Novel Technologies for Food Processing and Shelf Life Extension
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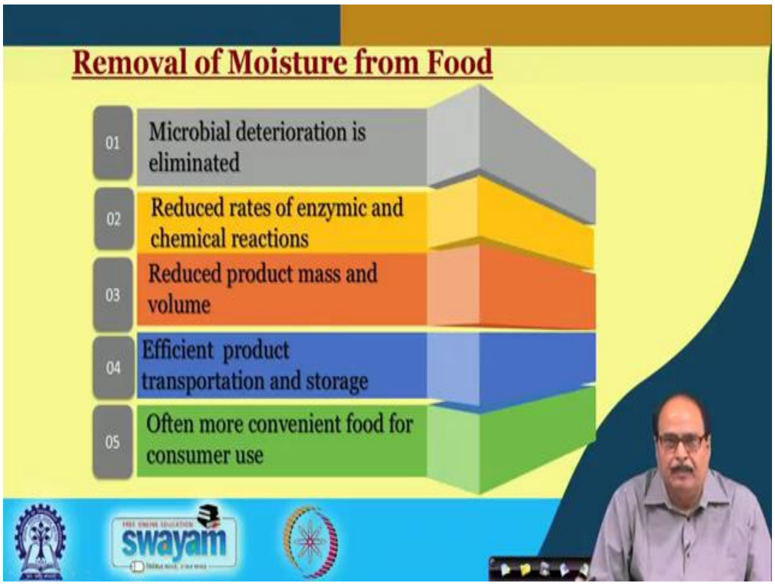
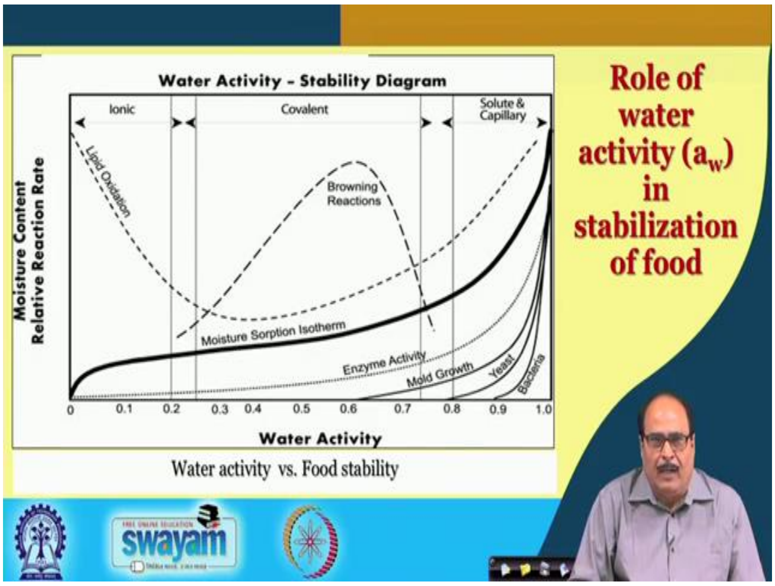
Lecture – 21
Freeze Drying (Part 1)

Freeze drying topic is divided in two parts. In part 1, the fundamental aspects of freeze drying and the systems is discussed while in the second part, the different equipment used for freeze drying of various foods, few case studies or applications of freeze drying in food are elaborated.



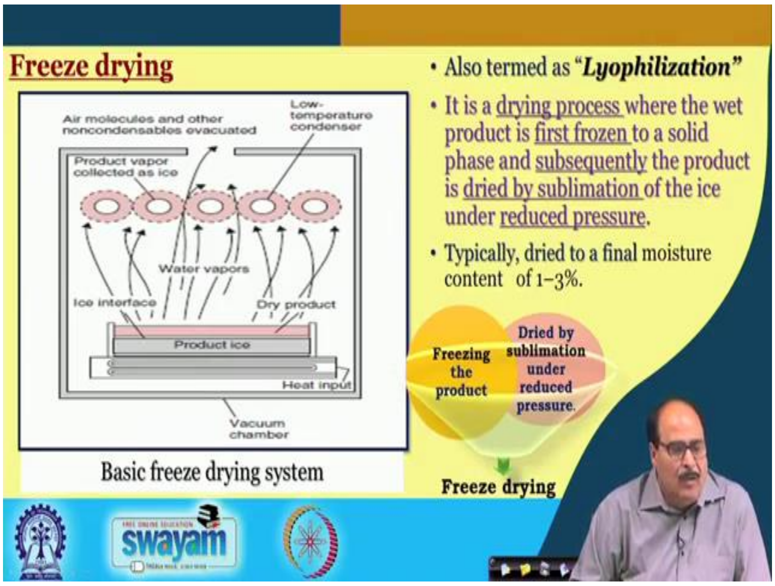
Removal of moisture from the food is an important operation for its preservation as it results in the microbial deterioration, reduced rates of enzyme and chemical reactions, reduced product mass and volume, efficient product transportation and storage and often more convenient food for consumer use.



Water activity vs. Food stability

Role of water activity (a_w) in stabilisation of food

The concept of water activity versus food stability is already described in earlier classes. In the figure, it could be seen that water present in ionic, covalent or in the capillary or physically entrapped in the tissue matrix take part in the chemical reactions. Bacteria and yeast grow only at water which is present in solute and capillary, whereas, the other process like browning reaction, enzyme activity, and mold growth can occur when water is present in covalent form. In the lipid oxidation reaction, even the ionic water which is more firmly bound in the food material can also be involved. So, this shows forms and presence of water in the food material which ultimately affect the dehydration process.

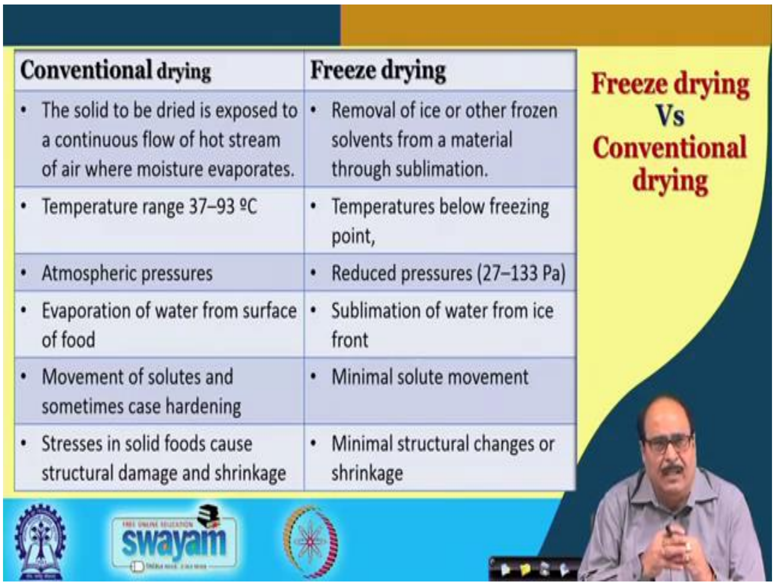


Basic freeze drying system

Freeze drying

It is also termed as lyophilisation. It is a drying process where the wet product is first frozen to a solid phase and this frozen mass is subsequently dried by sublimation of the ice under reduced pressure. Typically, the freeze dried foods might contain moisture as low as 1 to 3 %.

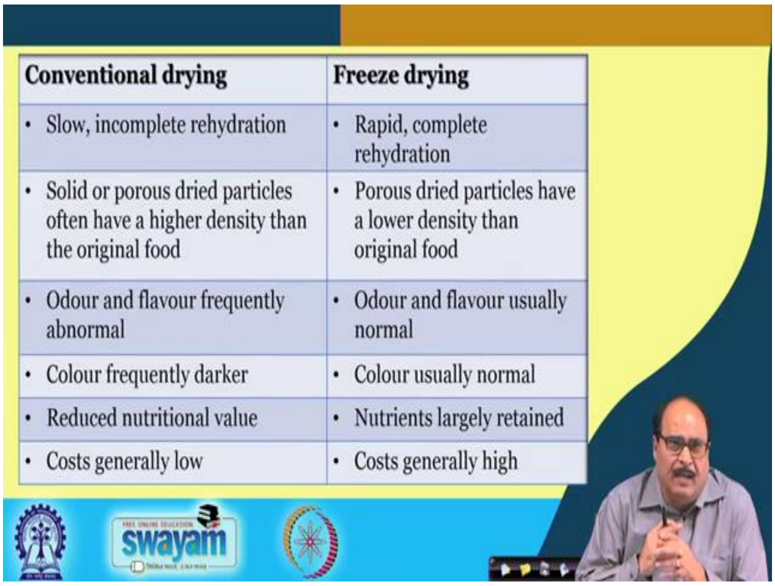
There are two basic steps in the freeze drying process (i) Freezing of the product and (ii) Drying of the frozen mass by sublimation process. The freeze drying process is represented in the figure wherein the moisture is removed from the product in ice form by sublimation process under reduced pressure.



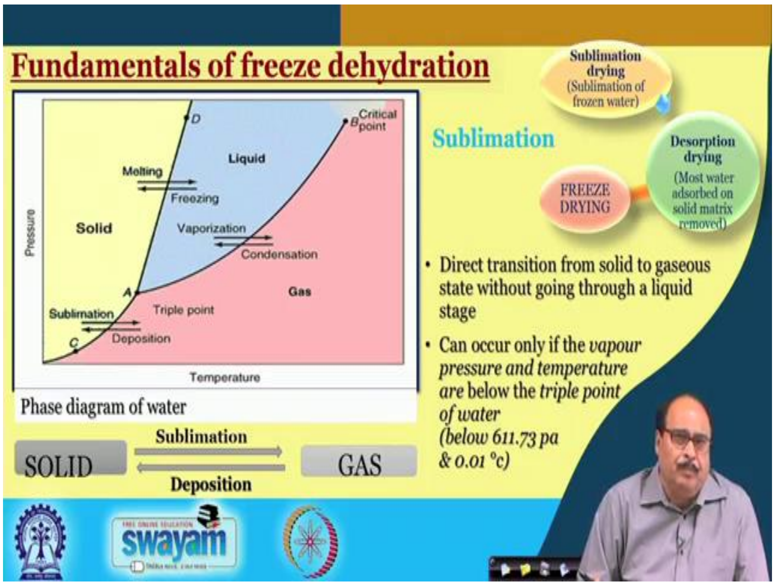
A brief comparison between the freeze drying process and conventional drying processes is presented (see table). The freeze drying is much better in many aspects than that of the conventional drying.

Conventional drying	**Freeze drying**
• The solid to be dried is exposed to a continuous flow of hot stream of air where moisture evaporates.	• Removal of ice or other frozen solvents from a material through sublimation.
• Temperature range 37–93 °C	• Temperatures below freezing point,
• Atmospheric pressures	• Reduced pressures (27–133 Pa)

• Evaporation of water from surface of food	• Sublimation of water from ice front
• Movement of solutes and sometimes case hardening	• Minimal solute movement
• Stresses in solid foods cause structural damage and shrinkage	• Minimal structural changes or shrinkage



Conventional drying	**Freeze drying**
• Slow, incomplete rehydration	• Rapid, complete rehydration
• Solid or porous dried particles often have a higher density than the original food	• Porous dried particles have a lower density than original food
• Odour and flavour frequently abnormal	• Odour and flavour usually normal
• Colour frequently darker	• Colour usually normal
• Reduced nutritional value	• Nutrients largely retained
• Costs generally low	• Costs generally high

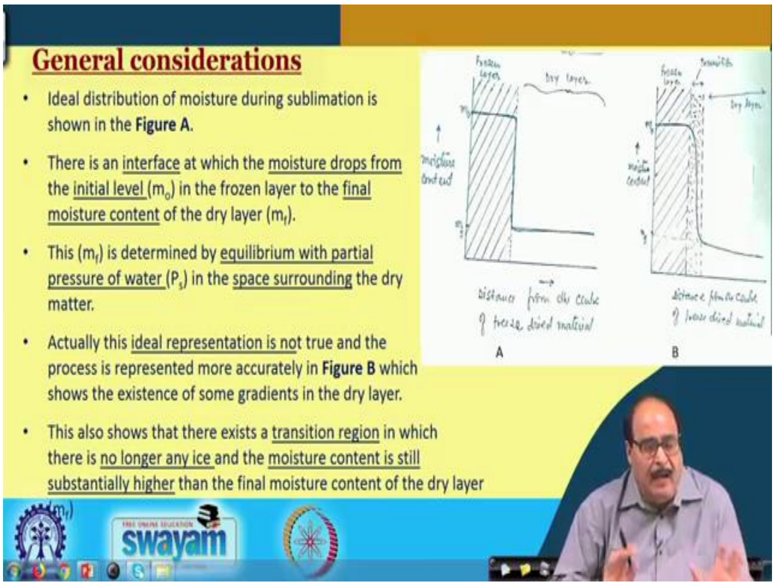


Fundamentals of freeze dehydration

The phase diagram of water (pressure vs. temperature) is presented in the figure. The changes in the pressure and temperature of the system change the state of the substance i.e. water. The state of the substance means solid, liquid and vapour state. In figure, the point 'A' is called as triple point because at this point, all the three states of water coexist. The pressure and temperature at this point is 4.18 Torr and 0°C or in other words it is about 611.73 Pa and 0.01°C.

If the system containing frozen solid mass i.e. ice mass maintained at the pressure and temperature below the triple point, then there is direct conversion of solid (ice) into gas (water vapour). This process is called as sublimation. In freezing, when the similar conditions are applied, the gas also can be directly converted into solid phase, which is called as deposition.

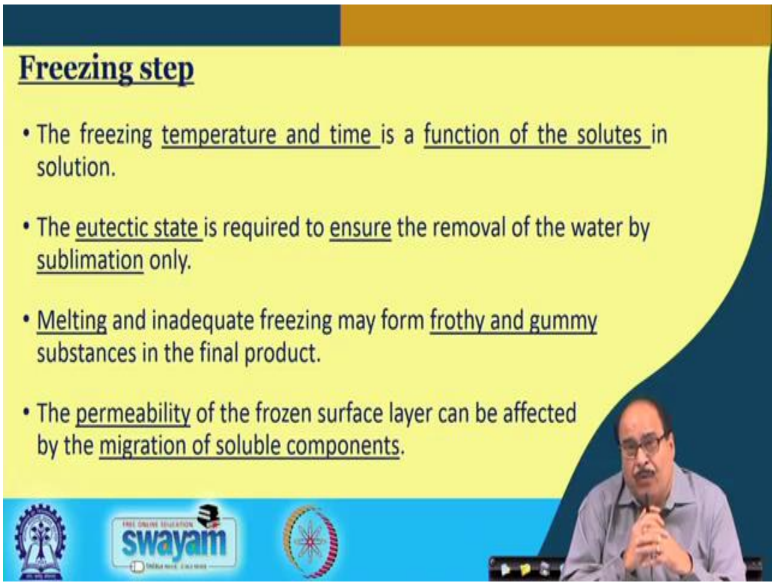
On the other hand, if the system pressure and temperature is above the triple point, the solid will change to liquid that is called as melting and the liquid will change to solid which is called freezing. Similarly, the liquid will change to gas which is known as vaporization and gas to liquid is called condensation. In this way, the change in the temperature and pressure influence various phase transition processes. In freeze drying process, the direct transition from solid to gaseous state without going through a liquid stage is important. This can only if the vapour pressure and temperature are below the triple point of water (below 611.73 Pa & 0.01 °c)



General considerations

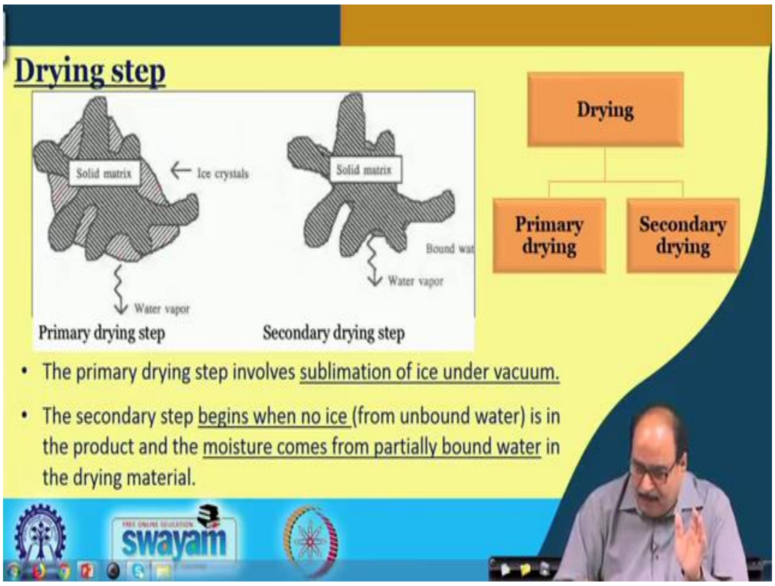
Ideal distribution of moisture during sublimation is shown in the Figure A. There is an interface at which the moisture drops from the initial level (m_o) in the frozen layer to the final moisture content of the dry layer (m_f). This (m_f) is determined by equilibrium with partial pressure of water (P_s) in the space surrounding the dry matter.

Actually this ideal representation is not true and the process is represented more accurately in Figure B which shows the existence of some gradients in the dry layer. This also shows that there exists a transition region in which there is no longer any ice and the moisture content is still substantially higher than the final moisture content of the dry layer (m_f).



Freezing step

- The freezing temperature and time is a function of the solutes in solution.
- The eutectic state is required to ensure the removal of the water by sublimation only.
- Melting and inadequate freezing may form frothy and gummy substances in the final product.
- The permeability of the frozen surface layer can be affected by the migration of soluble components.

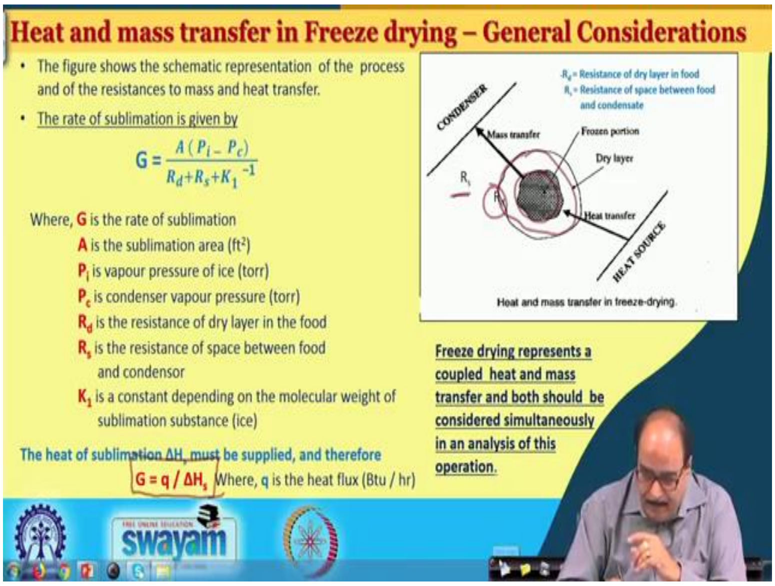


Drying step

Drying occurs in two parts (i) primary drying and (ii) secondary drying.

- The primary drying step involves <u>sublimation of ice under vacuum.</u>
- The secondary step <u>begins when no ice</u> (from unbound water) is in the product and the <u>moisture comes from partially bound water</u> in the drying material.

Therefore, the primary and secondary drying step are important for process analysis.



Heat and mass transfer in Freeze drying – General Considerations

The schematic representation of the heat and mass transfer occurring during freeze drying is shown in the figure. Freeze drying represents a coupled heat and mass transfer and both should be considered simultaneously in an analysis of the operation.

It can be seen in the figure that, the heat is supplied to the food material in proper conditions (temperature & pressure) which causes the sublimation of the ice. The water vapour is then removed through condenser.

The rate of sublimation is given by

$$G = \frac{A\,(P_i - P_c)}{R_d + R_s + K_1^{-1}}$$

Where, **G** is the rate of sublimation

A is the sublimation area (ft^2)

$\mathbf{P_i}$ is vapour pressure of ice (torr)

$\mathbf{P_c}$ is condenser vapour pressure (torr)

$\mathbf{R_d}$ is the resistance of dry layer in the food

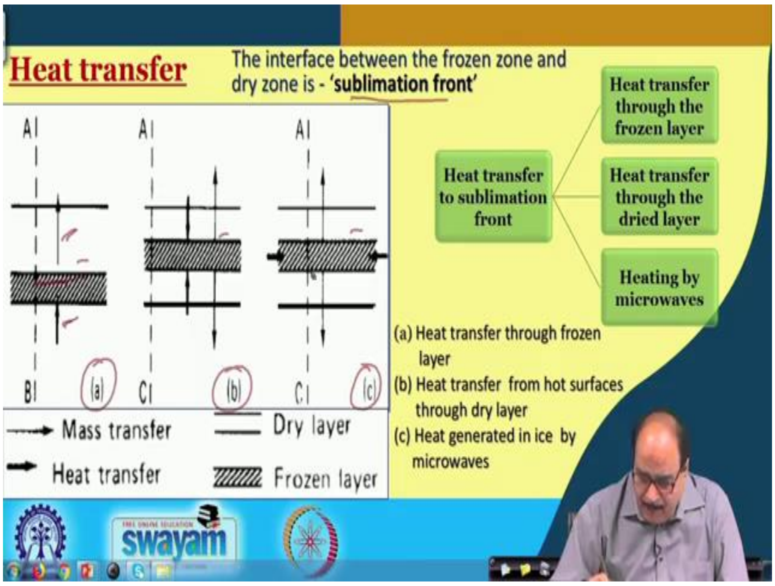
$\mathbf{R_s}$ is the resistance of space between food and condensor

$\mathbf{K_1}$ is a constant depending on the molecular weight of sublimation substance (ice)

At the same time, there must be a proper supply of heat of sublimation ΔH_s,

$$\mathbf{G = q / \Delta H_s}$$

Where, **q** is the heat flux (Btu / hr)

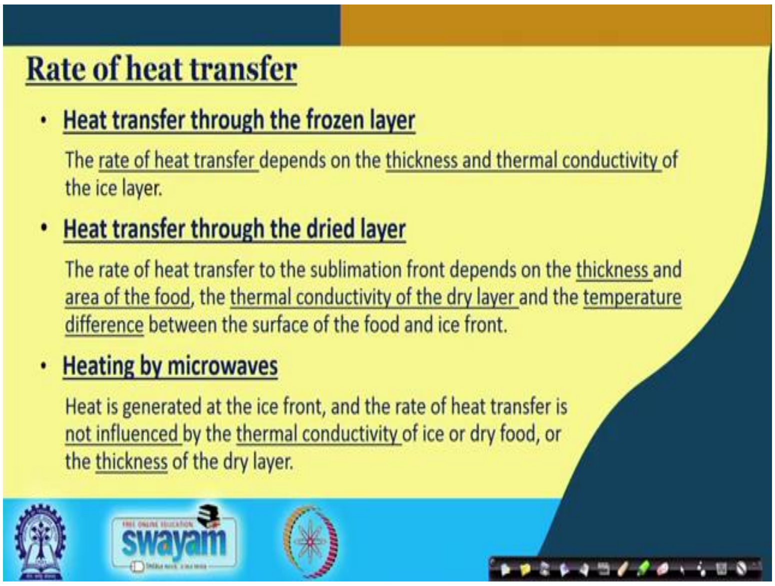


Heat transfer

The interface between the frozen zone and dry zone is - 'sublimation front' (see figures). From this front, solid is getting sublimed into vapour. So, it indicates that the heat transfer occurs in sublimation front. There are three ways in which this heat transfer might occur.

(i) Heat transfer through the frozen layer
(ii) Heat transfer through the dry layer
(iii) Internal heating by microwaves

In figure, A, B and C – three cases of the freeze drying processes are shown. In the case A, heat transfer occurs through the frozen layer and mass transfer through the dry layer and both of them are in the same direction. In case B, the heat transfer and mass transfer are through the dry layer and they are in opposite direction whereas, in the case C, the heat is generated internally by microwaves and heat transfer here is by radiation and mass transfer is through the dry layer. Therefore, while calculating the process efficiency, all the situations of the freeze drying should be properly considered.



Rate of heat transfer

a) **Heat transfer through the frozen layer**

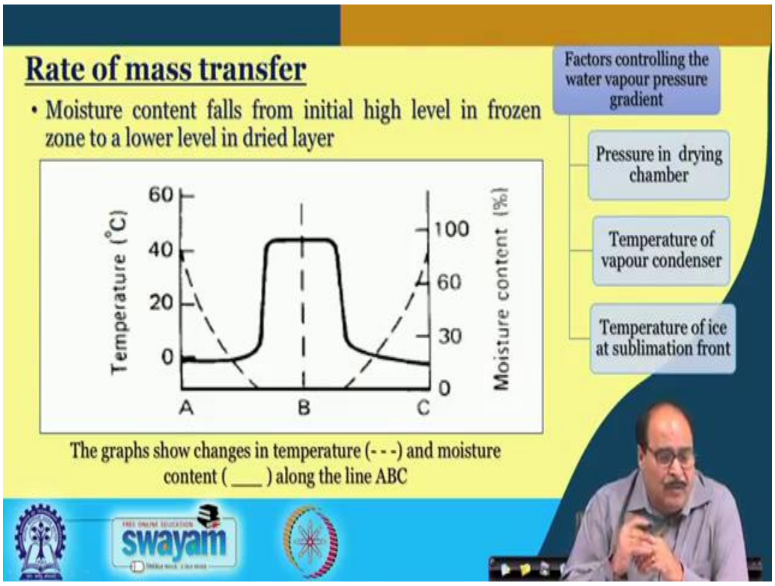
The rate of heat transfer depends on the thickness and thermal conductivity of the ice layer.

b) **Heat transfer through the dried layer**

The rate of heat transfer to the sublimation front depends on the thickness and area of the food, the thermal conductivity of the dry layer and the temperature difference between the surface of the food and ice front.

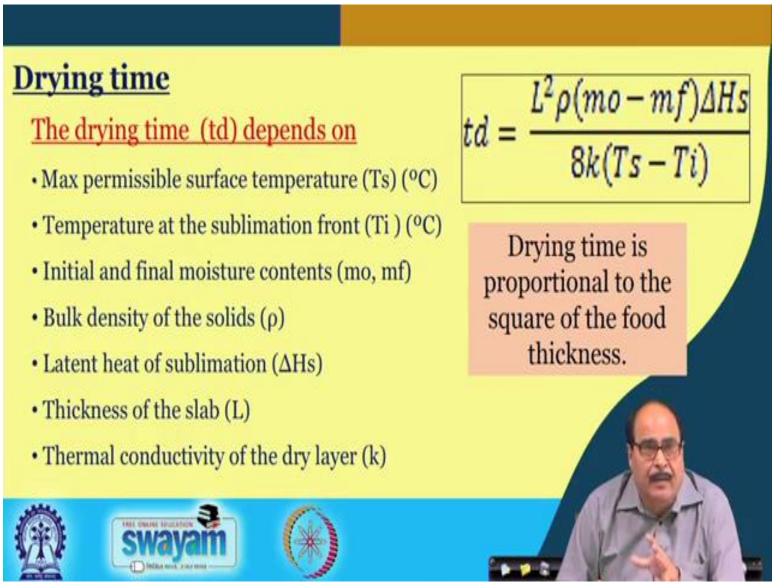
c) **Heating by microwaves**

Heat is generated at the ice front, and the rate of heat transfer is not influenced by the thermal conductivity of ice or dry food, or the thickness of the dry layer.



Rate of mass transfer

The figure shows the changes in temperature causes change in the rate of the mass transfer in terms of moisture change. The dotted line shows the temperature change and continuous line shows the moisture content. It can be seen that moisture content falls from initial high level in frozen zone to a lower level in dried layer. The water vapour pressure gradient actually determines the rate of removal of water and depends on the factors such as (i) pressure in the drying chamber temperature (ii) temperature of the vapour condenser and (iii) temperature of the ice at sublimation front.



<u>**Drying time**</u>

Drying time in the freeze drying process can be calculated by using the formula

$$td = \frac{L^2 \rho (mo - mf) \Delta Hs}{8k(Ts - Ti)}$$

Where, Ts is Max permissible surface temperature (ºC)

Ti is Temperature at the sublimation front () (ºC)

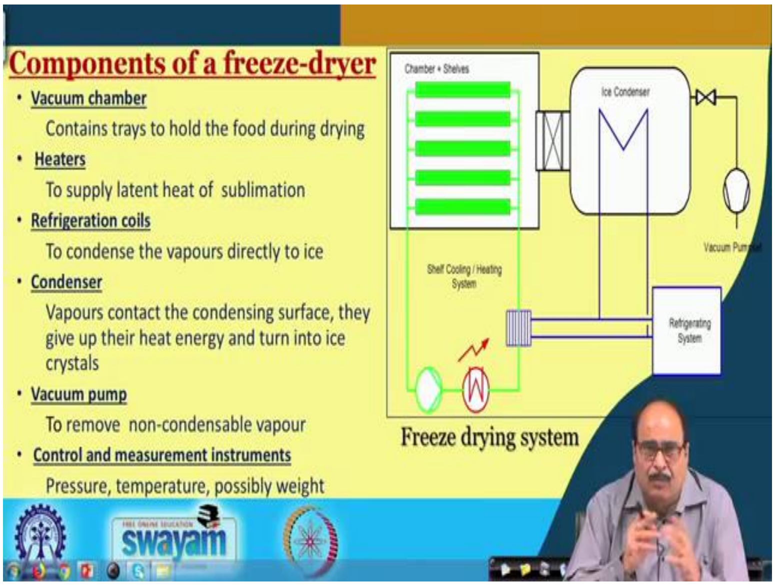
mo, mf are Initial and final moisture contents (%db)

ρ is Bulk density of the solids (kg/m^3)

ΔHs is Latent heat of sublimation (J/kg)

L is Thickness of the slab (m)

k is Thermal conductivity of the dry layer (W/m ºC)



Components of a freeze-dryer

1. **Vacuum chamber**

   Contains trays to hold the food during drying

2. **Heaters**

   To supply latent heat of sublimation

3. **Refrigeration coils**

   To condense the vapours directly to ice

4. **Condenser**

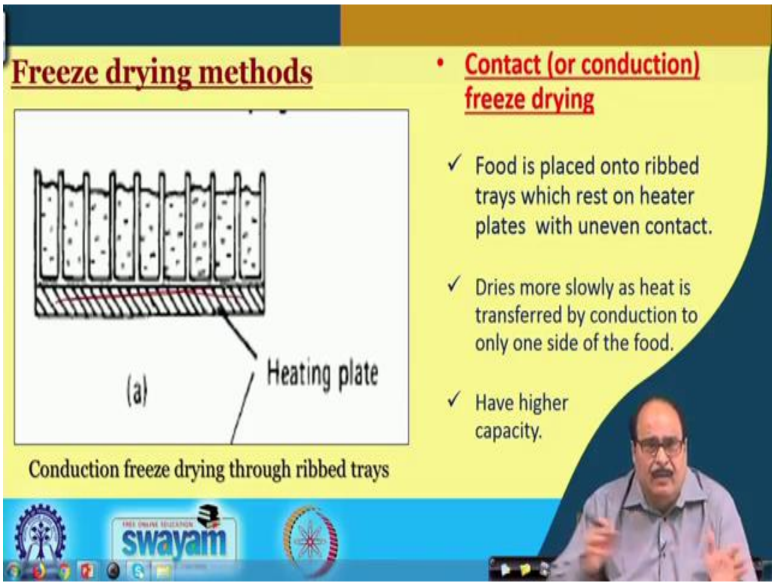
Vapours contact the condensing surface, they give up their heat energy and turn into ice crystals

5. **Vacuum pump**

To remove non-condensable vapour

6. **Control and measurement instruments**

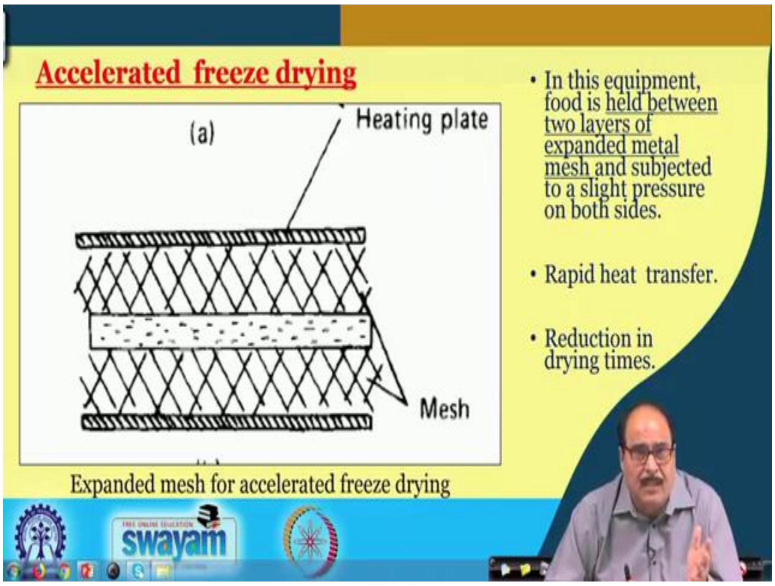
Pressure, temperature, possibly weight measurement instrument are provided in equipment.



Freeze drying methods

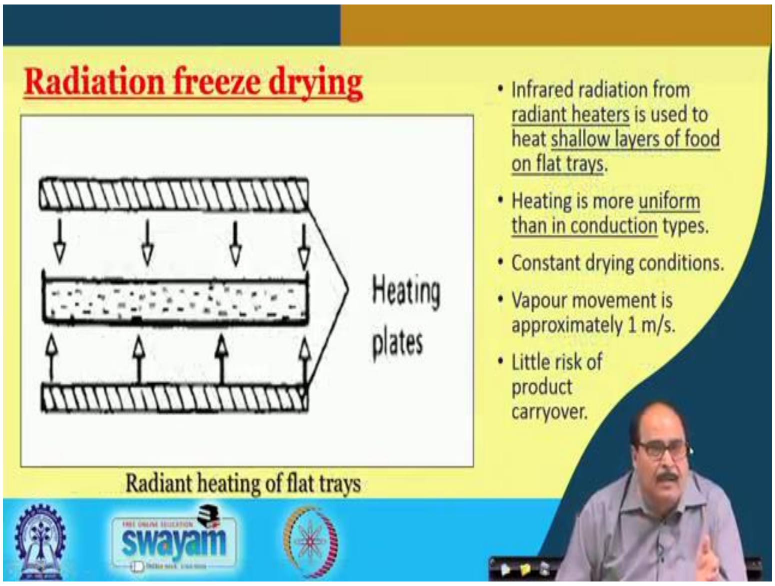
(a) **Contact (or conduction) freeze drying**

Food is placed onto ribbed trays which rest on heater plates with uneven contact. It dries the food more slowly as heat is transferred by conduction to only one side of the food. It is generally available in higher capacity.



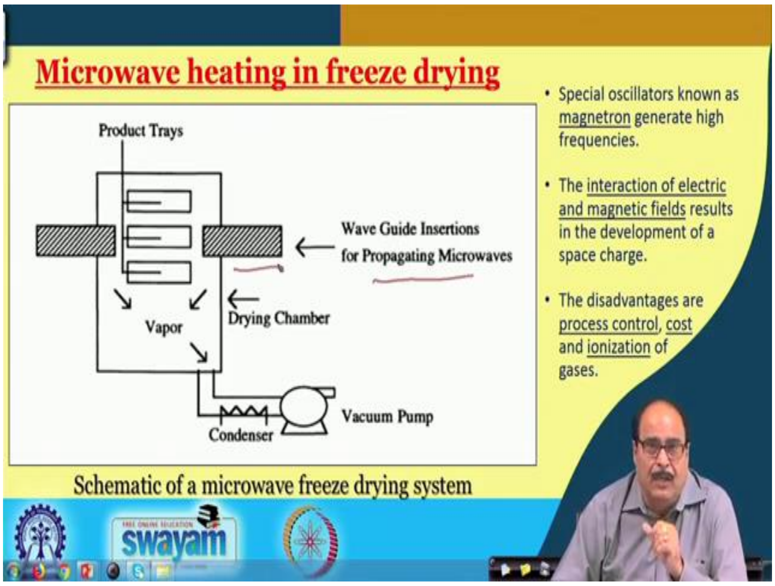
(b) **<u>Accelerated freeze drying</u>**

In this equipment, food is <u>held between two layers of expanded metal mesh</u> and subjected to a slight pressure on both sides. Rapid heat transfer occurs during the drying which ultimately cause reduction in drying times.



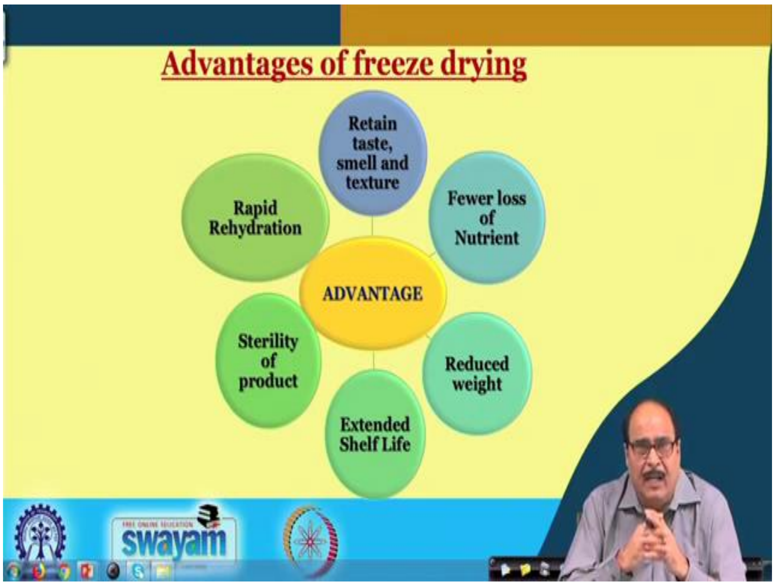
<u>(c) Radiation freeze drying</u>

In this method, infrared radiation from <u>radiant heaters</u> is used to heat <u>shallow layers of food on flat trays</u>. In this case, heating is more <u>uniform than in conduction</u> types and constant drying conditions are obtained. Vapour movement is approximately 1 m/s, so, it results in a fast drying. Also, there is little risk of product carryover in this process.



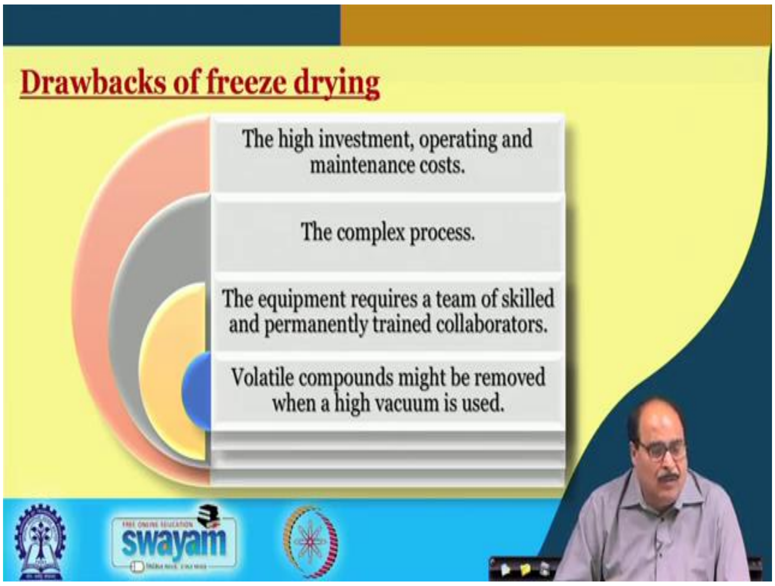
(d) Microwave heating in freeze drying

Like radiations, similarly the microwaves can be used to heat the foods during the freeze drying process. In this system, special oscillators known as magnetron generate high frequency microwaves. These microwaves with the help of waveguides are supplied to the vacuum chamber, which provide the heat energy or latent heat of sublimation to the product inside the vacuum chamber. The interaction of electric and magnetic fields results in the development of a space charge and the sublimation occurs. The disadvantages are process control, cost and ionization of gases. However, if suitable economic equipment is made available, this may be a comparatively much better process.



Advantages of freeze drying

(i) Retain taste, smell and texture
(ii) Fewer loss of Nutrient
(iii) Reduced weight
(iv) Extended Shelf Life
(v) Sterility of product
(vi) Rapid Rehydration



Drawbacks of freeze drying

(i) The high investment, operating and maintenance costs.
(ii) The complex process.
(iii) The equipment requires a team of skilled and permanently trained collaborators.
(iv) Volatile compounds might be removed when a high vacuum is used.