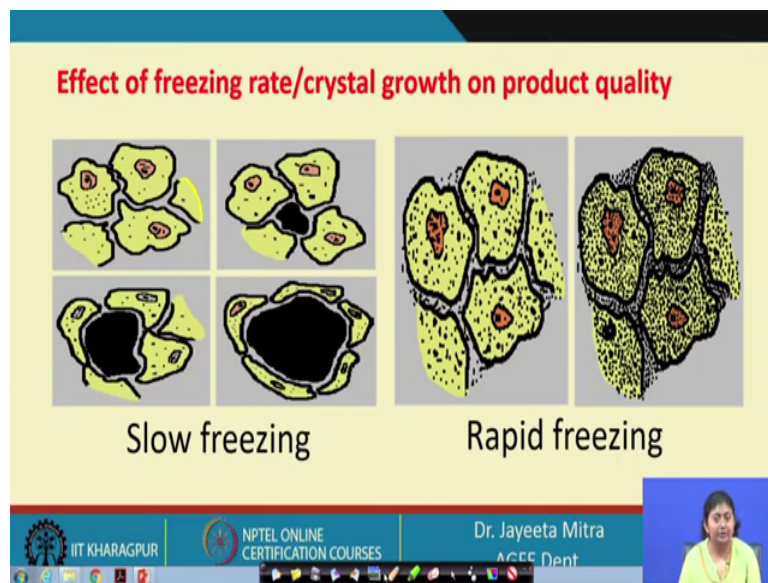


Fundamentals of Food Process Engineering
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Lecture – 33
Freezing and Freeze Drying (Contd.)

Hello everyone, welcome to the NPTEL online certification course on Fundamentals of Food Process Engineering. We will continue with the topic of Freezing and Freeze Drying, today. In the previous class we have discuss that how freezing takes place in case of a food material or in case of liquid water what are the mechanisms and what is the time required to calculate the total freezing time based on Planck's equation and Pham's method. So, today we will see that what are the changes, what are the quality changes takes place during freezing.

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So, effect of freezing rate or crystal growth on product quality. So, we have seen that the method by which we you know freeze the product is that first, the nucleation happens as we lower the temperature. So, nucleation happens and then the crystal growth starts ok. And the process at which I mean at the rate at which this process will takes place and that is again governed by the rate at which the heat removal takes place will have definite effect on the quality of the food.

So, if we see the structure of biological cells normally, we know that why we call freezing is a very effective method is because by freezing we can keep the nutritional quality almost intact, the flavor and colour to some extent we can preserve very well compared to the other preservation techniques, where we lower the water activity by heat treatment mechanism ok.

But the problem is that in the texture of the food. So, there may be some compromise we need to make because of freezing. So, how that happen actually? If we look into the structure of a biological cell let us say these are the cell and between the two cells the intercellular fluids are there

So, that fluid having the dilute concentration compared to the cellular material. So, when we lower the temperature then first that intercellular moisture will try to freeze out. So, the nucleation starts there first and then the I mean the further processing of the or further freezing will takes place. So, the osmotic pressure differential that you know present in the intercellular fluid and between the cell inner cell structure, that is maintained by the cell membrane or cell wall. So, that is called the targer of a cell ok. Now because of this pressure balance it the structure of the cell is intact.

Now, by anyway if we you know make some or cause some disbalance in the in the pressure differential, then what can happen? The moisture from the cell side will go to the intercellular space. So, that is another phenomena. So, during freezing these two mechanism can happen one is because of the pressure water vapor pressure fluctuation, the inter in a inner cellular moisture may come out to the intercellular spaces.

And another thing may happened that because of rate of freezing slow freezing or fast freezing, the different crystal growth formation takes place and that may cause you know breakage or destruction of the cell wall and for that also the structure or texture of the food will be hampered ok.

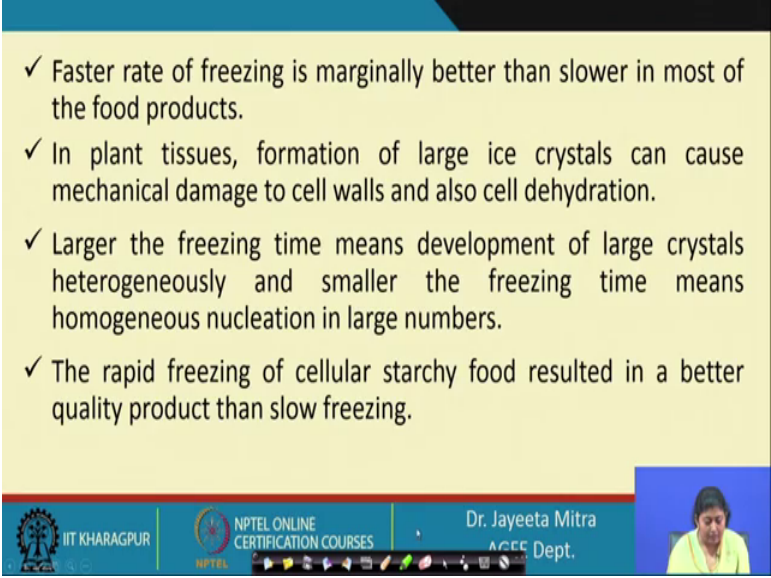
So, when this because of the freezing the moisture from the cell is comes out in the intercellular space, because the moisture in the intercellular space will freeze and the it will become more concentrated in the solute. So, that moisture cannot be regained during thawing.

So, if you want to reuse this you know frozen food and to want to consume it and we want to regain its initial structure by thawing. So, the moisture within the cell will not be regained. So, that is although that is not very much significant compared to the other drying mechanism because if we if we dry normally by heat treatment and then we go for rehydration for consumption of the food. So, then the structural damage is permanent most of the cases therefore, there is a gap we have found between the adsorption and desorption curve that we call hysteresis, we have discussed that in the water activity section

So, not that much damage we can expect in case of freezing, but some cellular structure damage may happen ok. And also if we perform the freezing at a faster rate that is the rapid freezing, then what happened that large number of nucleation happens in our more uniform or distributed pattern and there is not much large crystal growth at any point that can break or disturb the cell structure.

However if it is a slow freezing process, then what happened that the you know point of nucleation is very less and once it happens, because of slow freezing also the molecular mobility is less, but they try to adhere to the small nucleation that happens and that eventually get bigger and bigger ice crystal formation takes place and that will cause the distraction in the cell structure. Also they will break the structure and intercellular material can come out and all these things may happen. So, the structural damage may be there.

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- ✓ Faster rate of freezing is marginally better than slower in most of the food products.
- ✓ In plant tissues, formation of large ice crystals can cause mechanical damage to cell walls and also cell dehydration.
- ✓ Larger the freezing time means development of large crystals heterogeneously and smaller the freezing time means homogeneous nucleation in large numbers.
- ✓ The rapid freezing of cellular starchy food resulted in a better quality product than slow freezing.

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So, faster rate of freezing is marginally better than the slower in most of the food products. In plant tissues formation of large ice crystal can cause mechanical damage to the cell wall and also the cell dehydration. As I have mentioned that because the cell moisturize coming out to the intercellular spaces. So, cell dehydration will happen which is a permanent phenomena, we cannot gain it fully I am in regain the initial stage during the throwing process.

Larger the freezing time means development of large crystal heterogeneously and smaller the freezing time means homogenous nucleation in large numbers. The rapid freezing of cellular starchy food resulted in a better quality product than the slow freezing. And one more important thing is that because you know if this freezing thing happened that the structure if it have uniform effect during the freezing then throwing also will be uniform ok. So, that way also we should we should prefer the faster freezing mechanism.

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Physical Changes & Quality:

- ✓ During freezing most of the food products are susceptible to different deteriorative and enzymatic reactions which may affect shelf life.
- ✓ Weight loss in animal tissue happens as they lose moisture during the freezing process because their surface is exposed to heat and a moisture gradient exists within the environment.

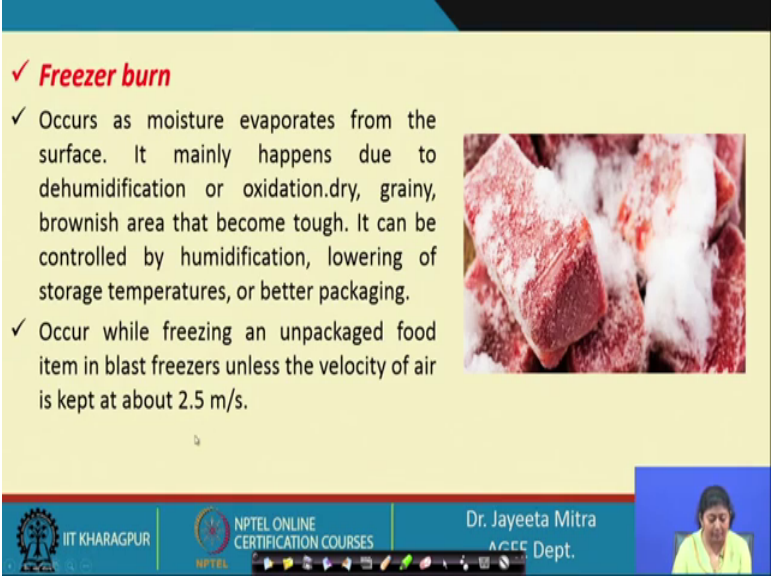
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So, physical change and quality if we discussed, so, during freezing most of the food products are susceptible to different deteriorative and enzymatic reaction, which may affect the shelf life. So, it is not that we are removing the moisture completely, we are freezing the moisture and thereby we are decreasing the mobility.

But we need to be very cautious that if any temperature fluctuation happens and the liquid will you know from the frozen stage to the liquid water conversion happens, then the quality or degradation may be there. And also the sometime it may happen that because all moisture in a food may not freeze ok.


As we lower the temperature because the water will become more concentrated in the solute present. So, it may happen that even if minus 40 degree Celsius also some amount of you know moisture remains and that has become concentrated in a particular solute or in enzymes and may happen that in very you know selective places some decorative changes can cause. Weight loss in animal tissue happens as they lose moisture during freezing process, because their surface is exposed to heat and moisture gradient exist within the environment.

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✓ **Freezer burn**

- ✓ Occurs as moisture evaporates from the surface. It mainly happens due to dehumidification or oxidation. dry, grainy, brownish area that become tough. It can be controlled by humidification, lowering of storage temperatures, or better packaging.
- ✓ Occur while freezing an unpackaged food item in blast freezers unless the velocity of air is kept at about 2.5 m/s.



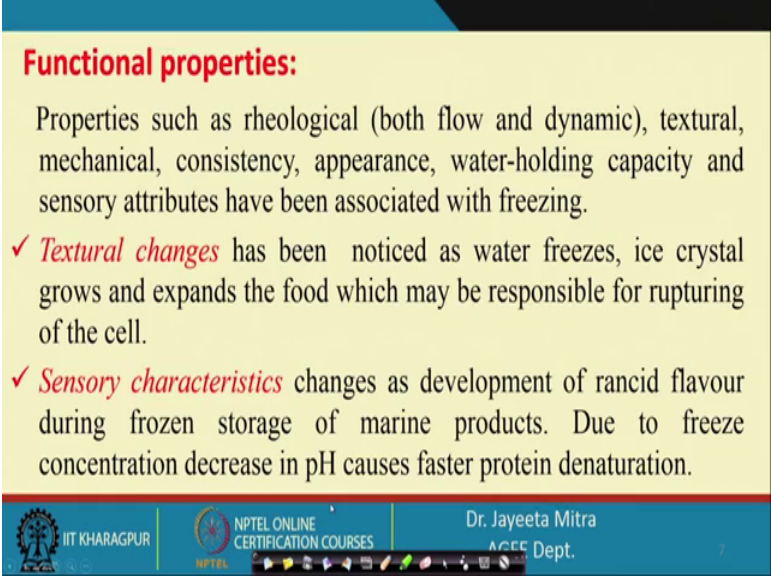
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So, this can happen and freezer burn. Freezer burn is an important phenomena which occurs as moisture evaporates from the surface. So, it mainly happens due to dehumidification or oxidation. Now, this we can see that freezer burn is may happen more commonly if we go for the freeze drying we will discuss the freeze drying later.

So, this will form a dry grainy brownish area that becomes tough eventually and it can be controlled by humidification or proper packaging and storage temperature. So, we can we can see this kind of phenomena if we expose the food to the blast freezer, unless the air velocity is escaped about 2.5 meter per second. So, if we keep the unpackage product and very high velocity be maintained, that time this kind of freezer burn may be observed.

So, this is not some change that will cause the food you know unconsumable, but only think the desirability will be slightly reduced as the colour formation is may not be like by the consumer.

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Functional properties:

Properties such as rheological (both flow and dynamic), textural, mechanical, consistency, appearance, water-holding capacity and sensory attributes have been associated with freezing.

- ✓ *Textural changes* has been noticed as water freezes, ice crystal grows and expands the food which may be responsible for rupturing of the cell.
- ✓ *Sensory characteristics* changes as development of rancid flavour during frozen storage of marine products. Due to freeze concentration decrease in pH causes faster protein denaturation.

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Certain functional properties also change because of freezing such as some rheological property that is floor property textural, mechanical, consistency appearance water holding capacity sensory attributes ok.

So, this all are associated all although most of the attributes are you know qualitative attributes can be properly managed with freezing phenomena. Textural changes has been noticed has water freezes ice crystal grows and expand the food which may be responsible for rupturing as we have mentioned in case of the slow freezing. Sensory characteristics changes as development of rancid flavor during frozen storage of marine products due to due to freeze concentration decreases in pH causes after protein denaturation. So, what happened that, if the moisture most of the moisture has been frozen.

So, sometimes the product is exposed to the ambient air or where the oxygen concentration will be higher and then some oxidative changes or may happen because if the if the fact portion is exposed to the oxidative environment. Also because of you know effect of the consent effect of the p H protein denaturation may happen. So, this all happened because of the change in the concentration these are required in a certain concentration for their activity, but if that concentration where is because of freezing because the moisture is you know freeze. So, is not available. So, therefore, this sensory undesirable sensory changes may be there.

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Chemical Changes & Quality:

- ✓ **Rancidity** develops due to the rapid acceleration of lipid oxidation in frozen fish.
- ✓ **Colour** also affected during frozen storage. The green color of vegetables is lost by chlorophyll degradation during freezing and frozen storage. In poultry carcasses with rapid surface freezing, which generates a smooth chalky white surface.
- ✓ **Flavour and aroma loss** for frozen strawberries as a result of rapid degradation of esters.
- ✓ The destruction of **vitamin C** (ascorbic acid) occurs during freezing and frozen storage.

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So, rancidity develops due to the rapid acceleration of lipid oxidation in frozen fish, colour affected during frozen storage the green colour of vegetable is lost by chlorophyll degradation during freezing and frozen storage and in poultry carcasses with rapid surface freezing which generates a smooth chalky white surface. Flavor and aroma loss for frozen strawberry as a result of rapid degradation of esters and the destruction of vitamin c occurs during freezing at frozen storage. So, vitamin c is very sensitive to temperature change that therefore, at low temperature also it is getting destroyed.

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THE FREEZE DRYING PROCESS

The diagram illustrates the four stages of the freeze-drying process:

- COOKING:** Ingredients and water are added to a pot.
- FREEZING:** The mixture is frozen, forming ice crystals.
- DRYING:** The frozen mixture is placed in a chamber where water vapor is removed.
- REHYDRATING:** The dried mixture is rehydrated with water.

FREEZE DRYING

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Now, next the important thing that we need to study is freeze drying. Till now we have discussed about freezing. So, in the freezing what we are doing we are not removing the water, but we are decreasing the mobility of the water by freezing it. So, the free availability of the water is not there. So, the contamination microbial contamination or enzymatic activity or qualitative degradation will be reduced to some extent. But in case of freezing we need to maintain the condition throughout the low temperature condition throughout that is why we need the cold chain arrangement.

Because if any fluctuation of temperature happens, then the liquid may convert from ice stage to the liquid water and then the deterioration reaction will start. Now when we say the freeze drying the freeze drying involves some a set of you know steps, where we freeze the liquid water to ice first and then we try to remove it from the food material.


So, that the moisture is not available there and then if any time we want to we want to consume it is a dry product we can then you know use it and the quality and the quality of the product the colour flavor everything is intact and very good.

Now, freeze drying mostly because it is very costly method. So, it has been mostly used in the pharmaceutical or biotechnology sector, for antibiotics and all those application, but after 1950 freeze drying has been used in a significant amount for the food material also. So, we will see that what are the steps of freeze drying, how we can improve the quality, what is the mechanism and what kind of dryers available and also the heat and mass transfer involve with the process.

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Contents:

- Fundamentals of freeze drying
- Transport phenomena in freeze drying
- Different types of freeze dryer
- Freeze drying related process
- Assignments



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So, first we will see all such contents we will cover one by one; and here we can see that freeze dried products looks very nice and the colour flavor texture is very much intact and we are getting very good porous structure in the freeze drying. The structured is not collapse like all other draying methods.

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Freeze drying

- ✓ Freeze drying also termed as **"Lyophilization"**
- ✓ The product is dried by direct sublimation of the ice under reduced pressure.
- ✓ Typically to a final moisture content of 1–4%.

Freeze Drying - How does it work?



1. Fresh or cooked foods are flash frozen then placed in a vacuum chamber

2. About 90% of the food's moisture is drawn off by evaporating the ice at temperatures as low as -50°C

3. The freeze - dried food is sealed in moisture-and-oxygen proof packaging to ensure freshness until opened.

4. When the water is replaced the food regains its original fresh flavour, aroma, texture and appearance.

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So, what is the fundamental concept of freeze drying or you also call it Lyophilization? The product is dried by direct sublimation of the ice crystal under reduced pressure. So, sublimation we know that by sublimation we convert the ice to vapor directly not the

intermediate freeze that is liquid phase is coming in this case and we want to dry it to final moisture content 1 to 4 percent with basis. So, what we freeze drying normally, the fresh fruits and vegetable muscle food like prawn, fish then meat product.

So, all such products fresh or cooked food these are frozen and placed in a vacuum chamber. So, we are freezing first and then we are keeping it in the vacuum chamber, then about 90 percent of the food moisture is drawn off by evaporating the ice at the temperature as low as minus 50 degree Celsius.

And after freeze drying we will we will pack the dry food in a proper package and then seal them will select the package in such a way. So, that the moisture formation or gas formation will be less and then when the water is replaced the food again its original fresh flavor aroma texture and appearance. That is after rehydration we can use them and we will get the most of the initial quality of the sample intact.

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Conventional drying	Freeze drying
The food material 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 <i>101.325 kPa.</i>	Reduced pressures (27–133 Pa)
Movement of solutes and sometimes case hardening	Minimal solute movement
Stresses in solid foods cause cell rupture and shrinkage	Minimal structural changes or shrinkage
Solid or porous dried particles often having a higher density than the original food	Porous dried particles having a lower density than original food
Odour and flavour frequently abnormal, reduced nutritional value	Odour and flavour usually normal, nutritional value retained

So, roughly if we compare the benefits of freeze drying over the conventional drying what we can get in the conventional drying? The food material is exposed to a continuous flow of hot stream of air where the moisture is evaporates. So, we expose them to a continuous flow of hot stream where in case of freeze drying removal ice or frozen solvent from a material through sublimation.

Then the temperature ranges from 37 to 93 degree Celsius in case of conventional drying and in case of freeze drying below freezing point we need to keep the temperature. Here we perform atmospheric pressure drying in case of conventional and we need reduce pressure environment for freeze drying.

So, here it is 1.325 kilo Pascal and here we can see 27 to 133 Pascal. Movement of solutes and sometime case hardening this happened; that means, movement of the solute is there because mobility of the liquid moisture is there and sometime case hardening happened when the surface gets dry quickly.

And then this kind of phenomena which is an undesirable changes can cause in case of the conventional drying and minimal solute movement we are getting in case of freeze drying. Stresses in solid food cause cell rupture and shrinkage and minimal structural changes or shrinkage happen in case of freeze drying.

So, solid or porous dried particle of an having a higher density, than the original food. Solid or product dried particle often have a higher density because of volume will reduce to a significant extent and here in case of freeze drying, porous dried particle having a lower density than the original food is normally observed.

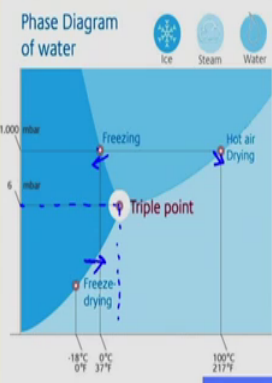
In case of conventional odour and flavor frequently abnormal reduce nutritional value we will get because all such happened because of the high temperature treatment that will cause the denaturation and reduction in the heat sensitive components. And here in case of freeze drying the odour flavor usually normal and nutritional value retained

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Thermodynamics behind freeze drying:

- ✓ Direct transition from solid to gaseous state without going through a liquid stage
- ✓ Can occur only if the vapour pressure and temperature are below the triple point of water (below 611.73 Pa & 0.01°C)

SOLID $\xrightarrow{\text{Sublimation}}$ **GAS**
 $\xleftarrow{\text{Deposition}}$



The phase diagram of water shows pressure on the y-axis (1,000 mbars, 6 mbars) and temperature on the x-axis (-18°C , 0°C , 100°C). The triple point is at 0.01°C and 611.73 Pa . The regions are labeled Ice, Steam, and Water. The processes shown are Freezing, Hot air Drying, and Freeze-drying.

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So, now we will see that how or what is the principle of this freeze drying. Here we can see the phase diagram of water. So, there we can see the all three phases solid liquid and gas solid liquid and gas this three phases are there and triple point which is the point where all three phases coexist right. So, we need to perform the operation somewhere below this point so, that we can directly convert ice to vapor.

So, by now we have learn all three methods, such as hot air drying that we have discussed where liquid to vapor transfer we have done. So, we are providing latent heat of vaporization and a moisture is coming out. Then we have seen the freezing phenomena where first sensible heat reduction and then latent heat of fusion that we have extracted or removed from the water and in the in this case sublimation ok.

So, here directly from the ice to vapor, so, here; obviously, we need to provide much larger amount of you know heat so, that it can be directly sublimed. So, direct transition from the solid to gaseous state without going to the liquid stage and this can occur only if the vapor pressure and temperature are below the triple point of water that is, below 611.73 Pascal and $0.01 \text{ degree Celsius}$.

So, here is the here is the triple point this is a pressure we need to perform below that and also the temperature. So, we need to perform below this point. So, freezing is at normally zero degree also it for the pure water sometime we have observed that super cooling will be there, which is a meta stable state. But in this case for sublimation we need to go

below 0.01 degree Celsius and 611.73 Pascal. So, which is the lower pressure and temperature condition compared to the triple point ok.

So, sublimation is the process of solid to gas transfer and deposition is gas to solid transfer. So, in case of freeze drying, what we do? First we freeze all the moisture in the product under the low temperature situation then we keep it in inside of vacuum and we will try to remove all the ice crystal formed in the in the food sample.

So, this is how we perform the freeze drying when then we will go to detail of this process and the equation involved for the heat and mass transfer. So, we will stop here and we will move on to the next lecture.

Thank you.