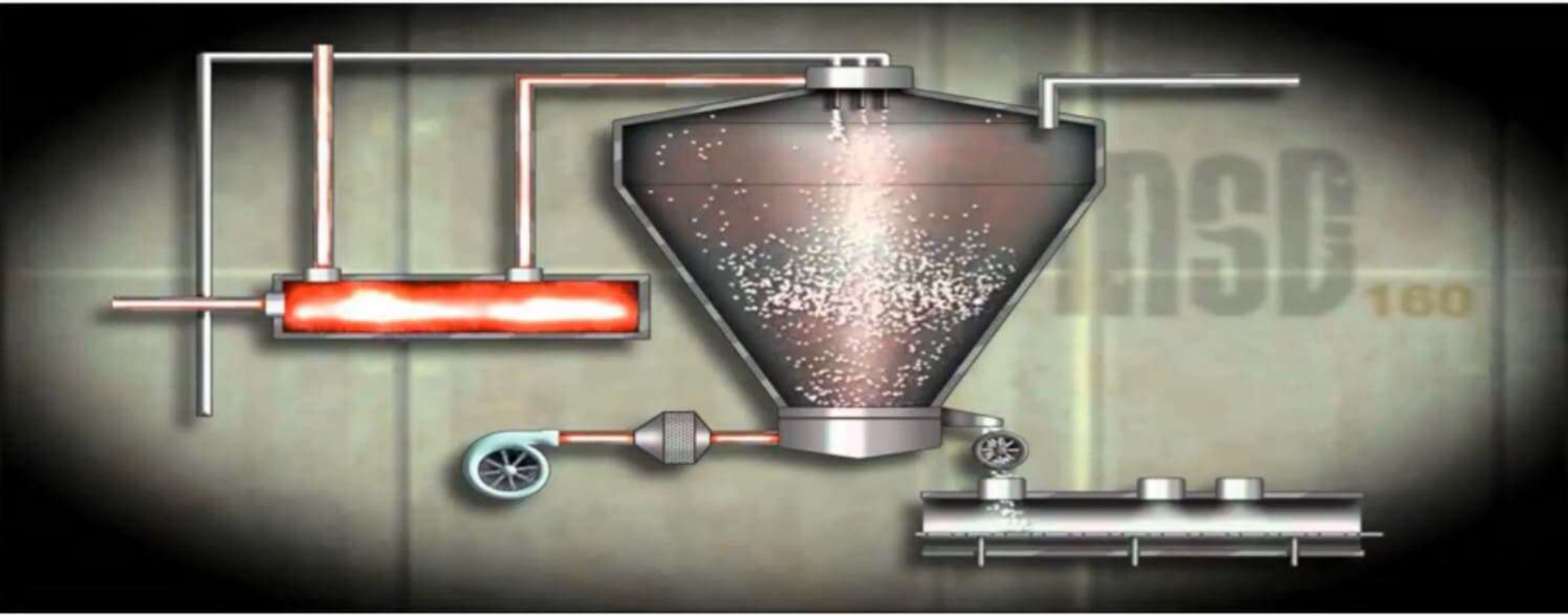
(a) Centrifugal atomizer with cocurrent air flow.



(b) Nozzle atomizer using mixed-flow conditions.

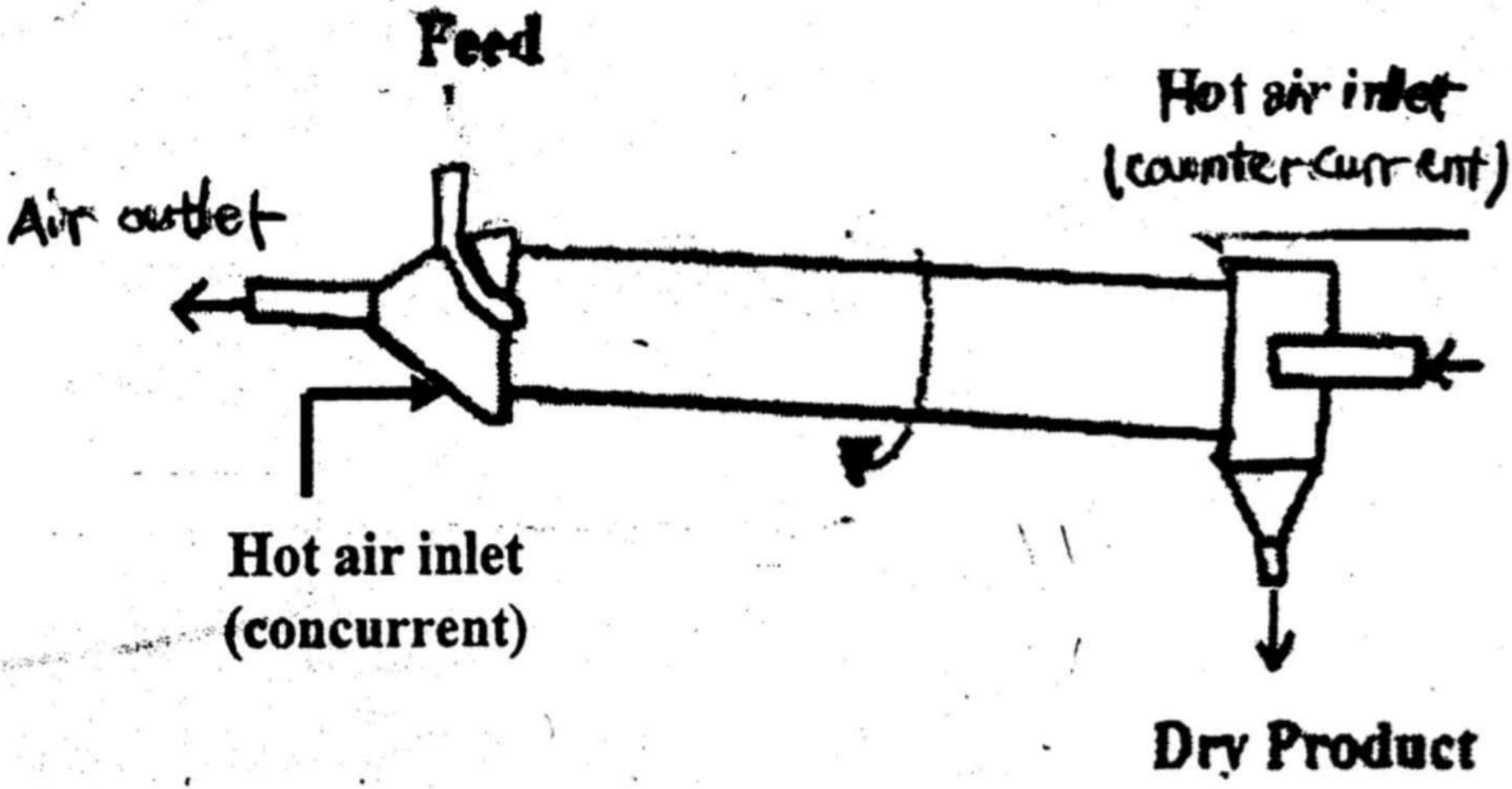
Spray Dryers





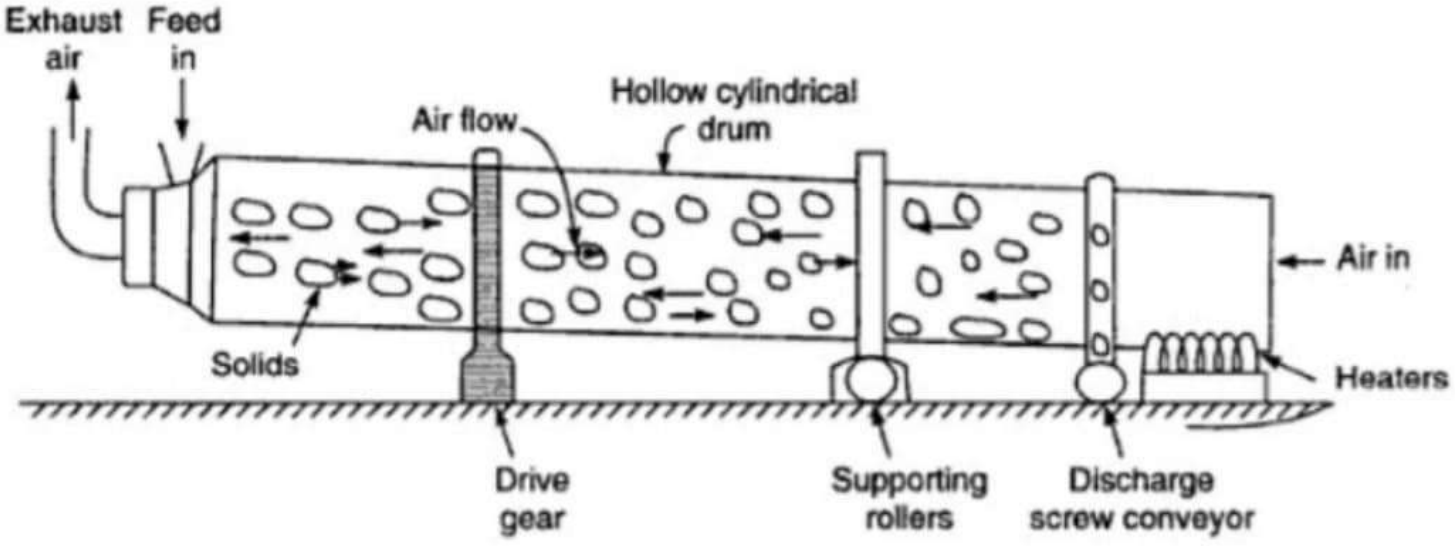
5- Rotary dryer

Rotary dryer is a device that uses rotation, gravity and warm gasses to dry a material.

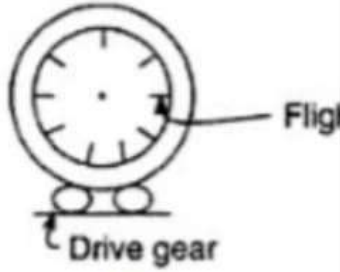


Rotary-Drum Dryer

- Consists of a rotating drum 2 to 3 m in diameter and 15 to 20 m long.
- The dryer is slightly inclined toward the outlet.
- The drum have *flights* used for lifting the substance.
- Air temperatures are usually 120-175°C
- Air velocity ranges from 2000-25,000 kg/m^{2}h for coarse particles



Rotary dryer.



Side view of rotary dryer

Types of rotary dryer

1- Directly heated rotary dryer

- Direct contact between solid and hot air may be classified according to air flow to:

A- Parallel (Co-current) air flow :

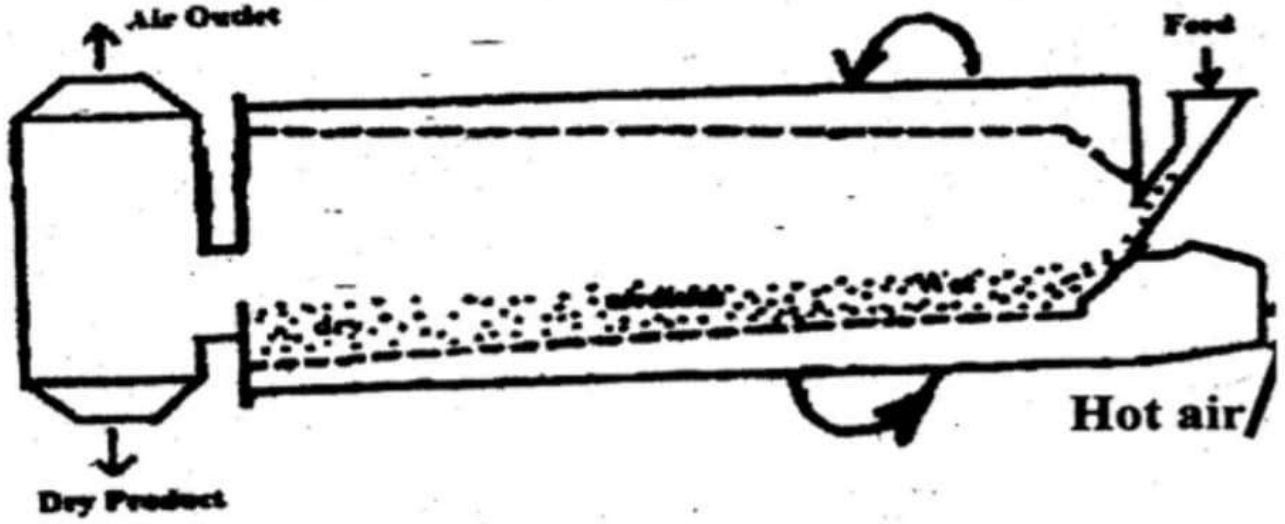
- ❖ Rapid surface drying
- ❖ Used for sticky materials (↑ moisture content)
- ❖ The final product obtained at low temperature
- ❖ Rapid lowering of hot air temperature at the beginning allow its use in heat sensitive materials.
- ❖ **Flights** are added to interfold the powder

B- Countercurrent air flow :

- Solids may be overheated
- The final product is obtained at minimum moisture content
- Final product is obtained at high temperature.

C- Flow through circulation (Roto-Louvre Dryer):

- It consists of 2 concentric rotating cylinders the inner cylinder is perforated to allow passage of hot air through solid bed..
- * The materials continually meets fresh air



2- Indirectly heated rotary dryer

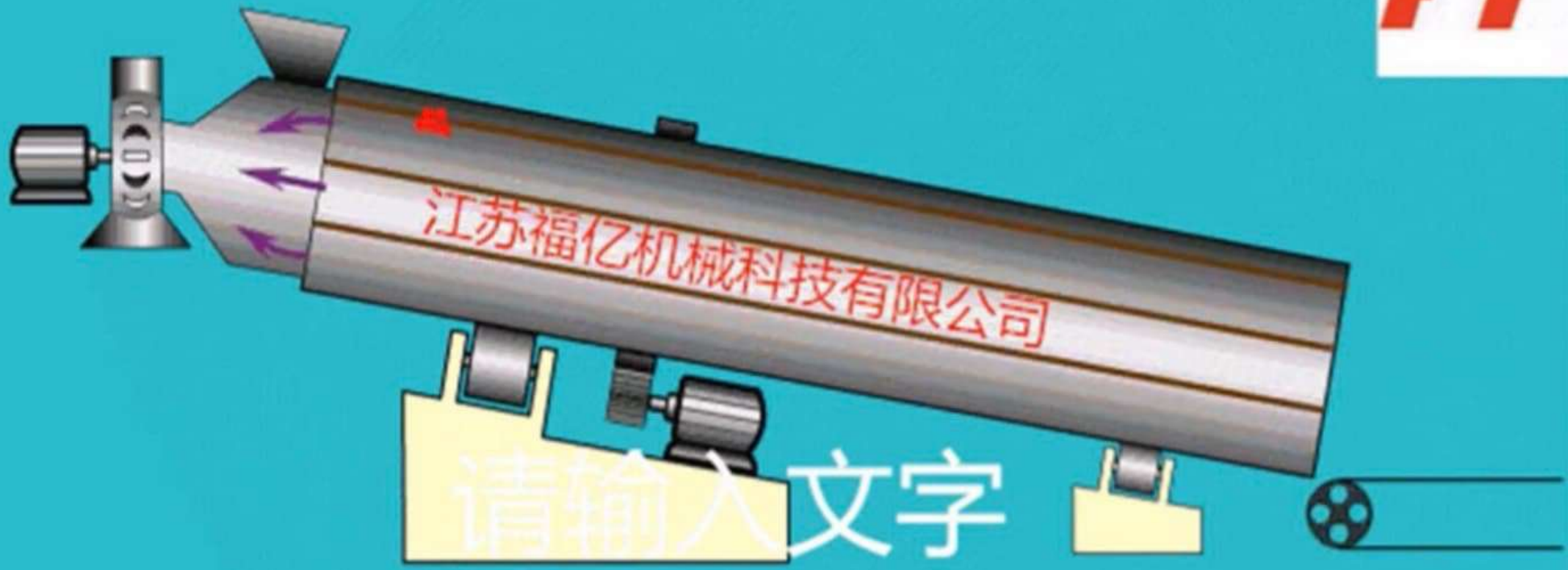
- ❖ Air heated : using indirectly hot air
- ❖ Steam heated : the heating was achieved through a series of steam pipes fitted a long the shell.

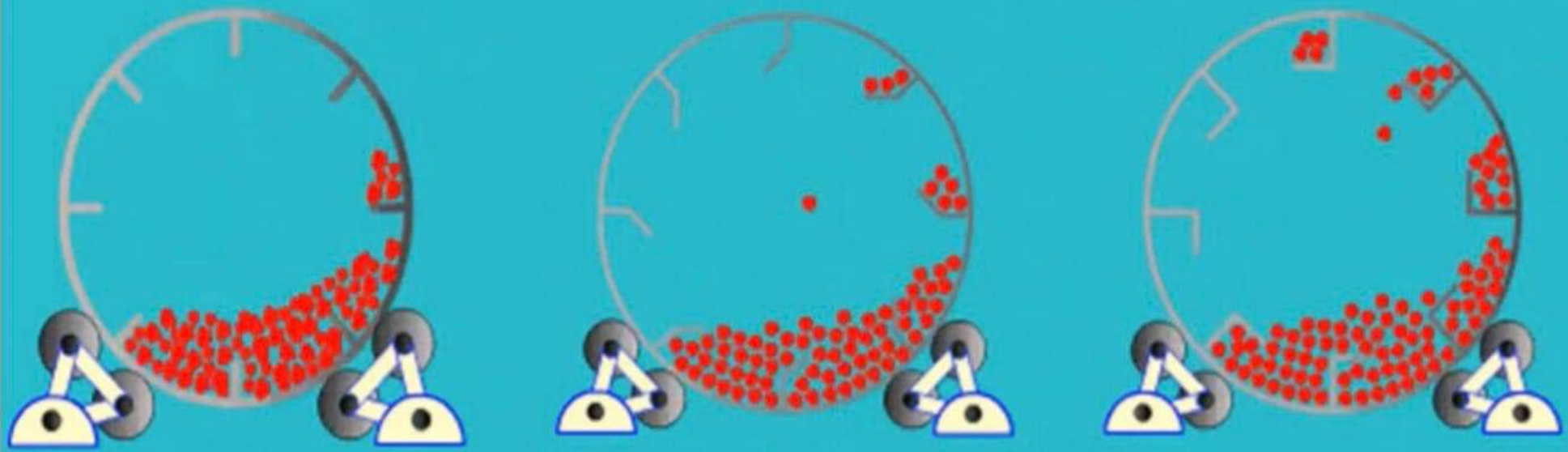
- ➢ In general rotary dryer is used for continuous drying on large scale (4 more tons/hr).

- ➢ The material must be granular crystalline.

加料

Rotary dryer

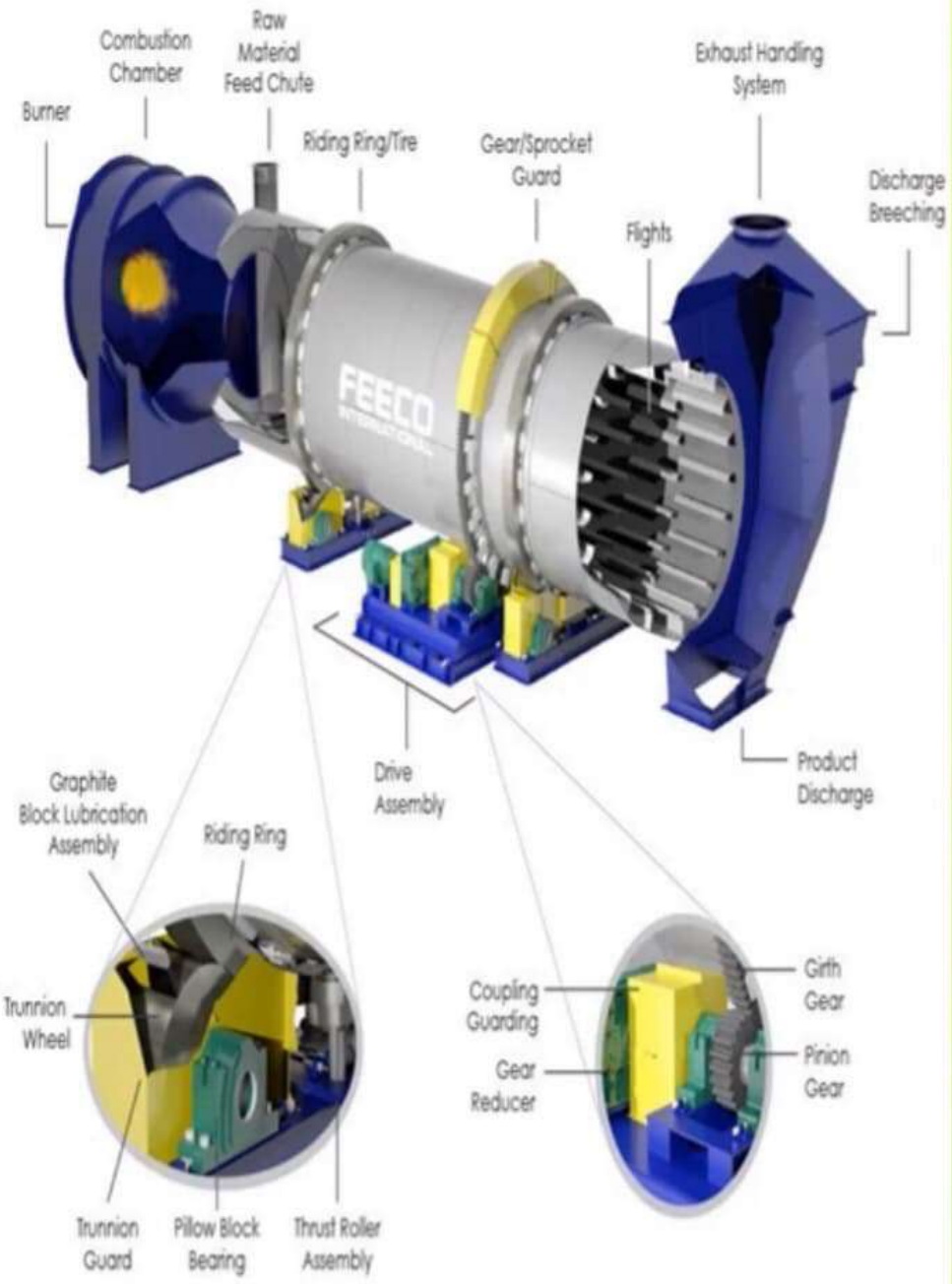




直立抄板　　45° 抄板　　90° 抄板

Mechanical Construction of a

ROTARY DRYER



Rotary drying process

6- Tunnel dryer (STATIC BED)

- They are used when large amounts of materials wanted to be dried.

- The materials are carried on cars and passes through a tunnel

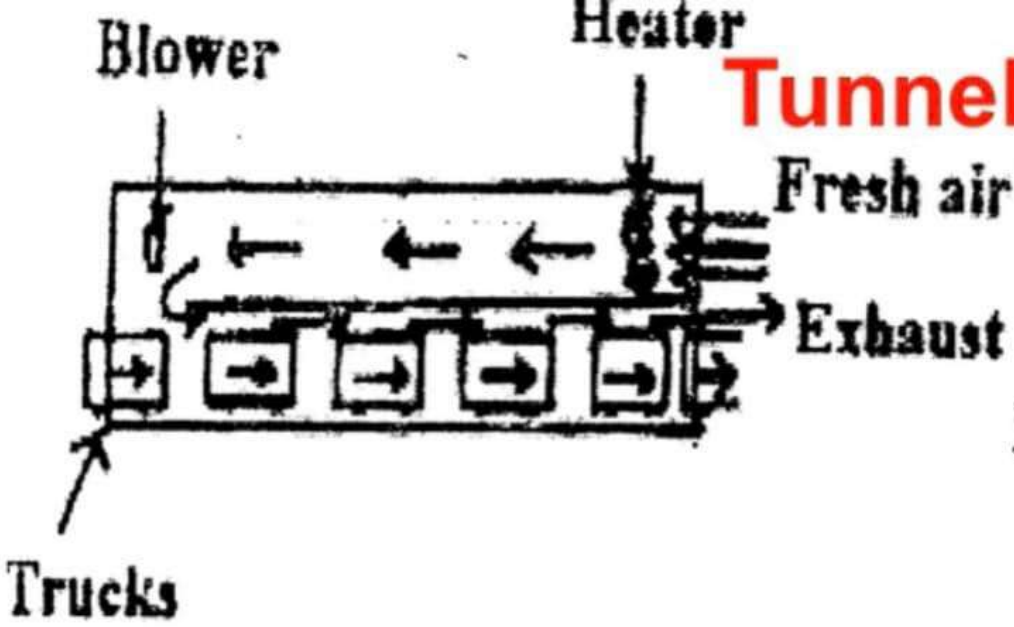
The air flow may be :

1. Parallel.
2. Countercurrent
3. Center-exhaust air flow

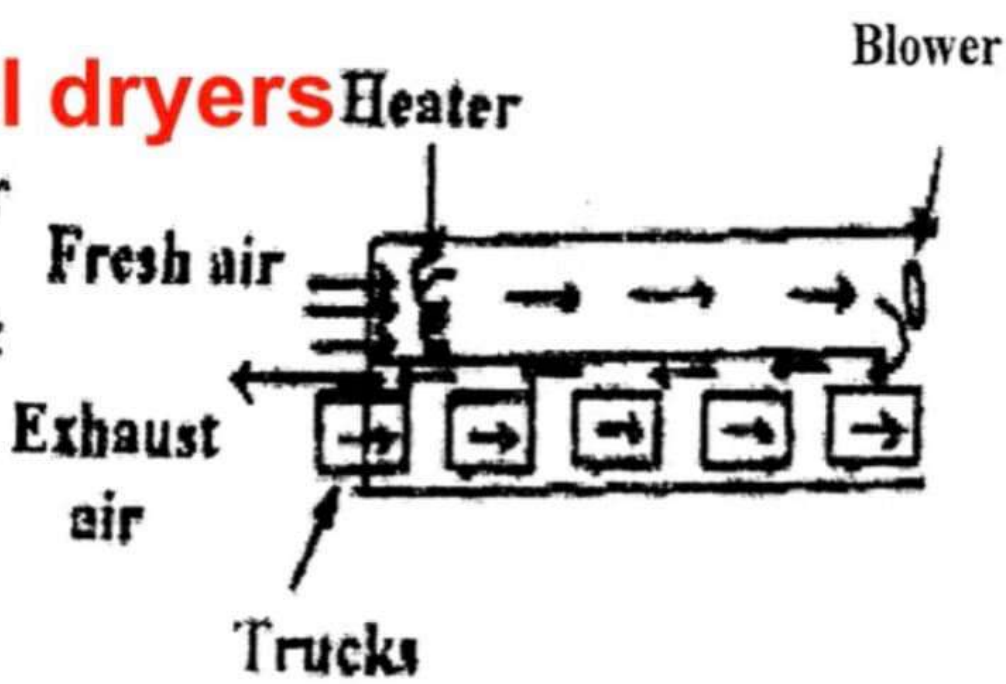
Uses:

Used for brick, ceramics, glassware and large amounts of herbs

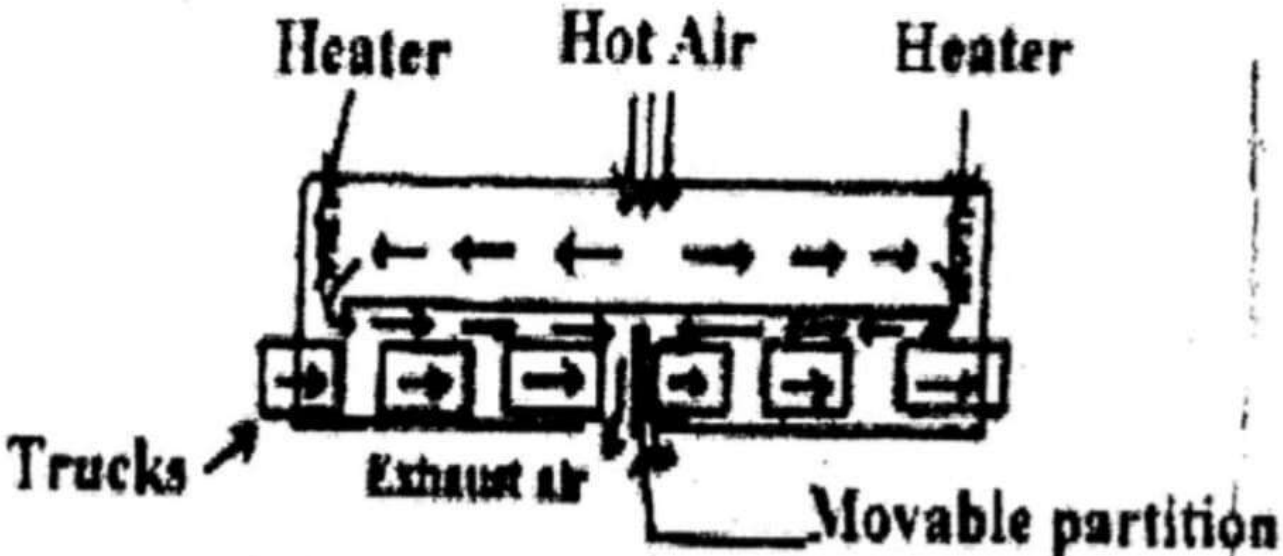
Tunnel dryers



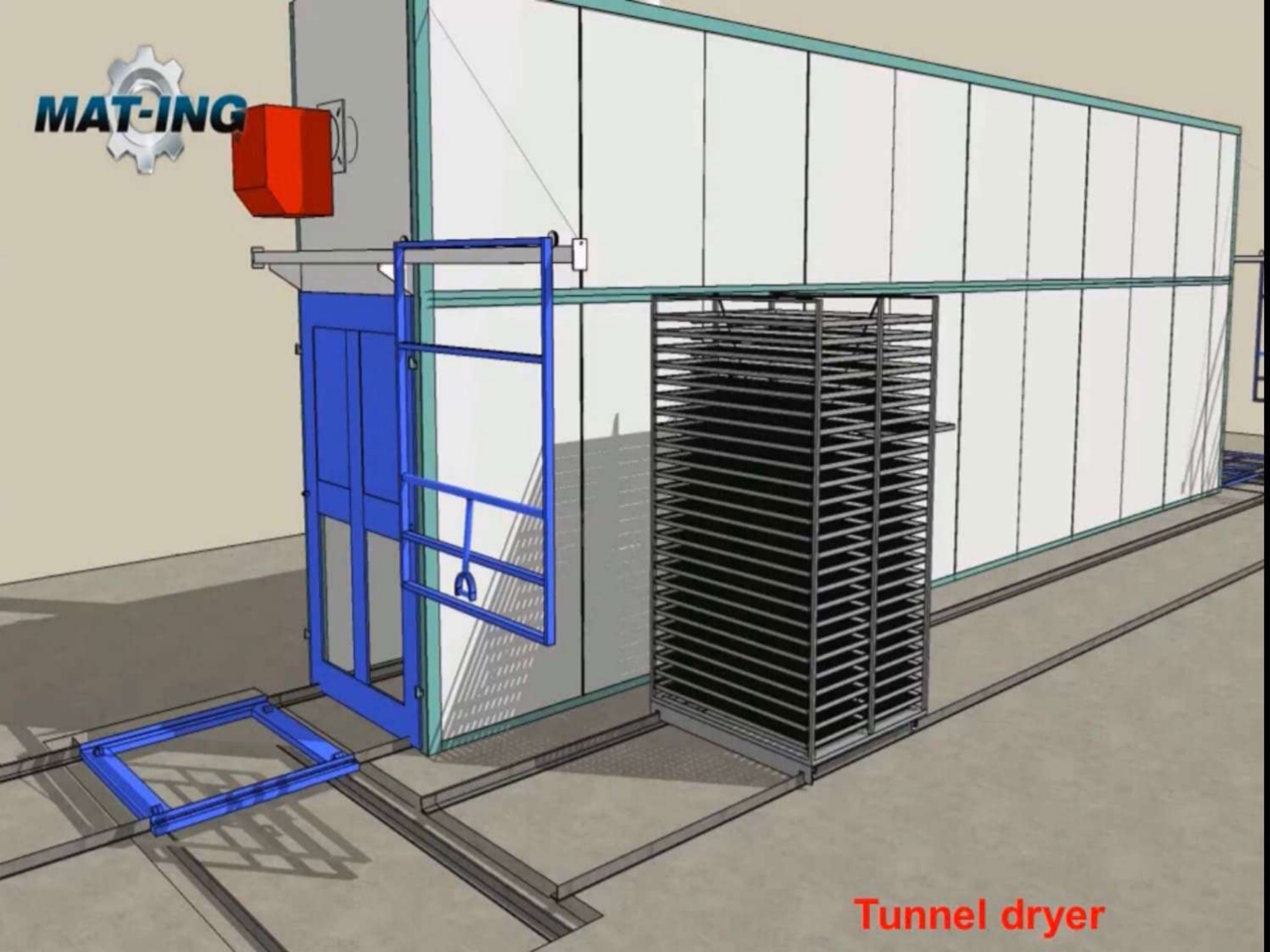
(a) Parallel Current Air Flow



(b) Counter Current Air Fl



(c) Center Exhaust Air Flow


MAT-ING
Tunnel dryer