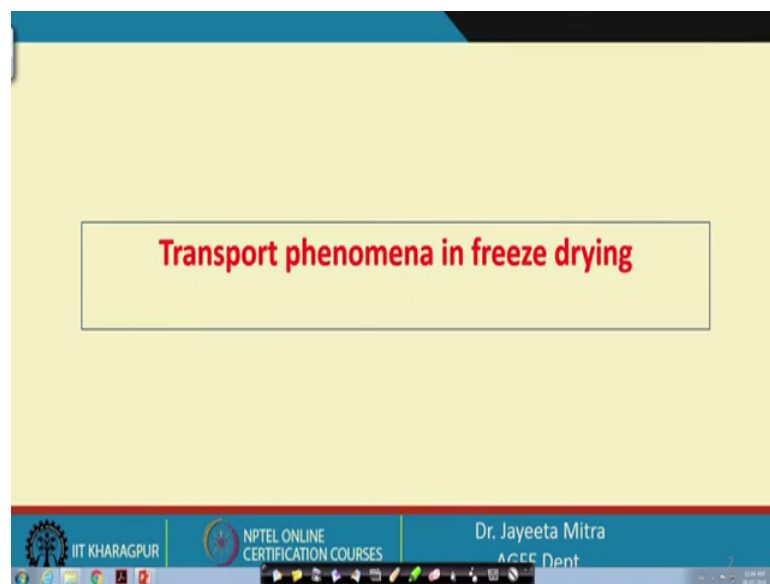


Fundamentals of Food Process Engineering
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Lecture - 34
Freezing & Freeze Drying (Contd.)

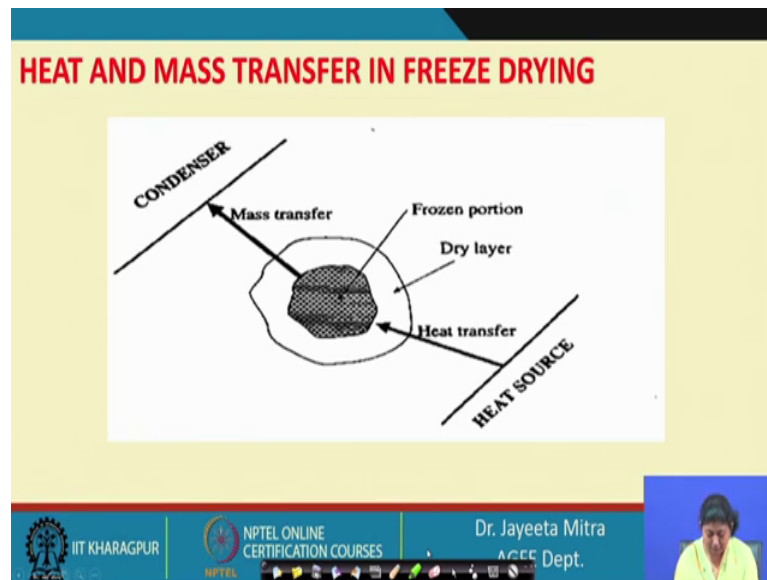
Hello everyone, welcome to the NPTEL online certification course on Fundamentals of Food Process Engineering. We will continue with the topic of Freeze Drying today.

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We have seen in the last class that what is the basic idea behind freeze drying that we need to perform the freeze drying below the condition of the triple point of water that is below 611.73 Pascal pressure and 0.01 degree Celsius temperature, in that condition we directly convert the ice crystal to vapor state, we convert them to vapor state and then we try to take out that moisture from the product. So, we will see how this you know transport of heat and mass that happening in case of a freeze drying.

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So, in case of freeze drying, let us say first we have a food sample and we first freeze it by lowering the temperature. So, all the moisture in the food become frozen, converted to ice and after that we will apply heat source we will apply heat to the material, so that as the heat will be transferred to the frozen layer, it will directly sublime under that reduce vacuum condition and that will be taken out by a vacuum pump.

Now, what happened that you know when this sublimation takes place that means, when we convert this ice to vapor at the reduced pressure, then specific volume of the that vapor will increase enormously. So, to take out all the vapors using a vacuum pump will require a very high capacity very large capacity of the pump right.

So, therefore, it has been you know design in such a way that this vapor will come in contact with a condenser and there it will again form the ice crystal and then it can be taken out from the condenser. So, this is how it happened. So, as the ice crystal will be sublime, there will be a dry layer ok and gradually this ice front will recede to the centre. It depends on the configuration and geometry where we keep this product ok. So, if the drying is taking place from one side or all the side or from top and bottom, so, based on the configuration of the freeze dryer, we can define you know dry layer section and the ice crystal for the frozen portion.

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Heat Transfer
The interface between the frozen zone and dry zone is - 'sublimation front'

(a) Heat transfer through frozen layer

Legend:
→ Mass transfer
→ Heat transfer
Dry layer
Frozen layer

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And we can see then the heat transfer first. So, what happens that the interface between the frozen zone and the dry zone which is called the sublimation front. So, first case we will look into this diagram, where the deeper line is of the heat transfer the deep black line and the lighter line is showing the mass transfer. So, heat transfer through the frozen layer that means heat is conducting to the frozen layers. So, here the conductivity of the frozen material will play the role as a parameter in the transfer of heat and then this we will go in to the dry layer and eventually the moisture otherwise will be sublimed directly, so that layers will eventually get dried.

So, this layer, this layer is eventually it was initially you know completely frozen completely frozen. So, as the heat is coming and it has it is going from this. So, mass transfer is taken from the top side. So, it is gradually the sublimation front is proceeding backward.

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Heat Transfer
The interface between the frozen zone and dry zone is - 'sublimation front'

(a) Heat transfer through frozen layer
(b) Heat transfer from hot surfaces through dry layer
(c) Heat generated in ice by microwaves

Legend:
→ Mass transfer
← Heat transfer
— Dry layer
▨ Frozen layer

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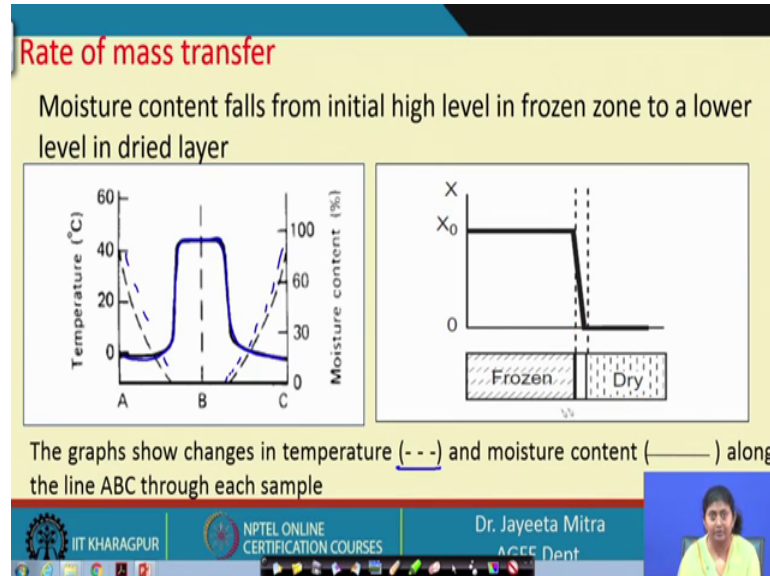
Now, next is heat transfer from hot surface through dry layer. So, if it happens that the mechanism is such that all the configuration in such that the product is getting heat from both the side ok. So, then what happened that as it is it is coming from the dry layer, so the conductivity again will be different for the dry layer. So, the rate of heat transfer from the source to the frozen layer will be different, rate transfer in the case in the first case and the second case will be different and then it is getting dried from both the side. So, eventually the time requirement will be actually half because from the both the side we are we are drying it. So, when we reach the centre, it will be dried from both the side ok, so that is why mass transfer is in the reverse direction of the heat transfer.

Now, in the third case heat generated in ice by microwave; so, heat generation if it by microwave, we know that microwave causes volumetric heat generation, internal heat generation because of dielectric property of the material, by here it is for the moisture. So, what will happen that internally the heat generation will be there; and because of that the mass transfer will be towards both the detection and of course, in this case it will be you know depending on the on the property depending on the dielectric property of the material, how much heat will be evolved in the internal section and that will govern the you know rate of heat transfer for sublimation.

So, in those cases, in all the cases actually the rate of heat transfer is very important because that will cause the sublimation and that will again govern by whether the heat is

coming through the frozen layer or through the dry layer or for the internal volumetric heat generation.

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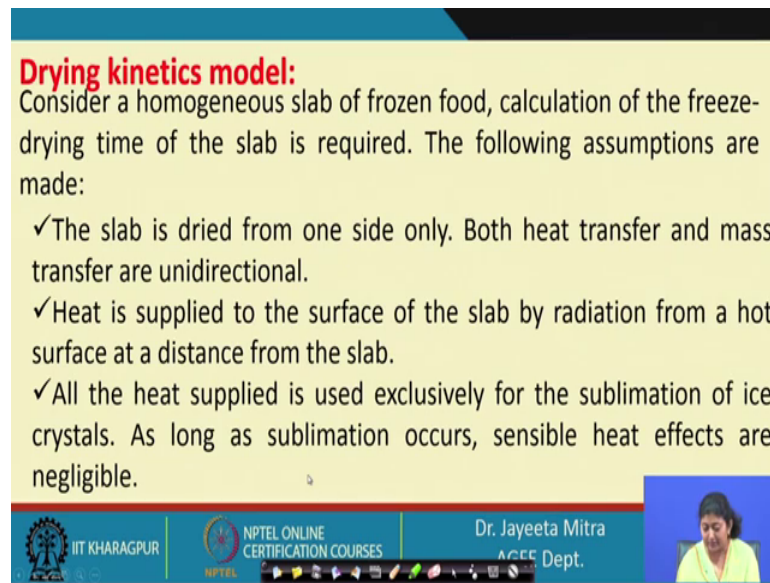


Now, rate of mass transfer, so moisture content falls from the initial high level in the frozen zone to the lower level in the dried layer ok. So, if we want to see here, the change in the temperature which is shown in the dotted line the change in the temperature and moisture content along the line. So, what will happen in this particular case, the temperature is reducing from both the side that means, we are providing heat from both the side if we consider this whole as a as a you know total slab of the material.

So, the dotted line since it is reducing from both the sides. It shows that the temperature or the heat is given to the material from both the side and it is coming through the dry layer, because the moisture profile is like this ok, so that means, so that means, the moisture in the in the centre is high. And at the surface it has been reduced, so that means, it is getting dried from both the layer and eventually when it reaches the central line that means, the drying front will completely merge from both the side and the product will be dried.

Now, if we look into this transfer from the frozen to the dry, it is kind of a step function, so the moisture content from very high to a very low value if it is suddenly dropped to that that condition. It is not kind of a gradual transition, so it is an average transition because of the sublimation.

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Drying kinetics model:
Consider a homogeneous slab of frozen food, calculation of the freeze-drying time of the slab is required. The following assumptions are made:

- ✓The slab is dried from one side only. Both heat transfer and mass transfer are unidirectional.
- ✓Heat is supplied to the surface of the slab by radiation from a hot surface at a distance from the slab.
- ✓All the heat supplied is used exclusively for the sublimation of ice crystals. As long as sublimation occurs, sensible heat effects are negligible.

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So, drying kinetics model; now, what we can do as most of the cases for this time for this kinetics development, we will consider we will try to consider a steady state case that means whatever heat we are providing to the frozen layer that will be utilized for sublimation of the ice front and we consider of course, no heat loss. And a homogeneous slab we have taken here of frozen food and we will calculate the freeze drying time of the slab.

So, our assumption will be the slab is dried from one side only. Both heat and mass transfer at unidirectional. Heat is supplied to the surface of the slab by radiation from a hot surface at a distance from the slab. All the heat supplied is used exclusively for the sublimation of ice crystal. As long as sublimation occurs, sensible heat effects are negligible.

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✓There is a sharp sublimation front between the totally iceless (dry) zone and the frozen zone.

✓The vapors are condensed on a cold surface (condenser) as ice.

✓The resistance of the chamber space between the slab surface and the condenser to mass transfer is negligible because of the high vacuum. Hence, the water vapor pressure at the condenser surface is nearly equal to that measured in the chamber.

Freezing
↓
Sublimation
↓
Removal of ice crystals.

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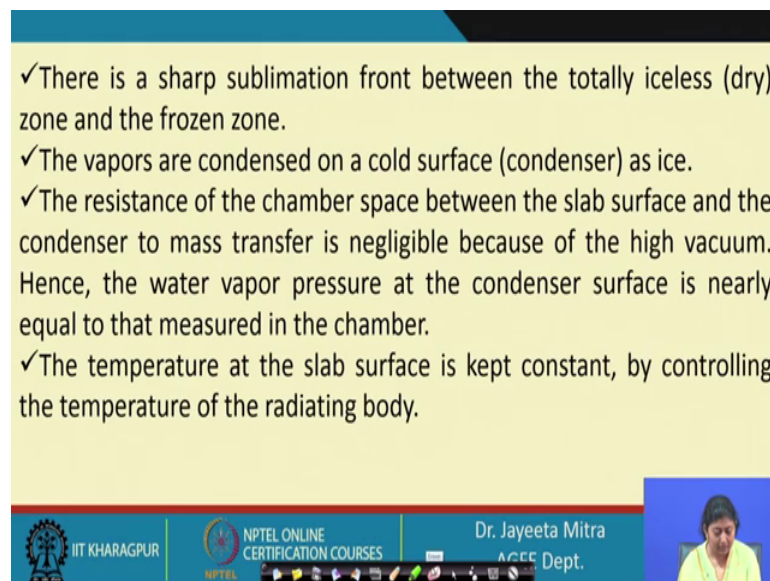
So, there is a sharp sublimation front between the totally iceless or dry zone and the frozen zone that we have already seen that these kind of a step function or there is a sharp sublimation front exist. And the vapor are condensed on a cold surface or condenser as ice. The resistance of the chamber space between the slab surface and the condenser to mass transfer is negligible because of the high vacuum. Hence, the water vapor pressure at the condenser surface is nearly equal to that measures in the chamber because there is a high vacuum and we will presume that it will be instantly get getting extracted from the chamber.

So, as we have seen that we can see here three distinct process will happen. First is freezing for formation of ice because of lowering of temperature ok. And after that sublimation because we keep it in vacuum and as we provide this as we provide the little heat it will directly sublime at that reduce pressure condition. And then what we need to have removal of that removal of ice crystals.

So, sometime it has been taken as the you know primary drying and secondary drying condition. So, here the whole process will complete unless we can remove, only sublimation I mean when we do sublimation as I have mentioned that because of very large amount of specific volume of that vapor at reduced pressure, it will it will be difficult to take out all that.

If we cannot through the very high capacity vacuum pump, therefore, that has been removed by using a condenser that has been first comes in contact with the condenser. They will form ice crystal and then that will be again removed. So, because of that vacuum we are considering that the resistance of the chamber space between the slab surface and the condenser to mass transfer is negligible. So, the water vapor pressure at the condenser surface is nearly equal to that measure in the chamber.

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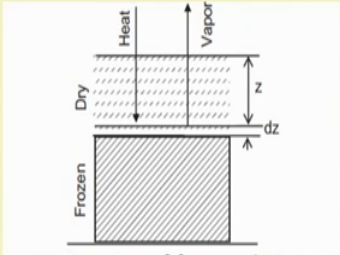
- ✓ There is a sharp sublimation front between the totally iceless (dry) zone and the frozen zone.
- ✓ The vapors are condensed on a cold surface (condenser) as ice.
- ✓ The resistance of the chamber space between the slab surface and the condenser to mass transfer is negligible because of the high vacuum. Hence, the water vapor pressure at the condenser surface is nearly equal to that measured in the chamber.
- ✓ The temperature at the slab surface is kept constant, by controlling the temperature of the radiating body.

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The temperature at the slab surface is kept constant by controlling the temperature of the radiating body.

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✓The temperature of the frozen product and hence of the sublimation front is constant.
✓The gas in the freeze-dryer chamber consists, practically, of water vapor only. The proportion of non-condensables (air) is negligible.



Kinetics of freeze drying.

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So, the temperature of the frozen product and hence the sublimation front is constant because we are making that constant by controlling the source of radiation. The gas in the freeze-dryer chamber consists, practically, of water vapor only because it is a very high vacuum. So, we are not expecting any other gases to be present there the proportion of non-condensable that is air that is negligible in that condition. So, what will happen that we are drying from one side only has we have mentioned.

So, it is it is coming to the dry heat is coming through the dry zone to the frozen zone and mass is transferred through that dry layer only. So, we can consider that with every instant some amount of distance we can I mean that that frozen layer can received some amount of distance very small amount of distance with a small time that will be the first basis of removal of the size.




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Assume that an incremental thickness dz of the slab is dried in a time increment dt . The rate of ice sublimation dw/dt is:


$$\frac{dw}{dt} = A\rho_i(w_i - w_f)\frac{dz}{dt}$$

A = area of the slab surface, m^2
 ρ_i = density of the frozen food, $kg \cdot m^{-3}$
 w_i, w_f = initial and final water content, respectively, $kg \cdot kg^{-1}$

At steady state, the rate of sublimation must be in accordance both with the rate of heat transfer to the sublimation front and with the rate of mass transfer from the sublimation front.



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So, we will consider that an incremental thickness dz of the slab is dried in time increment dt ok. So, we consider that very small amount dz amount of layer has been frozen within a time dt . So, we can write that what will be the amount of water removal per unit time. So, for that what we need we need the surface area across which this happened multiplied with dz by dt that is how much how much distance the ice front or the drying front is moving sublimation front is moving with time dt and we need to multiply with the density, so that we are getting kg in terms of kg of the water per unit time multiplied with the initial and final moisture content of the product.

So, then we are getting that how much amount of the you know product will be how much amount of moisture will be evaporated. So, because the density of the food multiplied with the moisture content and initial and final condition, so that will give us the amount of moisture evaporated in per unit time.

Now, this amount of moisture that needs to evaporate. So, therefore, we need to we need to multiply with this the latent heat of sublimation that is required, so that the amount of heat transfer needed can be calculated. So, at steady state, the rate of sublimation must be in accordance both with the rate of heat transfer to the sublimation front and with the rate of mass transfer from the sublimation front.

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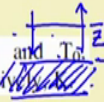
Let us consider heat transfer first. The rate of heat supply q (J/s) must be equal to the rate of sublimation multiplied by the latent heat of sublimation λ_s

$$q = A\rho_i(w_i - w_f)\lambda_s \frac{dz}{dt}$$

On the other hand, q is given by the rate of conductive transport from the slab surface to the sublimation front through the dry layer:

$$q = \frac{kA(T_0 - T_i)}{z}$$

where k = thermal conductivity of the dry layer, $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, and T_0 and T_i = temperature at the slab surface and at the sublimation front, respectively.



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So, let us consider the heat transfer first. The rate of heat supply q in joule per second must be equal to the rate of sublimation multiplied by the latent heat of sublimation. So, we are just multiplying with all the dw by dt factor that we have calculated in the last slide multiplied λ_s . q is given by rate of conductive heat transport rate of conductive transport from the slab surface to the sublimation front through the dry layer because we have seen that it is from the from one side only. So, here the dry here the frozen layer is that and this is the dry layer, so heat is coming through the dry layer and the mass is going out ok.

So, therefore, next is the heat conducted through this layer is the thickness of the layer is z , if the thickness of the layer is z through which the heat is coming. So, kA into initial temperature T_0 minus T_i by z where T_i is the temperature of the slab surface at the sublimation sorry T_0 and T_i temperature at the slab surface and at the sublimation front. So, T_i is at the sublimation front and T_0 is the initial condition. So, this we are getting now we need to equate this to find the time.

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Combining both equations and integrating from $z=0$ to $z=Z$, we get,

$$t = \frac{Z^2(w_i - w_f)}{2(T_0 - T_i)} \left[\frac{\rho_i \lambda_s}{k} \right]$$

Let us now consider mass transfer. At steady state, the rate of sublimation must be equal to the rate of removal of the vapors by mass transfer through the dry layer:

$$A \rho_i (w_i - w_f) \frac{dz}{dt} = \Pi A \frac{p_i - p_0}{z}$$

where Π = permeability of the dry layer to water vapor, $\text{kg} \cdot \text{s}^{-1} \cdot \text{m}^{-1} \cdot \text{Pa}^{-1}$
 p_i, p_0 = water vapor pressure at the sublimation front and at the slab surface, respectively, $\text{Pa} \cdot \text{s}$.

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So, for this equation, if we integrate from z equal to 0 at time t_0 to z equal to Z at time t , we can get t equal to Z square $w_i - w_f$ divided by $2(T_0 - T_i)$ into $\rho_i \lambda_s$ by k ok. So, now, if we consider the mass transfer at steady state, the rate of sublimation must be equal to the rate of removal of the vapors by mass transfer through the dry layer.

So, what will happen that mass transfer will cause because of the pressure differential partial pressure differential of the water in the ice front and in the dry layer or in the outside. So, for that this amount of mass transfer can be equated with capital $\pi A p_i - p_0$ by z where this capital π signifies here the permeability of the dry layer to water vapor. So, permeability on generally we express that in terms of diffusivity multiplied with the solubility. So, permeability express in $\text{kg per second per meter per pascal pressure differential}$. p_i and p_0 is the water vapor pressure at the sublimation front and the slab surface. So, p_i is at the sublimation front and p_0 is at the slab surface; that means, in the interface of the dry layer and the ambient condition for the vacuum condition ok.

So, from this the first one, the first equation where we have related the heat transfer with the amount of moisture removal and time. So, there we have found that t that is the time required for freeze drying has a linear relation with thickness z square ok, so that means, that if thickness varies significantly this will vary. And it also has an inverse relation

with conductivity and the temperature inverse relation with the temperature gradient and direct relation with the moisture differential in the in the slab surface and the frozen layer.

So, we from this we can get the idea that which parameter will going to affect the time in what way and accordingly we can optimize the freeze drying time. And in the second case, we have seen that what will be the permeability of the dry permeability of the dry layer to water vapor because whatever moisture will be evaporate that has to go through the dry layer to you know completely come out in the in the chamber and then from there to the condenser right.

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After integration we obtain:

$$t = \frac{Z^2(w_i - w_f) [\rho_i]}{2(p_i - p_0) [\Pi]}$$

An expression defining the conditions for steady state is given as follows:

$$\frac{p_i - p_0}{T_0 - T_i} = \frac{k}{\Pi \lambda_s} \Rightarrow T_0 = T_i + \frac{\Pi \lambda_s (p_i - p_0)}{k}$$

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So, here also after integrating we are getting this that t will be equal to Z square w i minus w f by 2 into p i minus p 0. Here also we are integrating with in z equal to 0 at t equal to 0 to z equal to Z at t equal to time t. So, here we are getting this factor multiplied with rho i by capital pi. So, an expression defining the condition for steady state is given as follows where p i minus p 0 by T 0 minus T i that will be equal to k by pi into lambda s.

So, this we are we can get this expression from both the equation of time where one once we relate the time from the heat transfer and in another case we are relating that time with the moisture permeation through the dry layer. So, with these two, we can get this expression. So, T 0 that is equal to T i plus capital pi into lambda s p i minus p 0 by

thermal conductivity of the dry layer ok. So, T_0 is the temperature at the surface of the slab and T_i is the temperature at the interface of the frozen layer for the sublimation front. λ_s is the latent heat of sublimation; p_i and p_0 as I have mentioned that the pressure at the sublimation front and partial pressure of the water vapor we should say partial pressure at the sublimation front and partial pressure in the chamber.

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The drying time (t) depends on :-

- ✓ Max permissible surface temperature (T_s) ($^{\circ}\text{C}$)
- ✓ Temperature at the sublimation front (T_i) ($^{\circ}\text{C}$)
- ✓ Initial and final moisture contents (W_i, W_f)
- ✓ Bulk density of the solids (ρ)
- ✓ Thickness of the slab (z)
- ✓ Thermal conductivity of the dry layer (k)
- ✓ inversely proportional to the permeability

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So, to summarize we can see that the drying time depends on maximum permissible surface temperature T_s , temperature at the sublimation front T_i . Initial and final moisture content W_i and W_f , bulk density of the solid, thickness of the slab and also the thermal conductivity of the dry layer. So, in this case it is dry layer because we have taken this assumption that it has passing through the dry layer only and it is inversely proportional to the permeability. So, we will stop here. And in the next class will continue with further explanation of the freeze drying equipments.

Thank you.