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Module - 02 Lecture - 37 Liquid-liquid extraction (L3)

Hello and welcome to the third class on liquid-liquid extraction. So, in the last two classes I had introduced liquid-liquid extraction to you. We had found out that what are the important features or what are the unique features of liquid-liquid extraction. It shares some common features with the other separation processes which involve mass separating agents be it a liquid or a vapor phase or a solid phase.

So, therefore, whenever you involve mass separating agents there are some particular common problems or some particular common features which have to be considered primarily with reference to solvent regeneration and recycling. And along with that liquid-liquid extraction it shares some specific problems we found out particularly because of the difficulty of separating the extract phase and the affiant phase after the mass transfer has taken place.

And I had also mentioned that due to this we find that the stage efficiency is generally much low, it is more for the inadequate separation compared to the insufficient mass transfer. And after that what we did? We found out that the simplest way to bring about liquid-liquid extraction is that when you bring a feed enters and the solvent solution together in some particular vessel they are agitated by means of a turbine impeller or a propeller whatever it is.

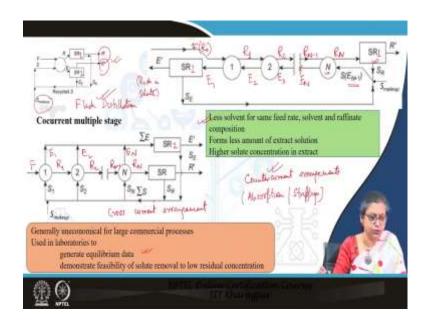
They agitated very well. Definitely in the vessel you will have one particular phase which is completely dispersed as droplets in the other phase. And I had already mentioned to you that the phase with the lower volume fraction that phase is usually dispersed. And after dispersion the two phases they come in contact with one another mass transfer has occurred.

After mass transfer has occurred you have to give a particular residence time for the mass transfer to occur and that, I had already mentioned, can be obtained from the expression of holding time. But, if you have to derive holding time from the basic mass

transfer equations then in that case you need to have a knowledge about the mass transfer coefficient as well as the interfacial area.

And which I had already found out that it is not very easy. So, therefore, generally these particular data they are obtained from pilot scale experiments. And then we had discussed a detailed design and the scale of equations for a mixer settler arrangement.

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Now, the mixer settler arrangement was discussed and the problem that was done primarily refer to a single stage extraction column where a feed and solvent it enters. Mass transfer occurs in this particular extractor, a raffinate and an extract streams they come out both of them they are introduced or they are taken into individual your solvent recovery units.

Raffinate is taken into one solvent recovery unit, the extract is taken in another solvent recovery unit. The solvents are recovered then they are combined and recycled and some amount of makeup solvent is also given. After the solvent is recovered naturally we have a solute rich phase extract phase which is given by E prime and also a solvent depleted phase from where the solvent has been completely extracted from the feed.

And we get the raffinate phase; these are the products that we get from the extractor. Now, in this particular case as we see that there is the feed and the solvent has been contacted just once only and then they are separated and the products are taken out. And in the problem that we had done we found that we could get sufficient amount of purity of the acetic acid that we had desired.

Well, but generally for most of the cases as you already know that if we have multiple contacting and separation then in that case to increase the efficiency of separation and to obtain greater purity products naturally its better if we go for multiple contacting and separations. So, in this particular case what we have? We have of a feed which is introduced here, the feed which is introduced here.

Normally, since we refer to the output as the raffinate and we denote the outputs with the stage from where it has come out. For example, the raffinate from stage 1 is subscripted as R 1. Raffinate from stage 2 is subscripted as R 2. In the same way the raffinate from stage N minus 1 which enters N it is subscripted as R N minus 1 and this is subscripted as R N. In the same way quite often the feed is often denoted with as R 0 or R subscripted as 0 or it is coming from the 0th feed.

So, what happens? The feed it flows in one particular direction and the solvent that flows in the reverse direction. That means, and normally the stages they are numbered according to the sequence of the feed movement through the stages or in other words the first stage which the feed contacts is known as stage 1 and so on, the last stage which the feed contacts is known as stage N.

So, therefore, this solvent it enters through the in the stage N and naturally the solvent which enters and the product which comes out that is denoted as the extract. So, the product which is coming out the extract part which is coming out from stage N this is denoted as E N. So, the solvent which is in introduced in the stage N is often subscripted as E N plus 1.

Now, this particular solvent what it contains? It contains the solvent from the two solvent recovery units; the solvent recovery unit for the raffinate and the solvent recovery unit for the extract and along with that the makeup solvent is given just like in the single stage contacting and then it is introduced in the stage N.

So, therefore, what we find? That the extract moves counter current to the raffinate and for the extract also the stage from where it is going out accordingly it is numbered, but

the numberings are fixed based on the sequence of contacting of the stages with the feed solution.

So, naturally the feed enters R 1 go goes out goes into stage 2, R 2 goes into stage 3 and so on. Finally, R N minus 1 goes to stage N and R N is the final raffinate which comes out from this entire counter current process. Same way S which is otherwise E N plus 1 enter stage N. E N comes out from here and then from the Nth stage E N minus 1 come. Finally, say E 3 enters stage 2 E 2 enters stage 1.

And finally, the product which comes out it is E 1 and this product or at this extract it goes to the solvent recovery from where the solvent is recovered; is recovered solvent is denoted as S_E and the product that we get after solvent recovery which is rich in the solute that is denoted by E prime. Anything which depleted from the solvent they are denoted with primes on the main nomenclatures.

Now, there is one other thing which I would also like to mention that in liquid-liquid extraction we can also have an arrangement where in where the feed flows from stage 1 to stage 2 stage 3 in the same way as it was flowing in the counter current operation, but in each stage it is contacted with a fresh solvent.

And from each stage we get individual extracts and all these extracts they are all combined. They are sent to the solvent recovery section 2 and then the entire solvent recovered it is recycled back and this solvent depleted solute rich extract is taken out as a product.

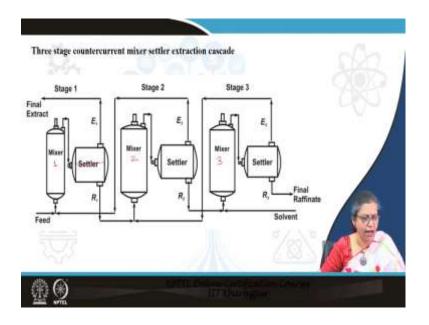
In this case you can very well understand that every time the solver the feed it is contacted with fresh solvent and this is known as a cross current arrangement. It is quite obvious that when we go to industries or send for industrial practices definitely we would prefer the counter current arrangement quite naturally just because the counter current arrangement it has also been mentioned when you were when the column internals and the vapor liquid and gas liquid contactors were discussed.

That for counter current arrangements quite naturally even if you have the same feed flow rate you have got the same solvent flow rate, you have got the same raffinate composition specification, the same feed composition specification you will be requiring less amount of solvent because the same solvent is going here. On the other end here you are giving fresh solvents for each particular case. And also along with the utilizing less solvent, it is also going to form less amount of the extract solution.

So, in the solvent recovery unit you have to deal with a lesser amount of liquid and you also get a higher solute concentration in the extract so, normally, we go for counter current arrangements. This cocurrent arrangements, they are just used in laboratories for generating the equilibrium data. If you want to generate equilibrium data which you will be using in the actual plant for that often you can use cocurrent arrangements.

And also suppose you would like to test if the solvent that you have used if that is going to be feasible and if it can bring down the concentration of the desired product to the desired concentration that you require just for testing the feasibility and for generating equilibrium data particularly for laboratory scale operation the cocurrent operation can be used or else for most of the cases we use a counter current arrangement.

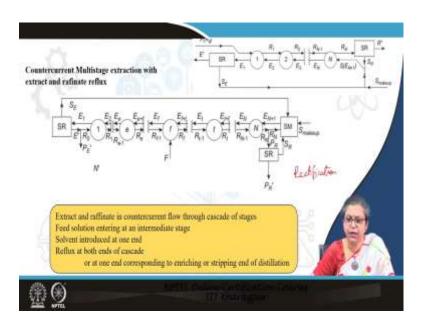
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So, a typical three stage counter current arrangement of mixer settlers it has been shown here. In this particular arrangement what we see? We find that the feed enters. The first mixer then the extract and the raffinate they are separated in the settler. The extract goes out. The raffinate is then sent in the mixer two and in the same way it moves to the mixer 3 and finally, we get the final raffinate.

And from each stage the extract the solvent it is recycled or rather it flows in a manner counter current to the feed solution. So, therefore, in this particular way we can just keep on increasing the number of mixer settlers and we can arrange them in series such that a mixer settler battery can be formed and quite naturally in that particular case we are going to have an improved recovery.

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Now, along with that there is also one other arrangement that we go for in order to ensure much better recovery. It is simply the way in the that we have done in the rectification columns. In this particular case also the raffinate and the extract they flow counter current to each other through all the stages.

And the feed is introduced at some particular intermediate stage which corresponds to the feed plate in the tray sections. Solvent is introduced at one end and there can be reflux either at one end or it can have reflux at both the ends corresponding to the enriching and the stripping sections of the distillation column.

We can have this. This definitely goes for an improved distillation. Generally, for extracting aromatics from lubricating oil, this particular arrangement is used. We can also ensure in this case that the temperature at the two ends are maintained at different levels. So, that the equilibrium at the two ends they are not the same and therefore, this can also help in improving the extraction.

And this difference in temperature or the different temperatures at different levels they can also be maintained by the circulating free flux arrangement which has already been discussed when we were discussing the introduction to the mass transfer or the separation operations. Now, if you see these each and every of these arrangements that we have seen it is quite evident that this single stage arrangement that is similar in operation to a flash distillation. Is not it?

So, this is akin to the operation of flash distillation. On the other hand, if you observe this counter current arrangement which is mostly done, do not you think that a similar sort of arrangement was discussed well while you are discussing the tray sections or if you are trying to find out the number of ideal stages while you are discussing absorption or stripping.

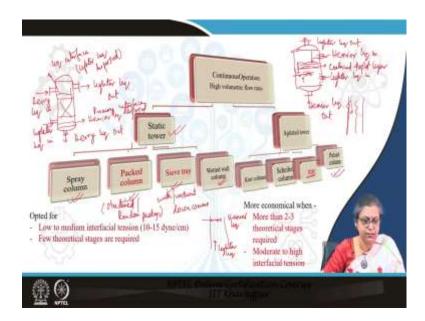
So, therefore, the counter current arrangement it is almost similar to this. And this particular arrangement the counter current multistage extraction with the reflux this is obviously, this is similar to the rectification column that we had discussed. So, from this it is quite evident that naturally the equations that were the mass balance equations that were written for flash distillation for absorption for rectification, the same equations should be applicable here.

The only problem we have it that definitely we can write the same equations. There we had used some notations. In this particular case the notations have to be replaced with the E and R corresponding to the extract phase and the raffinate phase. So, this is one thing. But, along with that we have to remember that due to this miscibility problem due to which we have to refer to the ternary plots just for that problem certain assumptions that we had taken there.

For example, constant equal overflow, a constant molar flow rates then linear operating line, linear equilibrium curve - these particular assumptions they generally do not hold except under very special circumstances. So, this is what you need to remember other than that the more or less the balance equations are the same. We write down the overall mass balance, the component mass balance.

From there we try to generate the operating curve the equilibrium curve, of course, we can generate separately and in that way we can proceed in this.

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Now, we know very well that for obvious reasons instead of number of mixer settlers and other can battery and it comprises of a large number of mixer settlers where the two phases are mixed and then separated and recirculated etcetera we would always like to go for a continuous operation. Naturally for in order to obtain or rather in order to meet the high volumetric flow rate requirements in most of the industries. This has also been discussed while you are discussing the gas liquid vapor liquid contacting devices.

So, therefore, for continuous operation just like we had discussed in gas liquid vapor liquid here also we are going to use the trays or the columns. And what are the different trays and columns? If you remember there we had discussed a packed column and we had discussed the tray columns. In the tray columns mainly you found out that the most common tray column that was used for distillation was bubble cap column.

But, in case of the extraction we generally go for the sieve tray, where, the sieve tray can be with or without down comers. We normally do not opt for a bubble tap tray due to its low efficiency and low throughput in case of extraction. Packed column is a very commonly adopted device and this packed column we adopted either with structured packing or with random packings.

Apart from these two columns if you remember that when you were discussing gas liquid flows a spray column was also mentioned to you. And it was told that in a spray column you have a column and then from here the one liquid is sprayed and the other liquid

flows up. Naturally, if you have a spray on the top and it is coming down, it is the heavier liquid which is being spread and the lighter liquid which goes up. This is generally not very common.

The only good part of it is it does not have too many accessories. So, therefore, it is cheaper, but it is not much used there is also another arrangement where you can use where you have a wetted wall column where one liquid flows as a very thin film and the other liquid flows through the between the annular film, but this is also rarely used. The most common devices which are used are packed columns and the sieve trays.

Now, one more thing also I would like to mention that when just only a very few theoretical stages are required ok or maybe the we are working with low to medium interfacial tension liquids. When where dispersion does not require a very high amount of energy for those cases we opt for the static towers.

The static towers what is the arrangement? Just let me show you this is a typical tower. In this typical tower normally what we have is that a liquid, it is the heavy liquid it flows through here and the heavy liquid it goes out from here. The lighter liquid it is normally it is flowing here and the lighter liquid it goes out through here. And normally here what we have is that we generally if we have a packed section we have a packed section here.

And remember one thing the either the lighter liquid or the heavier liquid can be dispersed as droplets and there can be different arrangements for dispersing them it can be a distributor it can be perforations etcetera that with different ways by which we can disperse them.

And depending upon which phase is dispersed if it is the lighter liquid which is dispersed the lighter liquid will be forming droplets. They will be rising up and then they will be coalescing and therefore, there will be a liquid interface there will be a liquid interface on the top of the column. So, when you have a liquid interface on the top you know that the lighter phase was dispersed.

In the same way if the heavier liquid is dispersed and it is coming down. So, as it comes down the droplets they coalesce and under that condition you will have a liquid interface at the bottom. So, when you have a liquid interface on the top it means that the lighter liquid dispersed.

This is the primary interface we say. Why we say? Because in case of flooding another second interface appears and that signifies flooding for the case of liquid-liquid extractors and when the heavier liquid is dispersed we will be having an interface here. So, this is heavier liquid dispersed we have the primary interface at the bottom.

And so, therefore, the arrangement for all the types of columns, it is it is always that the lighter liquid it goes up, the heavier liquid it comes down. Inside the column they are contacted and as has been already mentioned to you this particular contact it can be either through packings or through sieve trays.

And if I can draw a sieve tray the arrangement; see there are certain special things in the sieve tray that you have here. In this particular case, if I draw a sieve tray here the heavier liquid, suppose, it is being introduced through here, this is the heavier liquid in, this is the heavier liquid out and we have the lighter liquid in and here we have the lighter liquid out.

So, therefore, in this particular case we have a separator here. So, naturally we have trays like this. Wait I have I will just. So, we have trace like this just the way it was drawn there. So, therefore, from the heavier liquid comes here, it is flowing then it again comes and meets this particular downcomer and it keeps on flowing and from there it from here it goes down and it removes.

The lighter liquid, suppose, that is the dispersed phase if we feel, the lighter liquid in case it is the dispersed then that flows through the perforations here. As it is flowing through the perforations it forms droplets. These droplets as it moves it coalesces and forms as it coalesces and forms a layer like thing just beneath the tray.

Here we have a coalesced droplet layer and then again it gets redisperses. As it moves to the perforations of the higher tray and as it moves it as it exits the next tray it again coalesces and forms a layer of coalesces droplets just beneath the tray, re coalesces and in that particular way it goes on.

So, this was the case when the lighter liquid was dispersed. For the heavier liquid dispersed if you want to imagine this it is just the reverse. That means, if you just reverse this particular column then in that case the image that you are going to have that is the case which happens for the case of heavier liquid dispersed.

They get dispersed while they flow down. Just above the tray they form a layer and that that again gets dispersed and they keep on flowing as dispersed flow through the continuous lighter liquid so, therefore, these are the two most common arrangements for the static towers.

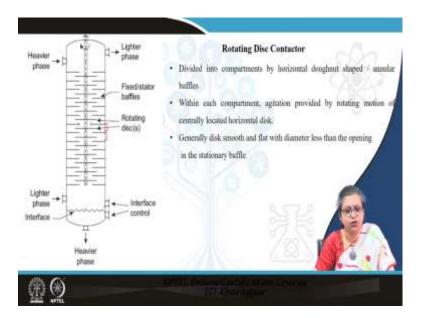
Now, let me tell you one thing that very frequently when we are going for this continuous operation for liquid-liquid extractors we find that we need a large number of theoretical stages. Why? Because in order to bring about the mass transfer good very large amount of theoretical stages are required and it can also happen that it is completely difficult to disperse the liquids due to their higher interfacial tension.

Now, for such a case if suppose we can give some sort of a motion or some sort of an agitation or some sort of a stirring to the liquid mixture quite naturally we find that the number of theoretical stages will definitely come down and it will be easy for us to disperse and get the mass transfer and along with that to get the required separation that we require.

So, quite often we find that in industries instead of static towers we go for agitated towers, where the agitation can be given either by some means of pulsing where some amount of air it is used as a pulsing medium. It can be used both for your packed column as well as for a sieve tray.

It can happen that we have a large number of trays and these trays they are given an up and down motion which gives some amount of motion to the liquid. It can also happen that we have a shaft and maybe a rotating disc which gives about the motion. Now, depending on the type of motion that is imparted, the moving part the columns they are named. The they are different types of columns.

I will be not going into details of this because naturally we do not have the time the most common type of agitated tower or agitated column which is used in industries is the rotating disc contactor. (Refer Slide Time: 27:56)



In the rotating disc contactor what we have? The same arrangement the lighter phase in that the heavier phase out. And this case as the interface is at the bottom its quite natural that the heavier phase was a dispersed phase. In this particular case the entire column it is divided into a large number of compartments.

Each of them are one-one compartment and they are divided by means of some baffles which are annular shaped baffles which have which divide the entire column into section. And in each section some sort of agitation is provided by means of the rotating discs.

Now, these rotating discs they are generally flat and they have a diameter slightly larger than the space here. So, therefore, each of these rotate each of these compartments they behave as if they are a mixer set mixer site of type of an arrangement. And for in each of these compartments good amount of stirring is provided and with that we find that the number of NTS, the number of theoretical stages comes down and this is particularly very effective for highly viscous products.

And therefore for furfural extract the lubricating oil extraction using furfural we use this type of a rotating disc contactor very frequently.

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Now, as I have mentioned to you in this particular case what do we do? For continuous operation columns be it static be it agitated there is a heavier phase there is a there is a lighter phase. The heavier phase comes, the lighter phase rises up, the heavier phase comes down and one of them is going to be dispersed as droplets into the other. This dispersion as we have found we can use nozzles, orifices, screens etcetera and we can also enhance this particular dispersion by means of agitators.

Now, the point is which particular phase are you going to be going to disperse. How do you decide depend regarding the dispersed phase? Now, if you recollect when you were to go discussing the gas liquid and the vapor liquid devices it was told that naturally that will be decided by the mass by the by the phase which controls the mass transfer resistance or the rather the phase which has a higher mass transfer resistance has to be dispersed in order to increase the interfacial area.

In this particular case there are certain other things that also need to be considered. Firstly, is the relative proportion of the two phases. In mixer settler arrangements I had already told you that the phase with a lower volume fraction is dispersed naturally it will require energy to disperse a smaller amount of the liquid.

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But in this particular case it is very interesting to note that in the case of sieve trays and packed towers the phase with the higher flow rate is dispersed and for all other the towers the phase with the lower flow rate is dispersed. Next thing is relative viscosity, this is something very interesting.

See the point is if you just try to imagine, suppose you are having the more viscous liquid as the dispersed phase then in that case what happens? This more viscous liquid it has to flow down through the less viscous liquid. Now, the terminal settling velocity will be higher through a lesser viscous medium.

So, therefore, the settling velocity will be higher. As the settling velocity is higher their coalescence will be faster. As a result, if we have the more viscous liquid as the dispersed phase we can opt for a higher capacity of the column. On the contrary if the less viscous liquid is dispersed quite naturally the diffusion rate through a lower viscous droplet will be higher. So, therefore, under this condition we will have a enhanced mass transfer which will result in an efficient operation.

So, there has to be a compromise between the two. Normally, we have the more viscous phase which is dispersed and emulsifying agents are added to increase the diffusion in the viscous droplets in order to give a higher mass transfer rate. Again we have to keep in mind that we do not want very fine dispersion. So, therefore, emulsifying agents have also to be increase used with some care.

The direction of mass transfer is naturally from the continuous to the dispersed phase. And when you are dealing with packings its important that the continuous phase has a greater weightability or it preferentially wets the packing material and also for sieve trays it preferentially wets the trays.

This is something which should be kept in mind. Otherwise definitely there will be some non uniform distribution due to nonpreferential wetting of the or rather preferential wetting of the dispersed phase of the tray internals. And the last option is of course, quite evident that when you have an inflammable liquid it is much more preferable that we disperse that particular liquid.

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Now, regarding, so, therefore now let us come to the different steps which have to be followed in order to design a continuous operation device may be a packed bed or a sieve tray or whatever it is. What will be the first step? First step is you need to have some liquid-liquid extraction.

So, a proper solvent has to be selected. And once the solvent has been selected the equilibrium data has to be collected over the entire operating range. Solvent selection was already discussed in your previous lecture. So, the same thing holds here. Next thing is that you have to find out once the solvent is selected the feasibility test and the equilibrium data has to be obtained from laboratory tests.

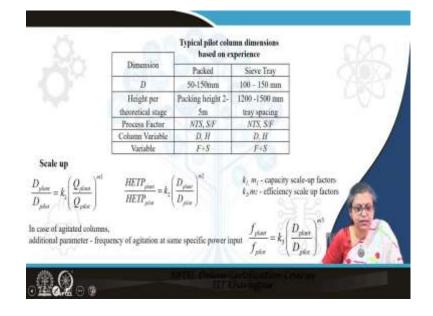
These as I have mentioned they can be done either in a back system or a cocurrent system also. After this the next thing is the contactor selection and then deciding on the dispersed and the continuous phase which we have just done just now. Now, once the contactor is selected then and you have decided on the dispersed and continuous phase the next work will be as I have told you to generate pilot plant data.

Because from the pilot plant data we are going to obtain the mass transfer data which will be used for your design of the actual column.

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So, therefore, regarding these things we are going to discuss in the next class.

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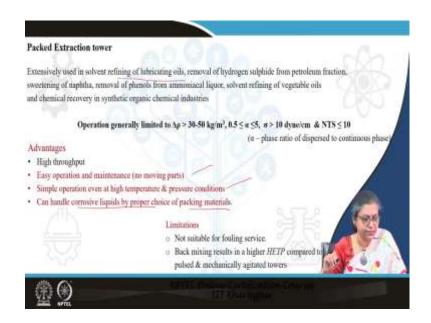


Today I will just be discussing the factors which are going to affect contactor selection and I am going to end this lecture. The next class will be discussing about pilot plant data and also the calculating the number of theoretical stages etcetera. Now, for the contactor selection we find that the factors which affect selection are naturally the density difference and the interfacial tension.

Interfacial tension high we go for agitated columns. Interfacial tension low we go for your static columns. When the difference the density difference is low we go for centrifugal extractors, otherwise we go for gravity separation and the other thing is number of theoretical stages. When you need a large number of stages we go for agitated columns when you need a smaller number of stages we go for static columns.

Certain preliminary guidelines are provided here. For example, for high number of stages an easy phase separation mixer settler. Static columns I have already said easy separation, low NTS, agitated columns for moderates and high NTS. For this high NTS we go for agitated columns.

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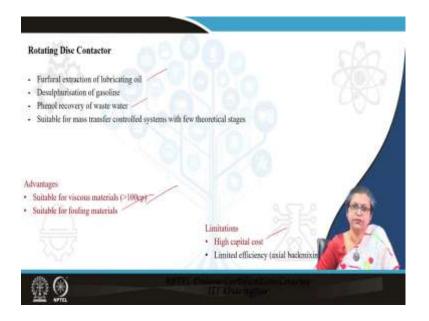
There are certain specific applications for which packed, sieve and RDs are used. Now, whatever are the salient points in each of these slides? We will find that they are marked in red, the points which you need to remember for your assessment tests. The there are other information which you will be requiring for your capstone project or for other references. We would also elaborate on them. If in case you have doubts, come back to us.

So, naturally packed extraction quite naturally it has no moving parts, a great advantage. Even at high pressure temperature simple operation. And very important if you choose the packing material properly you can handle the corrosive liquids. There are certain limitations of the operation and they and they are used very frequently for your refining of lubricating oils and etcetera.

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Sieve tray it has a high capacity good efficiency not much. And rotating disc as I have already mentioned it is very suitable for viscous material as well as for fouling material. Naturally, if you have agitation the capital cost is going to be high and it is as I have already said it is the conventional technique for extracting the aromatics from lubricating oil by using furfural. It is also a standard technique for phenol recovery of wastewater etcetera.

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So, with these few words [FL] mixer settler I had forgotten to super mention. Mixer settler had also said that definitely the limitations are large floor space. The advantage is less head room, low capital cost and the limitation is low throughput. So, based on all these things first we are going to select the contactor type.

We have already selected the solvent. We have generated the equilibrium data. Now, with all these we will go for a pilot plant study and then we will be going for designing the actual column. In the designing of the actual column just like the gas liquid or vapor liquid columns what are the things that we need to decide on? The tower height, the tower diameter.

For the tower height we need to find out the number of theoretical stages and tower diameter we will be estimated from flooding considerations. And then once we have done that then we go to look into the column internals and mostly they are they are decided from the thumb rules which are conventionally used in industries.

So, with this we end today. We continue the last lecture on liquid-liquid extraction in the next class.

Thank you so much.