**Fundamentals of Food Process Engineering**
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**Lecture - 55**
**Leaching and Extraction ( Contd. )**

Hello everyone, welcome to the NPETL online certification course on Fundamentals of Food Process Engineering. So, we are discussing about Leaching and Extraction and we have already discussed in our previous few classes on the basics of leaching and extraction and we have discussed the equilibrium in equilibrium condition in leaching and how we can draw the equilibrium diagram. And also we have seen the extraction process the basics of extraction process then, equilibrium diagram in extraction etcetera and today we will cover a few more topic in leaching and extraction.

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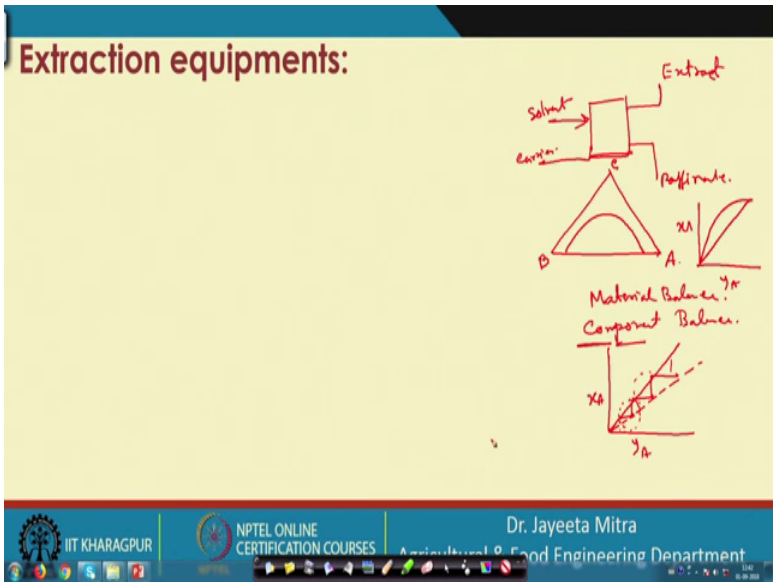


So, let see. So, extraction basically, when we call extraction we may define 2 cases either, the solid liquid extraction that is leaching and the equipments specific to leaching, we have already discussed in our first 2 classes. Today the equipments that, we will discuss those are used for liquid, liquid extraction or basically the solvent extraction and before going into that I once again try to just recapitulate that what happen in case of these extraction phenomena. So, we have to if any one of you need to design or need to assess any process, which involve extraction or leaching operation. So, you have to first

move with this you know, first you have to have the diagram of what is the solvent; what is the solvent coming in and what is the other solvent? Because in case of in case of this in case of this, liquid liquid extraction you have both the stream solvent both the streams are liquid ok.

So here, also even mention, that this is solvent and this is carrier ok. So, then you have the extract and raffinate, those 2 stream and you have to find, first that equilibrium diagram that you can read from those equilateral triangle diagram and if those 2 solvents are partially visible, you have you can have this. So, this will be your component C or A whatever and from that you need to find a fraction of all then, you have to see the material balance. Then you have to see the component balance, and always remember, whether it is in you know the leaching operation or extraction operation. Ideal case, is that you will get 1 plot, where your operation where, where you are you know X A by Y A if you plot where your X A is defining the concentration of the solute in the in the over flow or in the extract and Y A defining a concentration in the raffinate or in the under flow. So, you will have a 45 degree line, if the process is exactly ideal equilibrium condition.

But generally, it is not that. So, what the condition, we will get in each operating step. So, that is called the operating line. So, whatever the, you know mass, whatever the component balance will get from that, we can define that if the process is actually deviating, let us say actually deviating from the from the from the ideal case. So, you can find in each stage, if you have a multistage, if it is a single stage. So, you just get one such deviation block and if it is a multistage problem so, you will get a multistage or different stages, what will be the fraction and how much from the initial fraction to the final fraction? You can separate by this method.

Similarly, for the leaching if you remember, we had this kind of plot like, if this are the ideal plot for the X A verses Y A and then we have some operating plot, which have some variation from the ideal situation. So, all those things are there and finally, we will come to the equipment section. So, extraction equipments there are many kind of equipments and some of the thing will discuss here, only just to give you an brief idea of their types.

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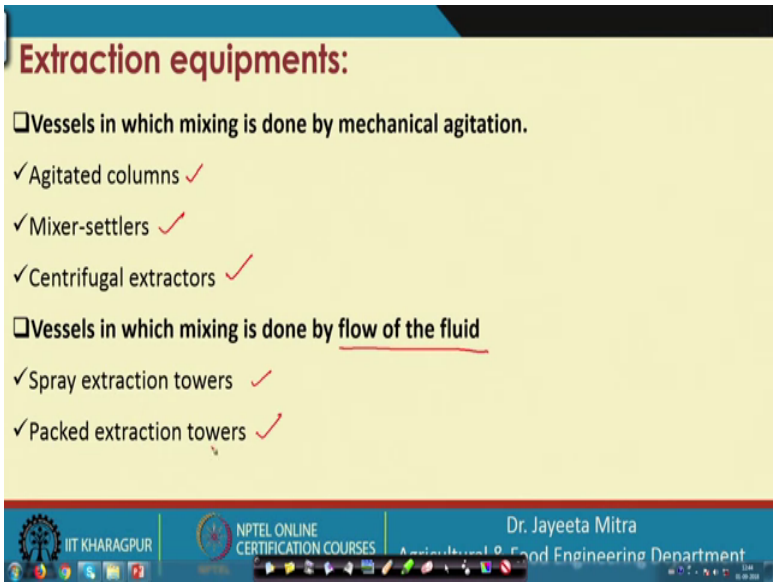


So, in the extraction equipment we have vessel, in which the mixing is done by mechanical agitation, we have seen initially that for extraction the agitation, that mechanical agitation and temperature, these are having an positive role because, this increases the rate of mass transfer.

So, for that we need to have vessels, in which the mixing is done and some agitation we need to provide there ok. So, agitation may be of different mechanical arrangement, we need to provide, but some agitation we is required because, it will help in the proper extraction. So, we have agitated columns mixer settler we are having and centrifugal extractor, this is also we are having. So, in both the cases we use some motion or some agitation mechanically or then we can design those vessel for extraction.

And mechanical agitation this 3 where as we can also have this mixing done by flow of the fluid. So, these are spray extraction tower and packed extraction tower. So, extraction should be should be done, where proper mixing of the 2 phases are there and the 2 phases coming proper mixing or in a proper properly dispersed condition. If you provide some mechanical agitation to that apart from mechanical agitation, we can also give the flow of the fluid that is in the form of spray of the packed extraction power.

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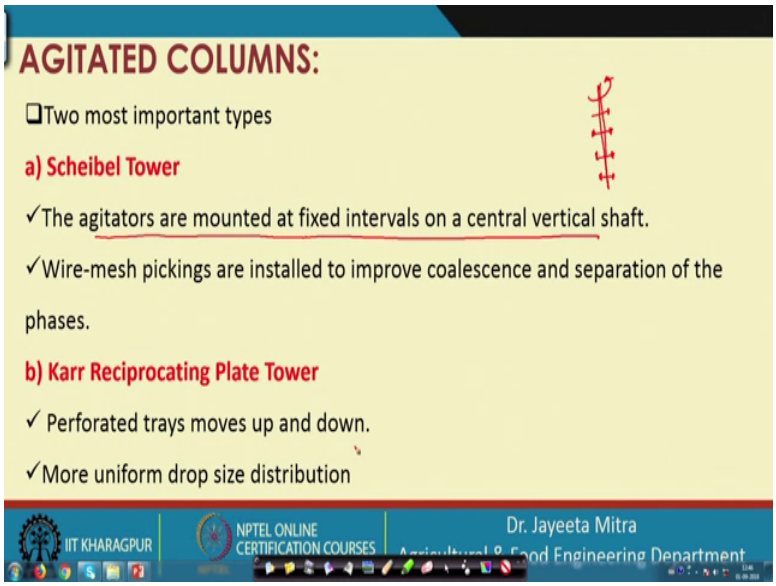


So, agitated columns, 2 most important types of agitated columns that is, used for liquid liquid extraction one is the Scheibel tower. The agitators are mounted at fixed interval on a central vertical shaft. So, this is kind of a mechanical agitation that, we provide here and how we provide this? We give agitators at fix interval, we give agitators we mount agitators at the fix interval on a central vertical shaft ok. So, there will be one central vertical shaft and in that we will fix some you know agitator that is will rotate and this thing will be kept in the tower.

So, wire-mesh picking are installed to improve the coalescence and separation of the phases ok. So, wire-mesh pickings are installed to improve the coalescence and separations of the phases, that is important and another is the Karr reciprocating plate tower. So, what is that here the perforated trays move up and down. So, the tray itself, are perforated. So, that the proper mixing will takes place and they move up and down. So, some shaker kind of arrangement or the vertical shaking arrangements are given to this reciprocating plate towers.

So, more uniform drop size distribution, we can find here. So, more uniform drop size distribution indicates that proper mixing will be there and the extraction efficiency will be very high.

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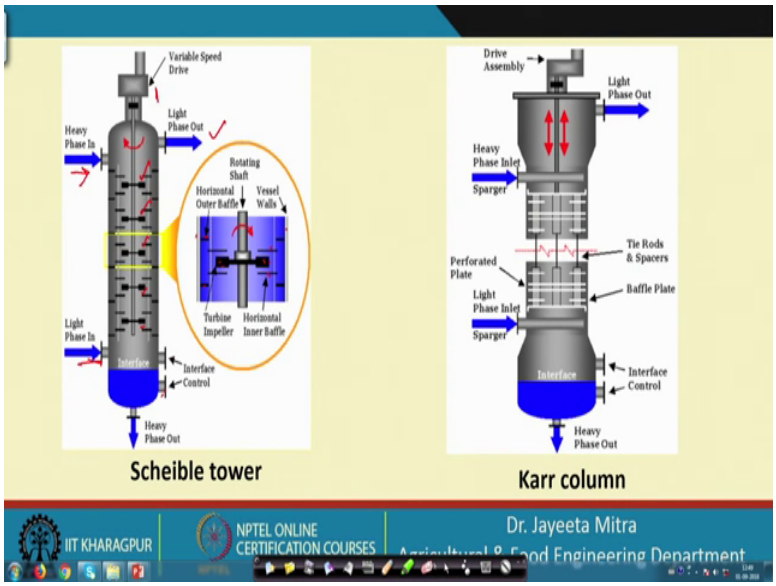


So, these are the 2 diagram that showing the Scheibel tower and Karr column. So, in the Scheibel tower, you can see that there is a variable speed drive. So, this variable speed drive is responsible for rotating the central vertical shaft at the required speed and there are agitators mounted at a fixed interval, you can see and we can provide different kinds of baffles there to have the proper mixing. So, in this the heavy phase is coming, in from the from the top and that heavy phase will come down eventually, while coming down that will have the effect of this agitators, there will be some mixing and the light phase is entering into the system from the bottom. So, the heavy phase will eventually, come down and exit from the bottom and the light phase will eventually, going to the upper right section and exit, the light phase exit from the upper right session.

There are 2 interface control is given and if you see that there is the turbine blades are there these are the turbine blades in one section, if you magnify one section in the Scheibel tower and see. So, there are horizontal outer baffles and there are horizontal inner baffles ok. So, 2 outer baffles are there and 2 inner baffles are there. Inside the inner baffles, the turbine impellers are there and outside there is a vessel wall and there is a rotating shaft. So, this is how this kind of tower operates and help in proper mixing.

The Karr column is there here, also the drive assembly is given, this drive assembly operates this in a reciprocating motion up and down reciprocating motions are there these are the perforated trays. So, again the heavy phase is entering from the top. So, the

simple thing is heavy phase always comes from the top because, otherwise we need to use a pump to just throw it from the bottom to the up. So, this design is pretty common in most of the cases and then this heavy phase will come down eventually, mix with the light phase that is that is entered from the bottom and there is a sparger given and these are the perforated plates, these are the baffle plate and tie rods and spacers are also given and then this liquid phase will go out.

So, because of this perforation and the reciprocating motion very well mixing, we can observe in Karr columns. So, this is very effective and there are some other kind of you know extractions towers also available.

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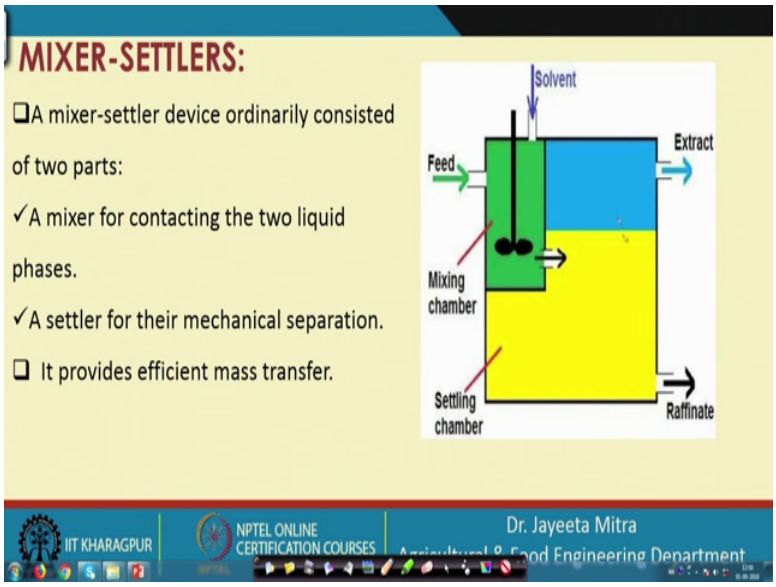


Mixers settlers, a mixer settler device ordinarily consisted of 2 parts. One is a mixer for contacting the 2 liquid phases and a settler for their mechanical separation, it provides sufficient mass transfer. So, what we can see here is that, the solvent and the feed enters into a mixing chamber ok. So, proper mixing will takes place and then it is settle in another chamber, which is called the settling chamber. So, this settling chamber, raffinate which is supposed to be heavier one will come down and the extract the lighter one will flow.

Now, there are certain system where the mixer and settler are separate. Separate chambers are there in one chamber, there will be mixer and when it is moved on to a cylindrical kind of chamber, which is used for the for the settling, but in this mixer

settler, it is combined system both the operation mixing and settling or separation is taking place ok. So, mixer for contacting the 2 liquid phase and settler for their mechanical separation, it provide efficient mass transfer.

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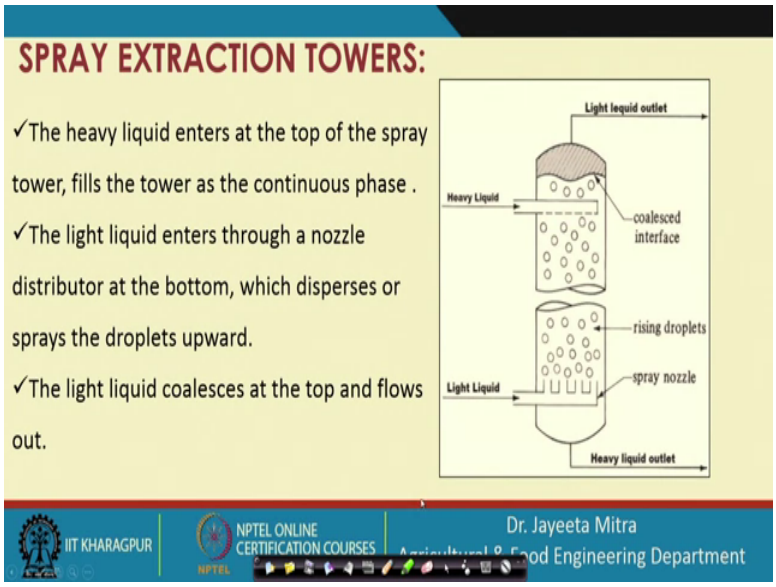


There are other spray extraction towers, the heavy liquids enters at the top and in the spray tower and since, the tower as the as the continuous phase, the light liquid enters through a nozzle distributor at the bottom and which disperses or spray the droplet upward.

So, because of this drop form they have very high efficiency to mix with the heavy liquid, that is coming and the light liquid coalesces at the top and flows out ok. So, the all the all this spray, that is the rising droplet form, it is coming and it coalesce and it going out from the top session and the heavy liquid that is coming from the bottom and you know extracted. So, while this light liquid is coming in contact with the heavy liquid and coming towards the top, the solute component or flavor component, that is it intent to take from the from the heavy liquid to the light liquid that transfer is taking place. So, this is how the spray extraction towers work.

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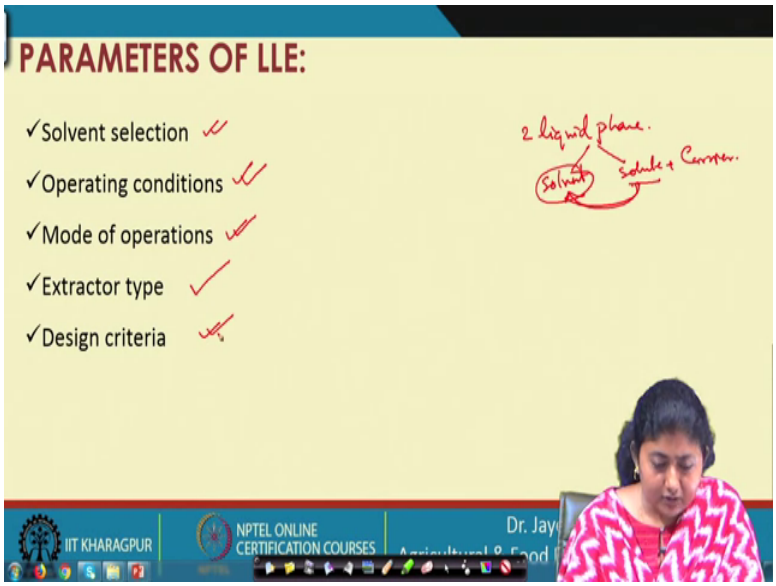


So, parameters of liquid liquid extraction so, first is the solvent selection solvent selection as we have seen that, these are these are very important because, in liquid liquid extraction the 2 liquid phase are there, 2 liquid phase we observe here. So, one is having the solvent generally, the pure solvent and the other is having the some solute plus carrier ok. So, since this solute has to come from this phase to this one and this only come is the chemical potential is higher here or if it is have, the affinity towards this solvent ok.

So, we have to carefully selected, this solvent so that, the solute can come out from this phase. So, solvent selection is very important, whether we want to separate some organic material some flavor compound. So based on that, we can select the solvent ok. So or if it is water soluble simply so, we can use that, then second is the operating condition. So, operating condition also in that we can consider temperature as 1, because it helps in extraction it, when it make the sample in a low viscous sample. So, it helps in extraction and mobility is increased and mode of operation. So, there will be you know mode of operation, in the sense the batch operation or continuous operation, then the concurrent or counter current operation. So, those will (Refer Time: 17:00) effect the efficiency of the liquid liquid extraction process.

When extracted type that we have seen many kinds of extractor are there spray type extractor or it is like we have seen the mechanical agitator type is there and what is the design criteria? So, design criteria that, the full design starting from all the port, all the

inlet and the flow line and the mechanical agitation part. So, all those things are important ok.

So, these are about the parameters of liquid liquid extraction system. Now we will move on to one more extraction method, that is becoming very common nowadays specially in some industry people are going to use that one in a huge scale because, they have some advantage although the cost may be a little high, but they have some advantage. So, let us see one such very important extraction technique.

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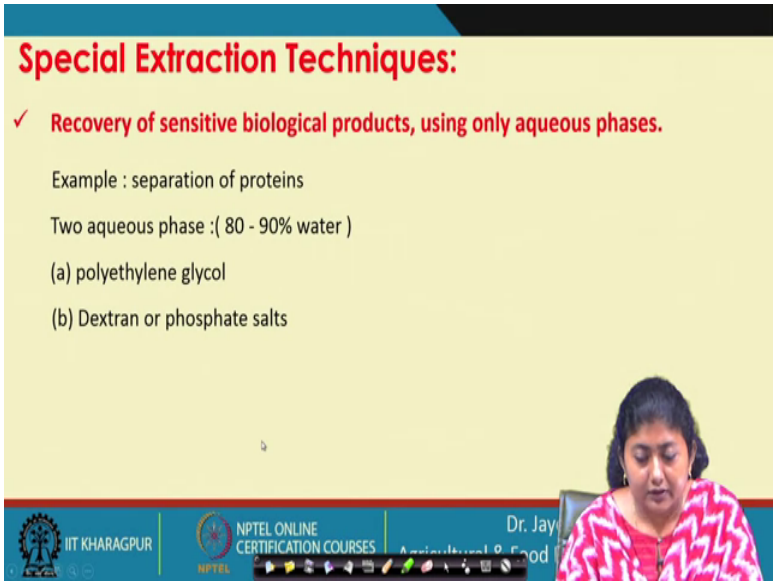


So, this technique is called super critical fluid extraction and with that some other special extraction technique will also see for example, the separation of protein, 2 aqueous phase, when 80 to 90 percent water is there like polythene and glycol then Dextran or phosphate salts are there and. So these are the sensitive biological products and recovery of this, we use only the aqueous phase ok.

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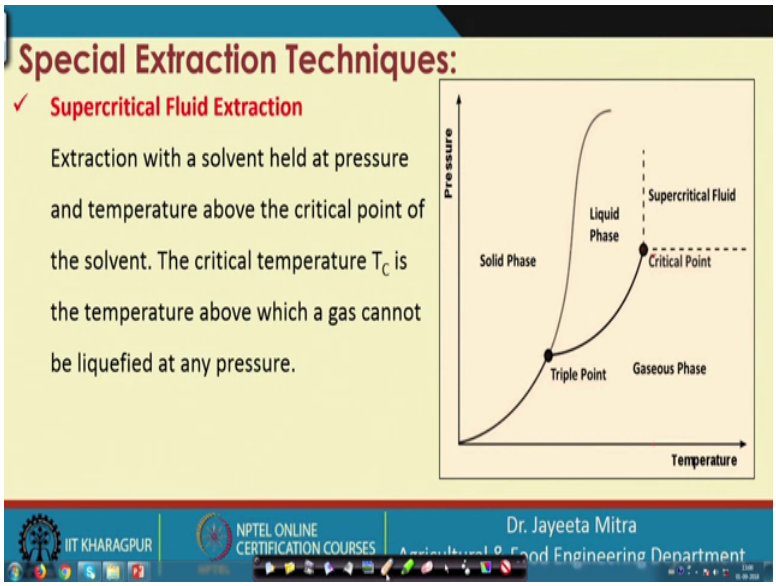


So, these are some special extraction methods. So, as I mentioned that in we mention that super critical fluid extraction, which is very important techniques nowadays.

Now here, what happens is that extraction with a solvent held at a pressure and temperature above the critical point of the solvent and the critical temperature T c is the temperature above which a gas cannot be liquefied at any pressure. So, look into the phase diagram, what happens is there the pressure and temperature, if we try to increase. So, eventually, from the solid phase, liquid phase and gaseous phase after that, one condition will come that is beyond the critical point. Critical point means the corresponding critical temperature and critical pressure, when we exceed then the fluid then the some gases are there that will become in a super critical fluid condition.

For example, carbon dioxide is one such gas that that achieve a super critical fluid condition, very easily and it is having very low critical temperature around I think 31.8 degree Celsius, this much is the critical temperature and the critical pressure. I think it is it is higher than 10.5 mega Pascal. So, it is higher then, that. So, if we have that much do, then we can have the super critical conditions. So, what is this condition actually? This condition the super critical fluid achieve some property, which are specific to this condition and that is neither belong to the gaseous phase nor belong to the total liquid phase, but in this situation in the super critical condition, the few parameters of the fluid

is increased so much that, they enhance in extraction for example, the solubility of a component.

Because we know that, the gases are of low density of low pressure right. So, the density of the gas is increased, when we increase the pressure; however, the liquids are not changing their, you know density over the pressures all most in a moderate pressure range or in the atmosphere range those are independent of pressure. So, when the gas are exposed to higher pressure and beyond certain limit, when it is pressure increases and it is density becomes higher. So, density reaches almost towards, the density of a liquid material although, it will not become liquid, but it is density will approach to the that of the liquid and that in a way increase the ability to solubilize the component.

So, solubility of that of that fluid is increased ok. So, that is why it is helping in extraction and the effect of pressure is very much observed in this particular case; however, the effect of temperature in the super critical extraction is not that much pronounced, because super critical fluid extraction has been developed on this principle basically, focusing on this principle that above the critical point, if we change the pressure, if we increase the pressure then, the solubility of the component in that super critical fluid will increase.

But if we look for temperature change so around 10 mega Pascal pressure temperature will not positively effect, the extraction rate rather it will degrade that and even if the temperature increases, when the pressure is very very high around 30 mega Pascal or. So, then again the temperature will cause the positive effect, but the intermediate phase it will not have that much effect or till not known that much what is the mechanism exactly for that temperature will help right.

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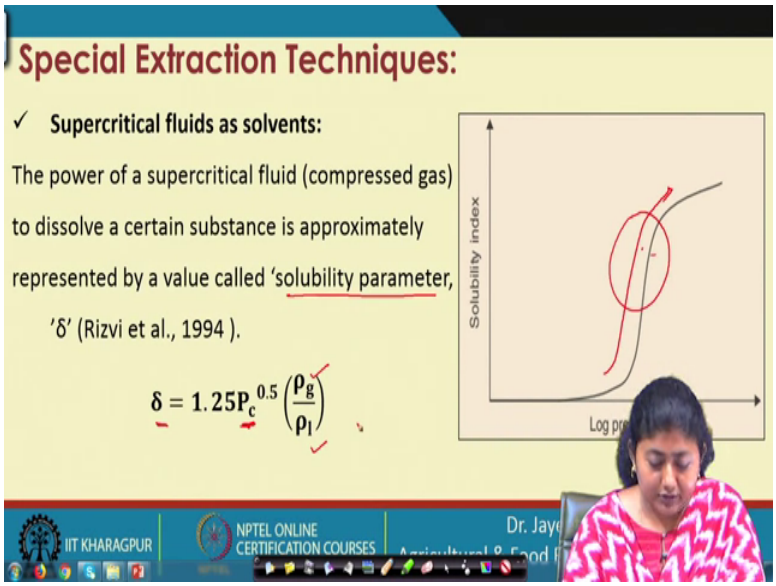


So, super critical fluid as solvent so, because of this property that at the higher pressure, this super critical fluid will behave in such kind of a liquid, which has higher solubility and therefore, it is acts as a solvent. So, the power of a super critical fluid or actually a compressed gas to dissolve a certain substance is approximately represented by a value called the solubility parameter ok. So, this is the solubility parameter, solubility parameter delta. So, solubility parameter is expressed by this equation 1.25 P c, that is the critical pressure, 2 to the power 0.5 into rho g by rho l so, density of gas and density of liquid these 2 ratio.

Now, we can see that when the gas is compressed. So, the solubility is very highly increased and this is the benefit that, we try to take out. So, this is why because of this, solubility increase of this super critical fluid under high pressure, we try to use this as a potential solvent for solvent extraction method and temperature as I have already mentioned that effect of temperature is not much pronounced; however, sometimes what we do that as a co solvent, we add some other solvent to this super critical fluid and that in a way increase the you know dissolution capacity of this super critical fluid. So normally, carbon dioxide is very common super critical fluid and in many food processing sections. Now they have started using the super critical extraction technique.

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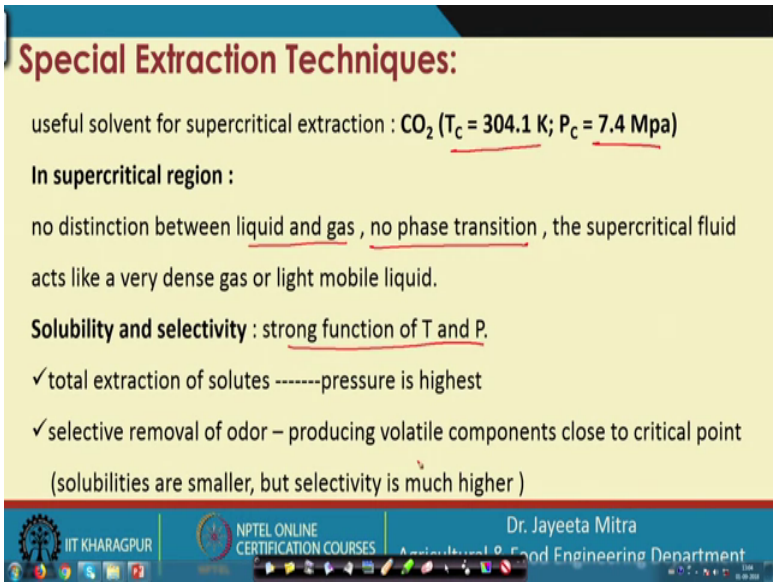


So, this is useful solvent for super critical extraction is generally carbon dioxide as I mentioned this is very common and it is temperature is 304.1 Kelvin ok. So, as I mentioned that 273 is a 273 is our starting point. So, from that we can consider that it, if it is 0 then what will be the temperature Celsius? And Pc is generally 7.4 mega Pascal.

So, in super critical region no distinction between the liquid and gas can be done as I mentioned that those, they will gas, but their density will approach to the liquid. No phase transition will be observed and the super critical fluid acts like a very dense gas or light mobile liquid solubility and selectivity. So, as I mentioned that the solubility and selectivity, this is a strong function of temperature and pressure. So, not only super critical gas will increase the selectivity, they will they can be use for many number of solute or many number of components. So, because of their broad selectivity, that is one advantage. Second advantage is higher solubility and we have seen that it is a strong function of pressure and temperature, when the pressure is at some higher range ok.

So, total extraction of the solute is possible, when the pressure is highest and selective removal of odor producing volatile components close to the critical point. So, close to the critical point we can separate the odor producing volatile component also. So, in this particular case the solubility may be smaller, but the selectivity is not much higher.

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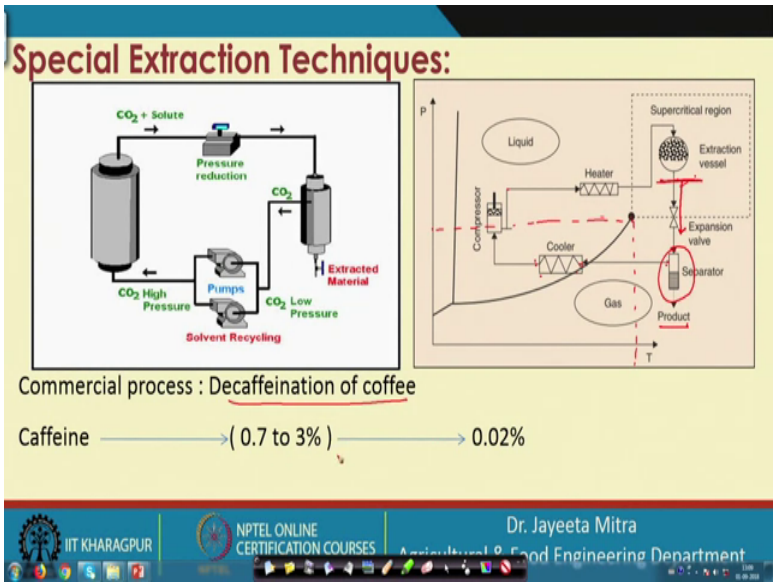


Now we will have a quick look on how the operation of super critical fluid extraction takes place. So, there are some part that we need for super critical fluid extraction. So, let us see the, this is the phase diagram of super critical carbon dioxide. So, we have in this zone initially, when the extraction takes place.

So, extraction takes place in the super critical region that is beyond the gas and liquid region, when you have the critical temperature, you have exceeded and the critical pressure that also you have exceeded ok. So, you have exceeded the critical pressure and temperature. So, we then have a extraction vessel. We then have a extraction vessel, where the extraction actually happens, after extraction we need to separate that because, extraction means we have the super critical fluid and we have the solute of the component that, we have to extracted from the other solvent.

So, then or from the feed solution so, then we need to decrease the pressure, when we decrease the pressure then, no longer the solubility is higher ok. So all the component will be separated and this carbon dioxide in a gas form we will get.

Now then, then we will get the carbon dioxide in a gas form, we will separate that; we will separate that in a separator and after separation, the product will come out from the bottom because, that will be deposited and the gas will taken out to a cooler. So, what we do now for that the temperature is lowered in that cooler? Ok the temperature is lowered and when the temperature is lowered, the gas is become eventually the liquid. So, there is

a heat exchanger here that helps in that a liquefication of that carbon dioxide and after that that will be taken in a tank, all the liquid carbon dioxide is taken in a tank and again since, we need to increase it from the lower pressure to the higher pressure side so, we increase the pressure by compressor. That compressor increase the pressure of that liquid carbon dioxide further, we increase it is temperature by again, passing through a heat exchanger and then it is entered to the extraction vessel and the same process will be repeated ok.

So, super critical region is there then, there is a lower pressure region then, lower temperature then again increase pressure and increase temperature and basically, we can categorize this to higher pressure side and the lower pressure side. So, in the higher pressure side, the compressor the heat exchanger or heater and the extraction part is there in a lower pressure side. We have this expansion valve we have separator and this heat exchanger for cooling of the super critical fluid ok.

So, here we can see that the solvent recycling, when we are doing we are using the pump and then by using that, we are sending it to the high pressure section then the CO 2 and the solute is the pressure reduction will be there. So, that; that means, we can see that without applying any extra energy, we can just by lowering the, we can separate the solute and the super critical fluid.

So, like an other extraction, we have seen that we need to apply the distillation or crystallization to separate the solute and the solvent, but here, the extracted material is very well can be separated by just pressure reduction ok. So, this is very efficient method in that way, now commercial process is decaffeination of coffee, this is done by super critical fluid extraction. So, commercial process is decaffeination of coffee from the caffeine, which is having 0.7 to 3 percent caffeine that we can extract up to 0.02 percent by using the super critical fluid extraction. So, this is how this method is very beneficial. So, this has some advantage and disadvantage, we will quickly look into them.

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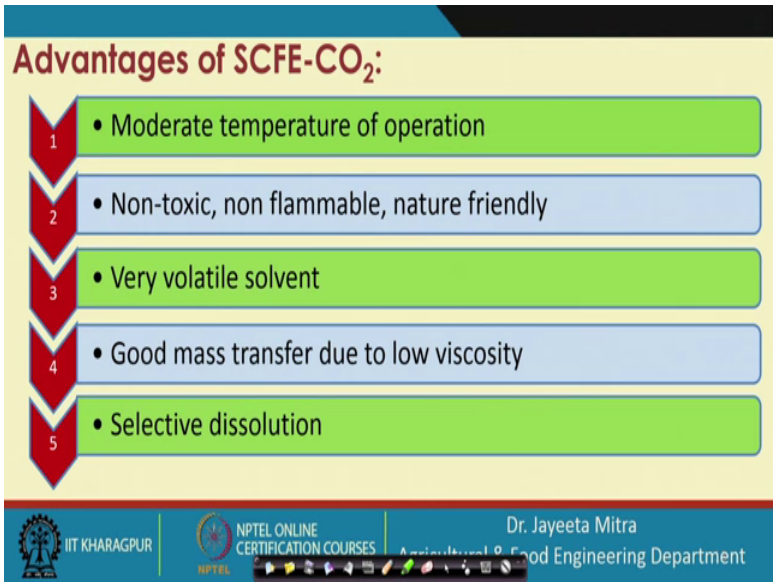


First let see the advantages of super critical fluid extraction using carbon dioxide. So, here we can operate it at moderate temperature of operation, we have seen that the temperature is around 304 Kelvin or 31.8 around degree Celsius. So, it is very helpful to operate, there is no high temperature needed. So, the heat sensitive material will also can be very well preserved.

Carbon dioxide is considered to be nontoxic, non flammable and nature friendly. So, using this liquid super critical you know carbon dioxide does not have any ill effect on the product or that and it does not have any residue also on the product it is very volatile solvent. So, when we try to separate, this at the low pressure, it completely gets separated from the solute or from the component that, we want to separate no residue is sound on the product.

And good mass transfer to low viscosity ok. So, although it is have in a super critical state it density is increases towards liquid, but it is viscosity remains low. So, because of this property low viscosity and high density their solubility increases ok. So, that that is helping in a mass transfer and selective dissolution this is another that is the selective dissolution happens, in case of liquid carbon dioxide for most of the material. So, that way it is very advantages.

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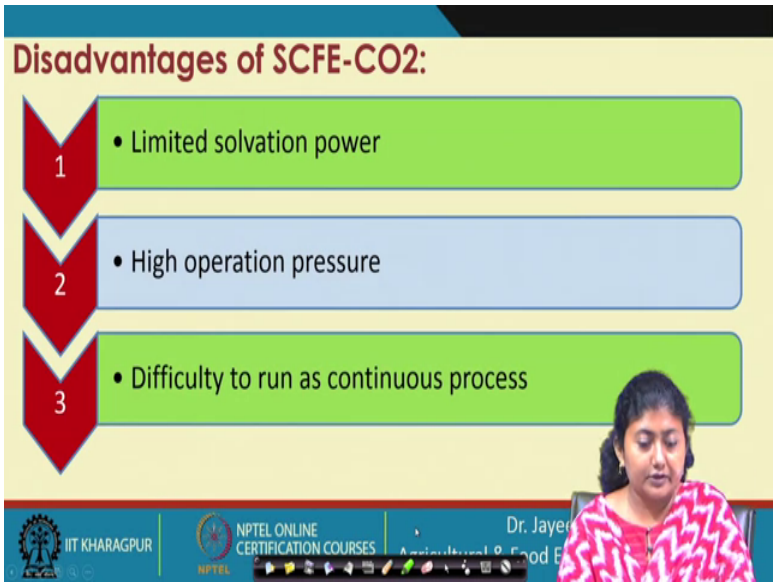


Now, what are the may be negative points of super critical fluid extraction? So, limited solvation power and this can be overcome by adding some co solvent as I was mentioning that some time, what happen that with this super critical CO 2, some other solvent is added and that in a way helping in a that that will mix properly with that and they will increase the solvation power of this super critical fluid and higher operation pressure. So, pressure is definitely one thing that we need to design proper system for that and for that the mechanical arrangements, if the equipment thing will increase and the cost will also increase. Because initially, you have to say have a very huge set up for all this pressure increase thing. So, the cost initial cost will be very high.

And this is there is a difficult to run as a continuous process. So, there is a difficulty to run, this as a continuous process and although we need to look for more advancement in that, but still in a batch process also this is an efficient one, if initial cost is more, but if you can if you can run it on the continuous basis and for a for a long run, we can get very good advantage of super critical fluid extraction, because eventually, the running energy that is that requires in case of separation because, maybe we have not mentioned that, but ideal cases when we go for extraction or leaching process.

The energy needed in the extraction is very less, but energy needed in separating the solute from the solvent of the solvent from the product of the solute slurry that takes a

huge energy because, where we need to apply the evaporation or the distillation or those kinds of techniques which are very energy intensive technique.

So there, actually 85 percent of the energy of the whole process is getting wasted or not waste, we can think of utilizing there itself for separation, but for extraction we only use 15 to 20 percent. So, if we use the super critical fluid extraction may be that separation thing is no cost involvement required, because it can very easily by lowering the pressure we can separate. So, may be that extra cost that we can reduce there can be utilized for the initial setup preparations. So, that way it is very beneficial.

So, this is all about the thing that will cover in you know extraction and leaching and we have come to the end of this particular topic and if you have some specific discussion, because we have not gone to very deep about this leaching and extraction process, but and there are detail mass transfer analysis is also required. I hope you will Refer to that mass transfer, in other courses that are running on the mass transfer many courses are there and if still you have any specific query please send that to a. So, I will try to clarify them.

Thank you.