Industrial Biotechnology
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Module 5
Lecture No 25
Downstream Processing Evaporator, Crystallizer

Welcome back to my course Industrial Biotechnology, today I shall continue my lecture on downstream processing and last class as you remember that we concentrated on solid- liquid separation process. And we have basically that these process are common to most of the chemical and biochemical industries and it has different names as for example, that I work with citric acid industry and during after the citric acid production we have so many different solid liquid separation process. As for example, initially we use the rotary vacuum filter to separate the mycelia or in the cell mass that present in the fermentation broth, after that we precipitated this citric acid in the form of calcium citrate and this calcium citrate we separated in a special another filter what you call pannevis filter.

And after that this calcium citrate we hydrolyse with concentrated H_2SO_4 and it produce citric acid and calcium sulphate and which is called gypsum and this is again passed through the another filter what you call gypsum filter, after that we pass this citric acid solution through the evaporator to concentrate the citric acid concentration to increase the citric acid concentration from 22 percent to 60 percent and then we passes through the crystallizer where solid crystals of citric acid is separated from the liquid. And then again it passes through the centrifuge to separate the solid crystals from the liquid, so we can see that lot of solid liquid separation process are there

And basically in the last class I discussed about rotary vacuum filter and also I discuss about the about the plate and frame filter press. Now, the whenever we use the any kind of filtration process that again it depends on the size of the particle that we are going to separate. As for example, the filtration process that is used for the bacteria may not be suitable may not be used for the yeast cells, actually Baker's yeast fermentation process we use the plate and frame filter press and for fungal cell mycelium separation we use the rotary vacuum filter.

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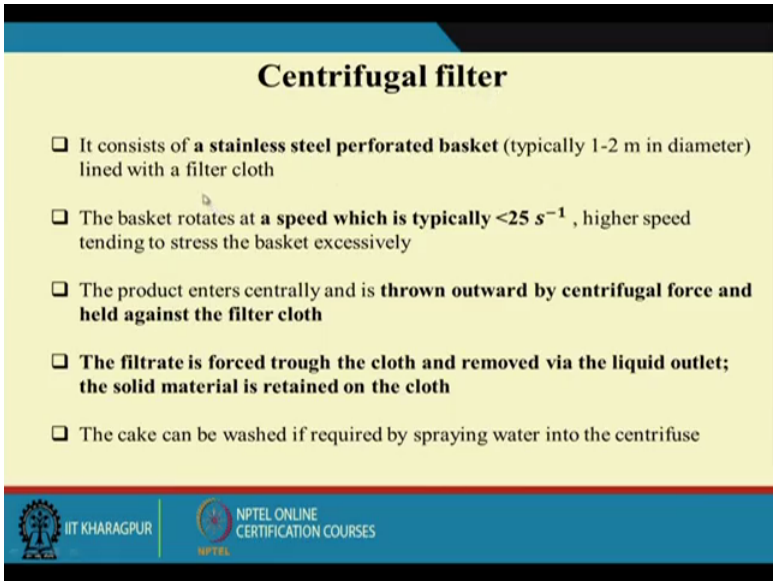

So this way I discussed in the last class now today another solid-liquid separation process is left that is centrifugation that I shall talk about, and then also I shall discuss about other downstream processing that we have. Now let me first discuss about the centrifugal filter, it consists of a stainless steel perforated basket typically 1 to 2 metre in diameter lined with filter cloth basically, we take the material inside the centrifuge and we rotate at the high speed. The solid material that throw to the surface where your filter cloth is there, so it is accumulated on the surface I shall show you and then liquid will comes out because this is perforated so liquid will go out of the system.

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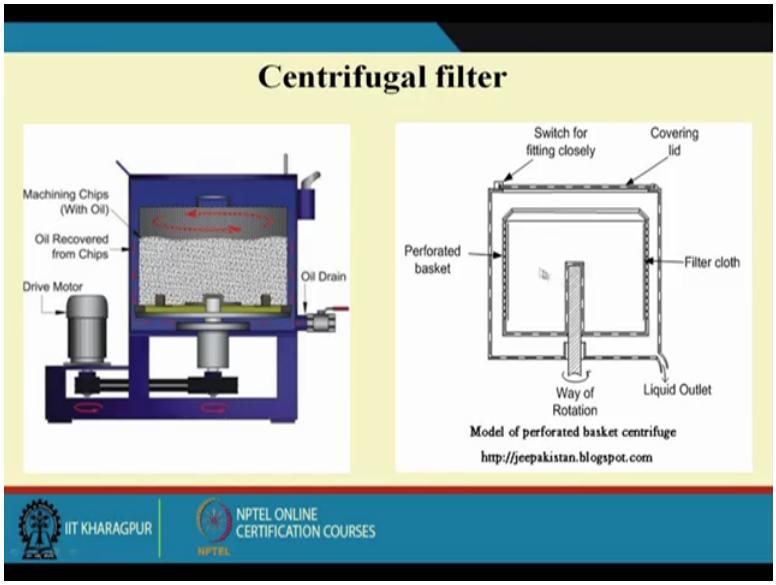

The basket is rotate at the speed of 25 per second high speed tending to stress the basket accessibly the product enters centrally and throw outwards by centrifugal force and held against the filter cloth, the filtrate is (())(04:28) of cloths and remove the liquid outlet, the solid material retain in the cloth. Let me show you this, this is this is actually the centrifuge where you take your that material and when you when you rotate it very high speed here then this is the perforated basket this is this is embedded with filter cloth, so when the solid material is strike to the surface and liquid will comes out through this perforated basket and liquid will going out and then when it is accumulated in take the material out and and make the take the filter cloth out and separate the separate the solid material.

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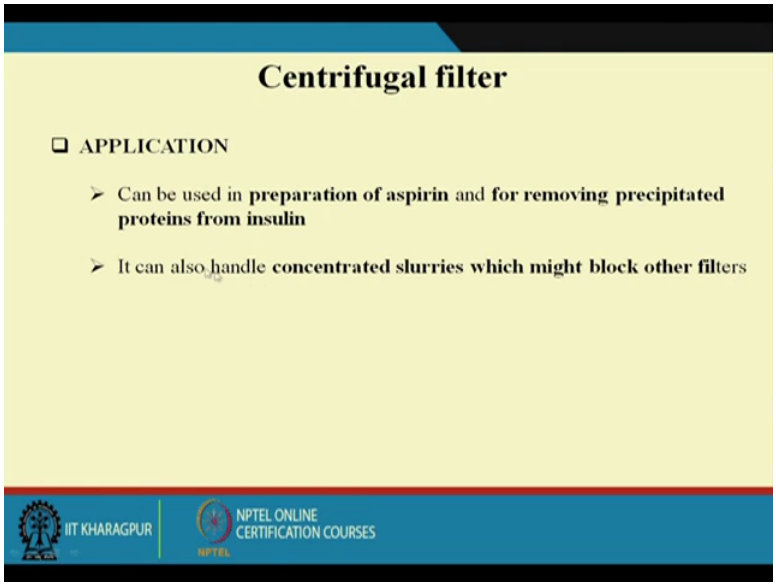

So it can be used in preparation of aspirin because in the pharmaceutical industry we have aspirin and from the removal of precipitated protein from insulin, as you know the insulin is kind of recombinant protein we usually produce through the fermentation process and it can also handle the concentrated slurry which might block other filters.

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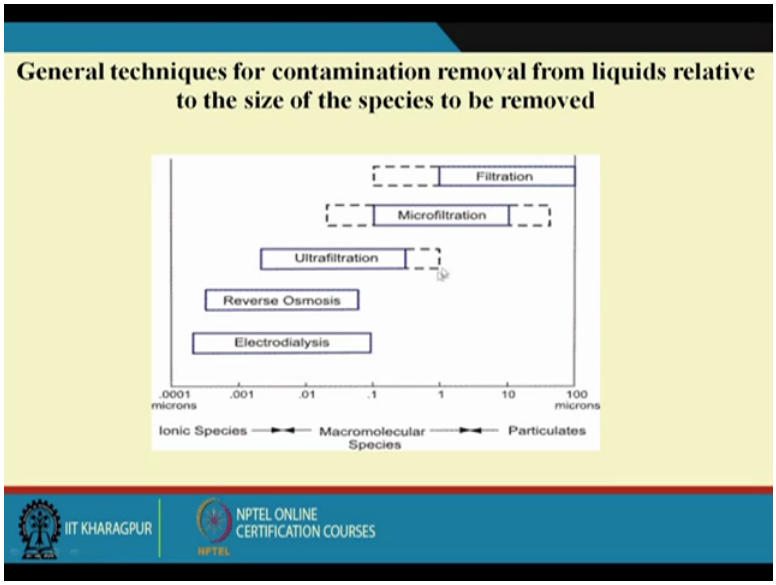

Now the different types of filtration techniques we have depending on the size of the filter as I mentioned that we have filtration that is usually size varies from 1 to 100 microns then micro filter this again take care the smaller particle alter filtration again is much smaller particles reverse osmosis will be more smaller particles and electro dialysis is more smaller particles, so this is ionic splitting this is macro molecules and this is particulates.

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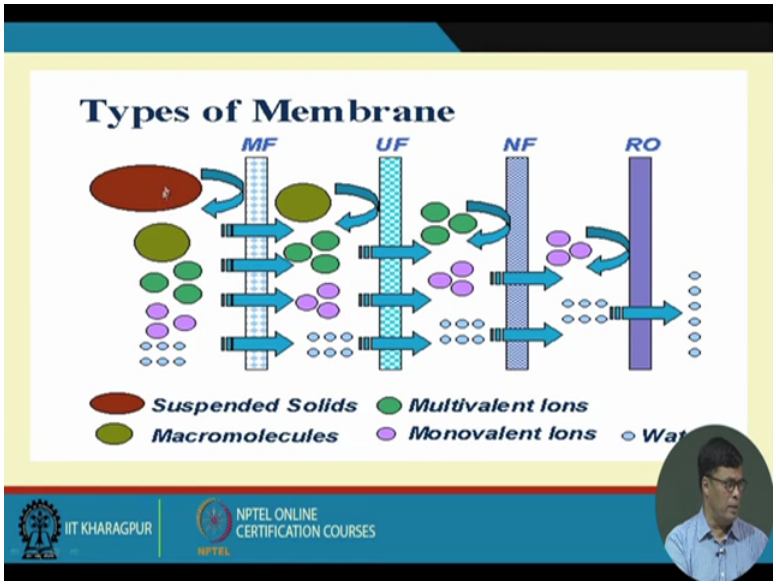

Now it is picturally it can be explained like this suppose your mixture is comprise of different types of particles the the membrane filter then ultra filtration, nano filter then reverse osmosis, you can see how the particle smaller particles becoming separated from the mixture.

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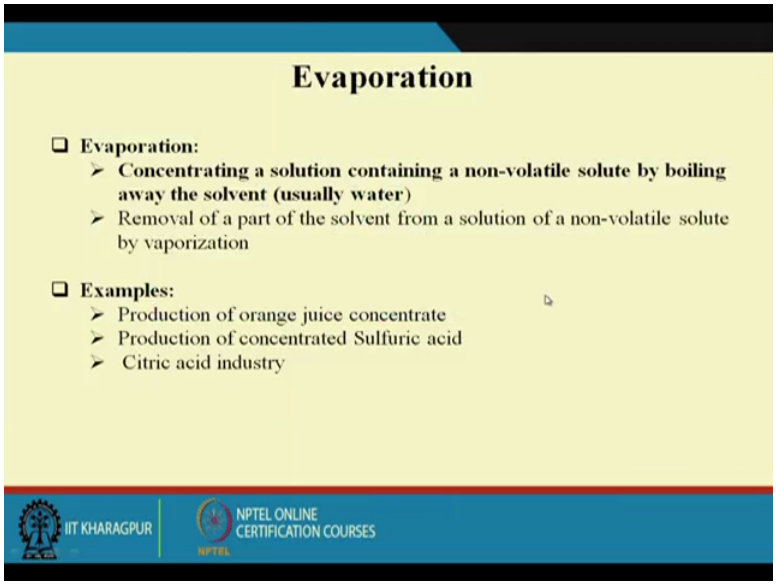

Now let me discuss about another another downstream processing that is largely used by the industry one is called evaporation, evaporation is nothing but concentration a solution containing non volatile solid by boiling away the solvents, just now I talking about citric acid concentration of the citric acid mixture from 22 percent to 60 percent now question comes how you how you do in the industry the what we have?

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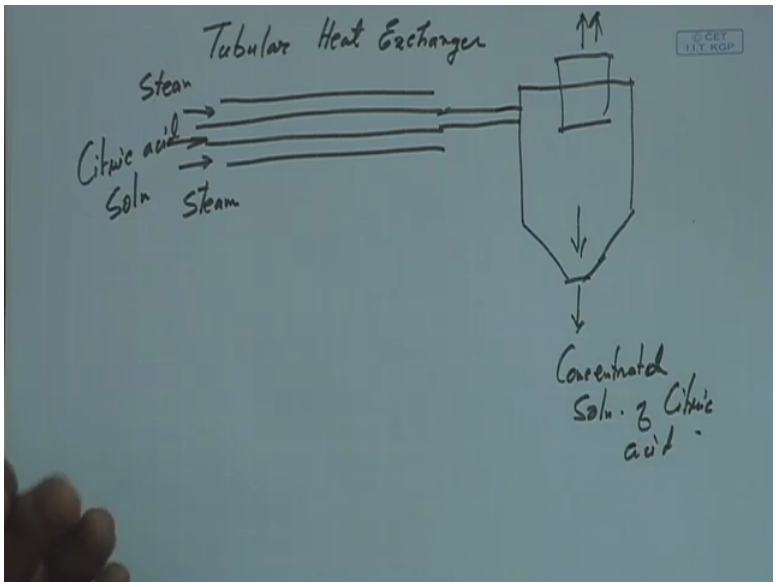

We have tubular heat exchanger this is called tubular heat exchanger, suppose we pass our that you know citric acid solution here citric acid solution and here we pass the steam, let us assume this is the steam angular space we have the steam this is steam. So when we pass together what will happen this citric acid solution will be heated and then what do you what

do you have, we throw it in the some kind of big chambers throw it like this and here when you throw this liquid here then what will happen that you know that vapour will go out like this and concentrate material liquid the concentrated liquid will comes concentrated solution of citric acid will comes out from the bottom.

So this is how the evaporation is required that is why I told you this is concentrating a solution containing non-volatile solute because we know we know that two types of solutes are there one is called volatile another is non-volatile. Non-volatile solute means that if you keep the suppose I make I can give a very simple example, we have non-volatile fatty acid, we have volatile fatty acid, now in case of volatile fatty acid like acetic acid butyric acid and all these things if you keep it in a beaker after some time you will find the everything will be evaporated out, that is why nothing will remain in the beaker so all are we call it volatile fatty acid

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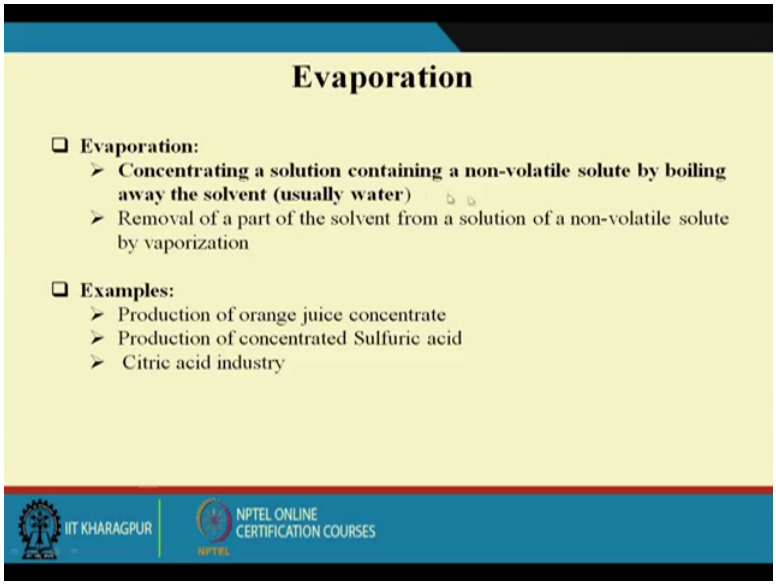

Now non volatile fatty acid means what, if you keep the as per example if you keep the citric acid in the solution then citric acid is the non volatile fatty acid so what will happen the water will be evaporated out and after some times the solid crystals of citric acid remain in the beaker, so this is how we differentiate the non volatile solid from the volatile solid. So this is evaporation basically used for concentrating the non volatile solute by boiling away the solvent and removal the part of solvent from the solution of non volatile by the vaporization. Examples are production of orange juice concentrate, production of concentrated sulphuric acid and citric acid industry.

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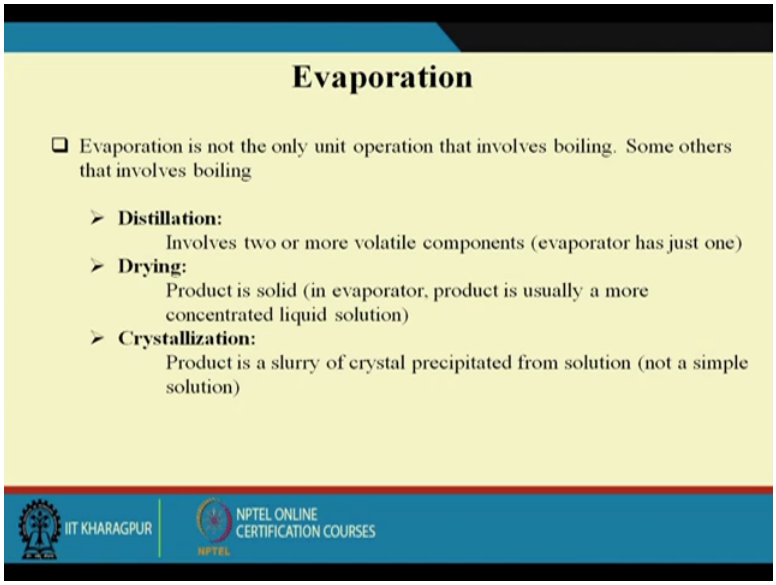

Now evaporation can be used for the different purpose, one is the distillation involved two or more volatile components there we can use that and then drying process where product is solid in evaporation product usually more concentrated in the liquid form and crystallization product in slurry crystals precipitated from the solution.

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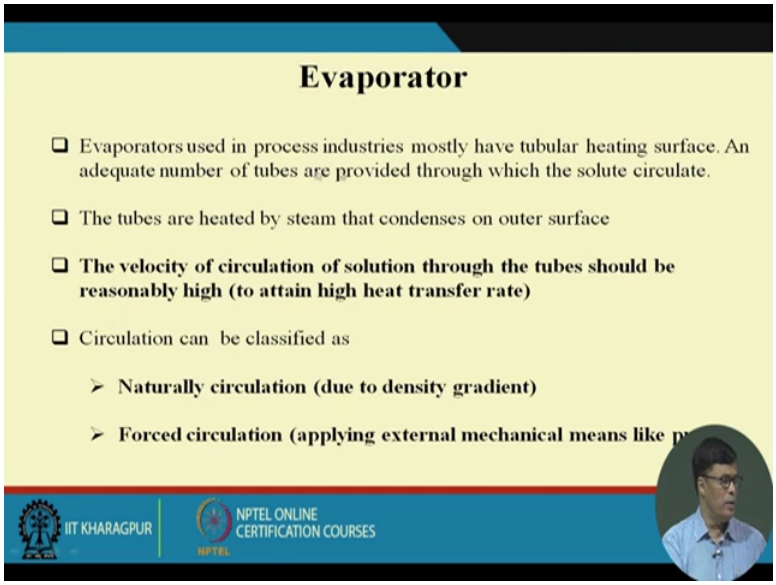

So evaporator used in the process industry, different types of evaporator, the velocity or circulation through the tube should be reasonably high to attain the high heat transfer rate. Circulation can be classified as natural circulation due to the density gradients and forced circulation applying the external mechanical means like pumps.

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So type of evaporators we have different we have that that tubular evaporator maybe long tube, vertical evaporator, upward flow and flow and downward flow another is forced circulation, another evaporator we call it agitated film evaporator.

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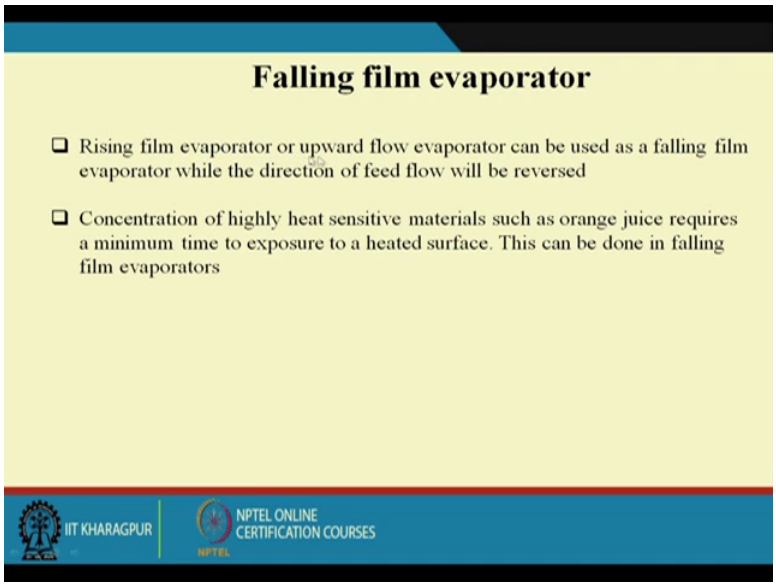

Now falling film evaporator rising film evaporator or upward flow evaporator can be used as a falling film evaporator while the direction of the feed will be reversed because it is flowing up and you know that concentrated liquid will go down. The concentration of highly heat sensitive material such as orange juice requires a minimum time to expose to a heat surface, this can be done falling film evaporator, then we have four circulation evaporator this is used for high viscous liquid and liquid containing the suspended particles.

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We have agitated fixed film evaporator modify film, falling film evaporator with single jacketed tubes containing a internal agitator. Application is that if the liquid is viscous, its viscosity is very high then this can be used high capital cost and high maintenance cost is required in this.

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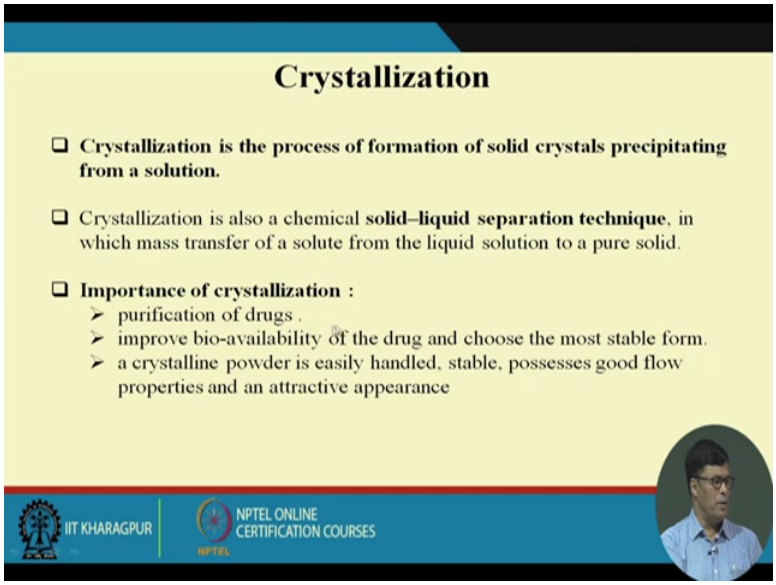

Now crystallization this is all about the evaporator, now I told you that the crystallization is the process of formation of solid crystal precipitating from the solution, crystals is also a chemical solid liquid separation techniques in which the mass transfer of the solid from the liquid solution to a pure solid.

Importance of crystallization is purification of drug, improvement of improve bioactive bioavailability of drug choose the most stable form and a crystalline powder easily handle stable and possesses good flow properties and attractive appearance. So you know that this is the a crystallization so there are different examples we have in the crystallization process I can give a typical example of this sugar production. In the sugar production is usually can be done in two different sources one is cane from the cane and beet.

Beet is largely used in the western countries and cane is used mostly the county like India and Brazil. Now how we separate the separate this sugar from the cane juice, cane juice usually concentrate through the process of evaporation after that we reduce the temperature so the crystals of sugar formation takes place and then with the help of centrifuge we separate the crystals of sugar from the liquid and liquid we call it cane molasses and sugar crystals we wash with water and dry it and sell it in market.

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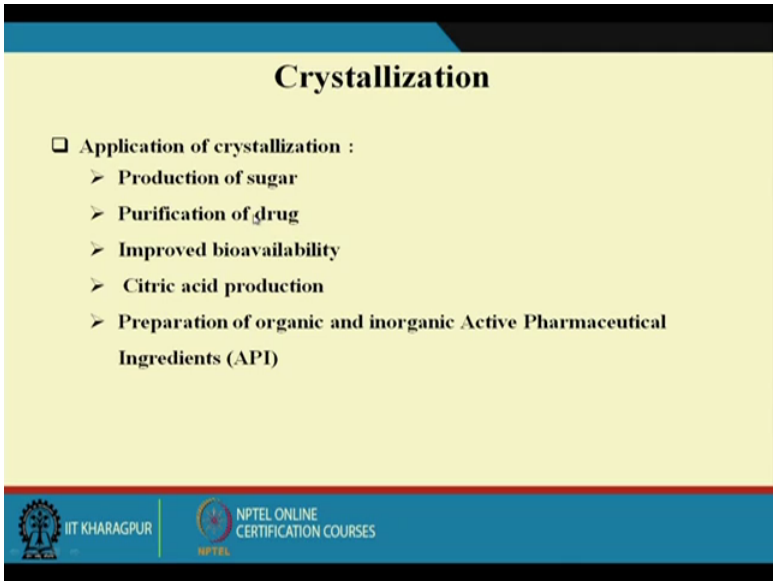

So one is largely used in the production of sugar, then we have purification of drug it is largely used then improvement of improved bioavailability citric acid production I just discussed that and preparation of organic and inorganic inactive pharmaceutical ingredients. In a pharmaceutical industry it is (())(15:04) as for example it is largely used.

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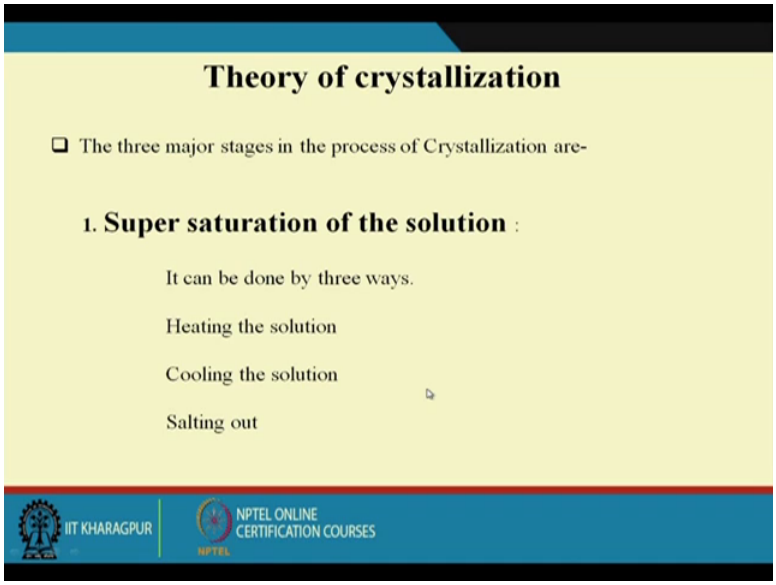

Now theory of crystallization is very interesting, how the crystal formation takes place, the first we shall have to make the super saturation because if we it is under saturation, then crystallization will not take place, if it is super saturation then and only then crystallization will take place. It can be done in three ways heating the solution, cooling the solution and salting out. The first we heating the solution what is the purpose of heating heating just concentrate the liquid and then cooling it and I can give a simple example I was talking about citric acid fermenter citric acid separation process.

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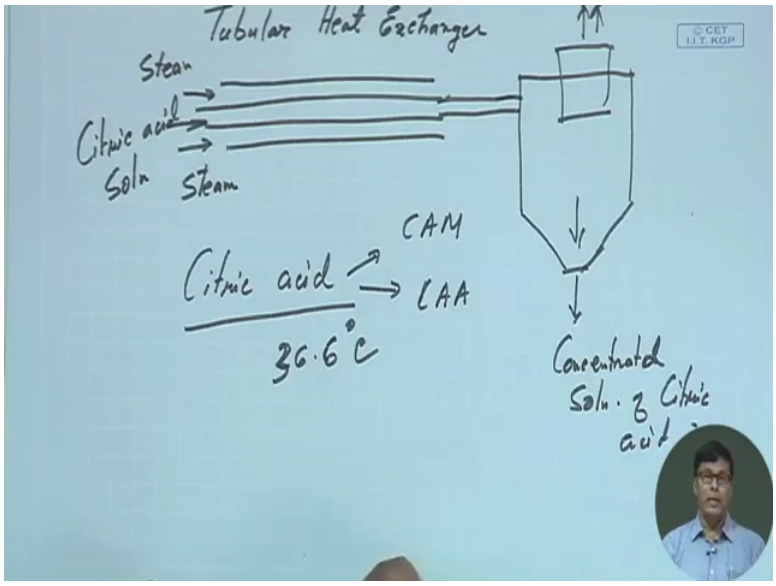

And citric acid has a transient temperature 26.6 degree centigrade sorry 36.6 degree centigrade is the transient temperature, what does it mean that at above 36.6 degree

centigrade it citric acid is available in two different forms one is called citric acid monomer and another is citric acid anhydrous. So what happens that if the temperature is more than 36.6 degree centigrade we get citric acid anhydrous and in case of citric if the temperature we keep less than 36.6 degree centigrade, we get CAM.

So usually at low temperature crystallization more crystal formation take place so what we prefer we prefer that we produce more crystal that is why we usually initial crystallization we take place with the help of at low temperature so that we can produce the CAM and later on CAM we can convert it to citric acid anhydrous though by keeping the temperature of the mother liquor which containing about 30 percent of citric acid at 40 degree centigrade, so this is how we can do that so this is separation of the crystals we can say it is salting out then then then how the crystal formation takes place this is very interesting.

The atoms or molecules or ions come closer to one another and form aggregate called cluster, so atoms they come to each other and form the cluster and these clusters will combine to form embryo and in this stage only the lattice formation begins and this embryos combines to form nuclei this nuclei crystal from nuclei crystals are formed, so these are the different steps involved in case of crystallization process. First we have segregated molecules they slow-slow they come closure and they form the cluster from the cluster they form the embryo, from the embryo they form the nuclei and from the nuclei it produce the crystal.

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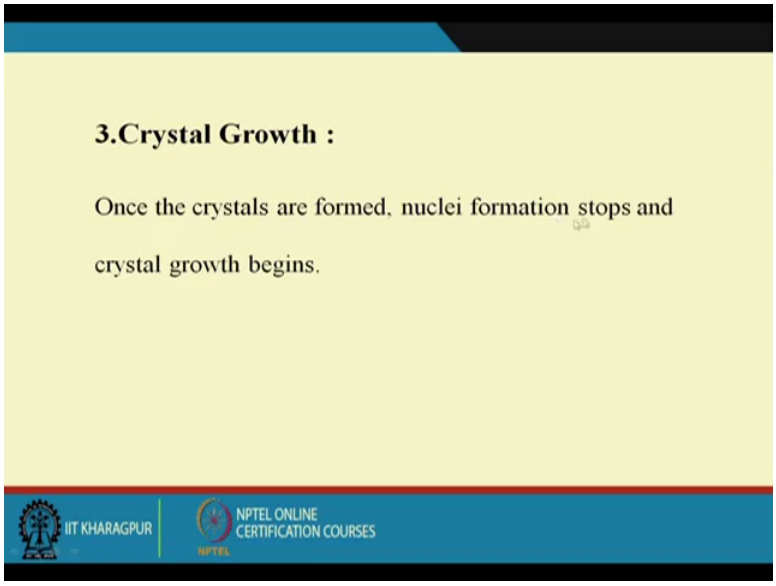

And once the crystal growth has formed the nuclei formation stops and crystal growth begins, so one crystal it may be bigger crystal it may be smaller crystal it depending on the nature of the crystal that characteristics of the solute that we have in the solution.

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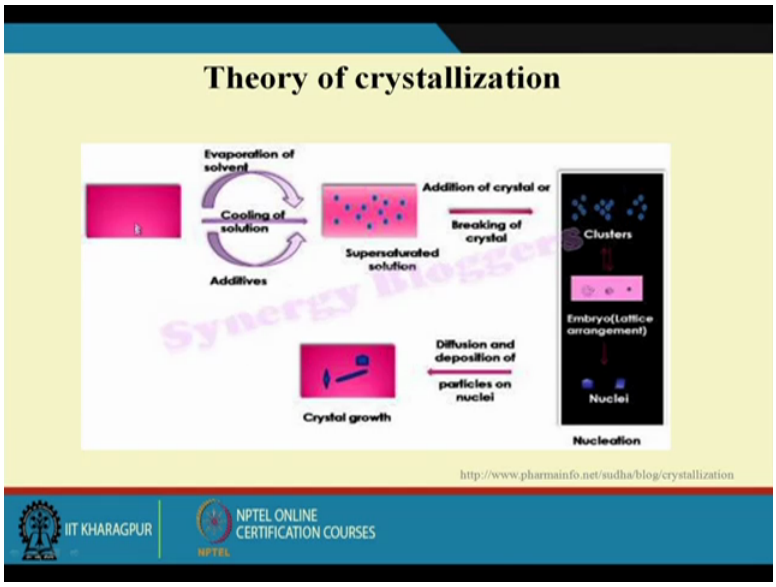

Now the pictorially that process can be explained like this, this is like this, this is the solution you can see when we evaporate the solvent then we cooling the solution this then particle this is we form the super saturated solution we cool down then particle formation take place that is you know that molecule. The addition of crystal of breaking this crystal is from the cluster of crystals, you can see the crystals they will be attaching with each other, after that they embryo formation is there you can see this some kind of embryo formation after that the nuclei is taking place, this nuclei from that we can get the crystals, crystals of different shape and size that we have, we can have the different size of crystal this is how the crystallization takes place.

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Now different type of crystallization equipment that is in practise one is draft tube baffle crystallizer, forced circulation evaporative crystallizer, circulating liquid crystallizer and tank crystallizer. There are different type of crystallizer that is available in the industries.

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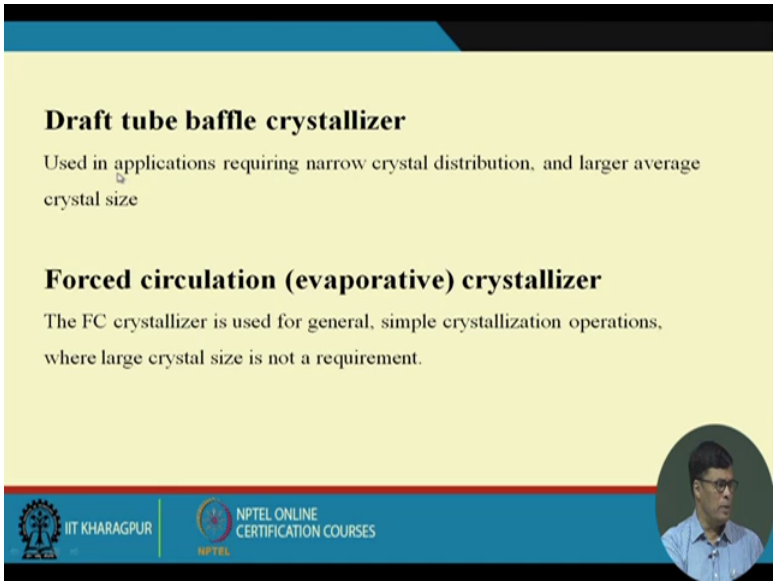

Now draft tube crystallizer used in application requiring narrow crystal distribution and large average crystal size because I work with citric acid industry and I work with the crystals like tank crystallizer I shall I shall I shall tell little bit details on that and then information of this crystallization process already available in the internet, if anybody interested they can see the details of this process. Forced circulation or evaporated crystallizer is used generally simple

crystallization operation where the large crystal size is not required as for then we have forced circulation evaporative crystallizer

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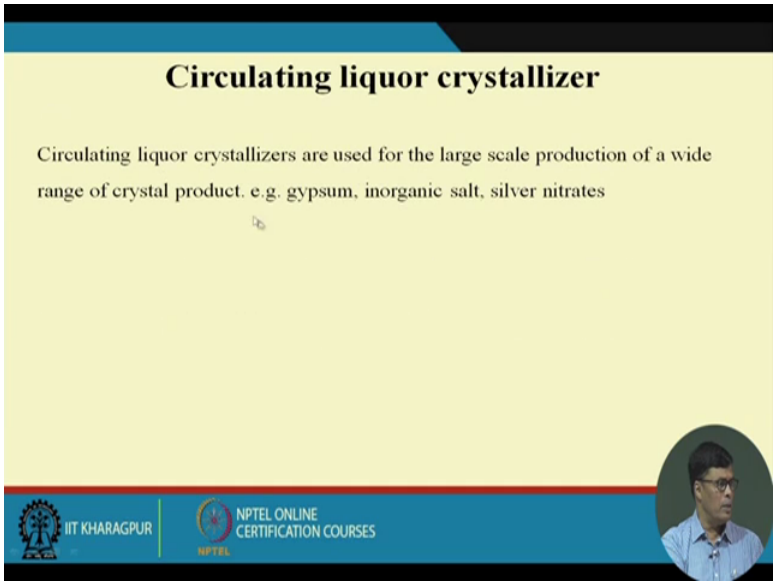

Then we have circulating liquid crystallizers circulating liquid crystallizers are used for large scale production of a wide range of crystal product like gypsum and inorganic salt and silver salts, silver nitrate largely is used.

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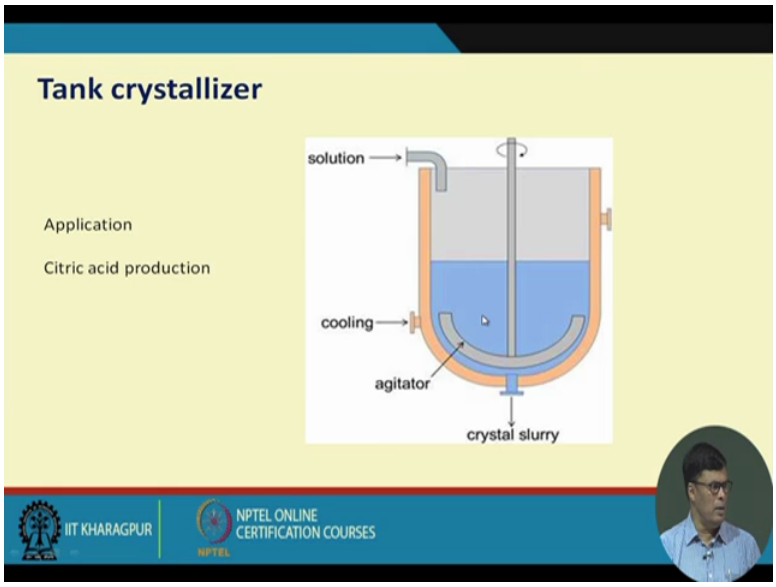

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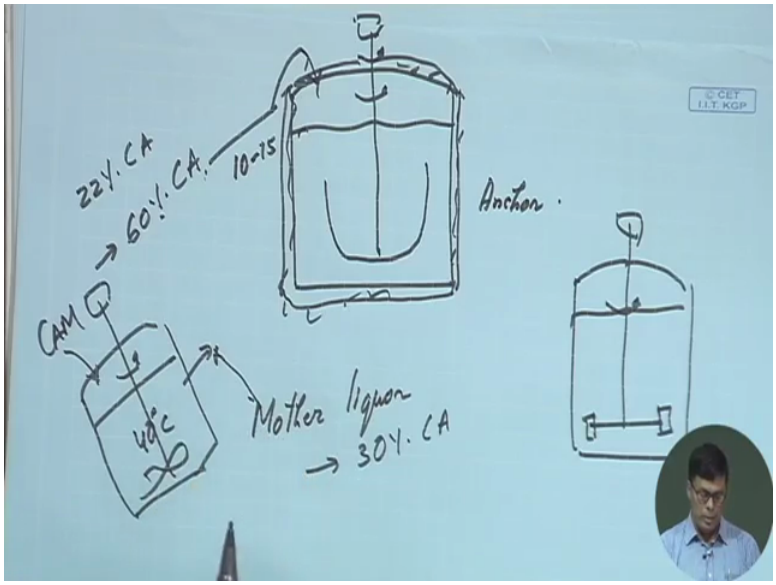

Now this is the tank crystallizer that we that we use in the industry and let me explain this process, it is very simple that we have the crystallizer that we have simple it is a tank and we have agitator and this agitator is the special type of agitator we we call it anchor because there are different type of agitator that we have but here we use the agitator which has very low shear forces because you know if the because I you can remember I I explained then in the fermentation industry we use the impaler, impaler is like this which has high shear forces, now if you use this high shear forces then what will happen that that crystal formation will be affected

Because if you have high shear force the crystal formation would be inhibited but if you have low shear forces then the crystal formation will not be inhibited, so we use this kind of crystal we reduce the temperature after concentrating this that you know I told you that 22 percent citric acid concentration we increase the concentration to 60 percent and then we put this here 60 percent this we put it here in this and we reduce the temperature may be we reduce the temperature about 10 to 15 degree centigrade. And then this is insulated because the outside is insulated, so the heat loss should be as minimum as possible, so it is a heat loss that is totally insulation outside

So we put it and we move this starrer and what will happen with respect to time then there would be kind of crystal formation that will take place inside this and then when the crystal formation maximum crystal formation is taken place, then we take the material out and we passes through the centrifugation process. And through the centrifugation process we can

separate that separate the crystals from the liquid and the liquid we consider as the mother liquid because after centrifugation whatever liquid that remain that is called mother liquidand this mother liquor contains about 30 percent of citric acid, so this can be used for the production of citric acid unhydrous.

I told you we that transition temperature of citric acid is about 36.6 degree centigrade, now what we do we take this in a vessel and we increase the temperature of this vessel to about 40 degree centigrade and then we put this is the mother liquor we have this is the mother liquor we put it here and which contains about 30 percent of citric acid, then we put it CAM here CAM we put and this the agitator we have, we put a agitator here and with the when we put the agitator then what will happen the water that present in the citric acid crystals that will go into the solution.

So this is things that you have then total the water molecule present in the CAM when it go to the soluble form then we take the material out and centrifuge we get the citric acid unhydrous, which is largely used In the pharmaceutical industries, so in this today's presentation I tried to discuss three things one is centrifugation how the through the centrifugation, how we can separate the solids from the liquid then I talk about the evaporation techniques different types of evaporation techniques is used.

I work with the citric acid industry I told you there we use the tubular heat exchanger just to heat the liquid and we throw it in a vessel where the vapour is goes up and concentrated liquid will go down and this concentrated liquid we cool down and take it in the crystallization process. Crystallization is a separation process I explained how the crystal formation takes place, it takes place after the different stages of operation the and I think next presentation I shall talk about other downstream processing that is used in the biochemical industries, Thank you.