Principles and Practices of Process Equipment and Plant Design

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Module - 02
Lecture - 36
Liquid-liquid extraction (L2)

Hello and welcome to the second lecture on extraction. In the last class what we did? We discussed the challenges involved in extraction and based on the challenges or keeping the challenges in mind, we had small discussion regarding the choice of solvent. Today we will be going for the contacting devices. What is the simplest contacting device?

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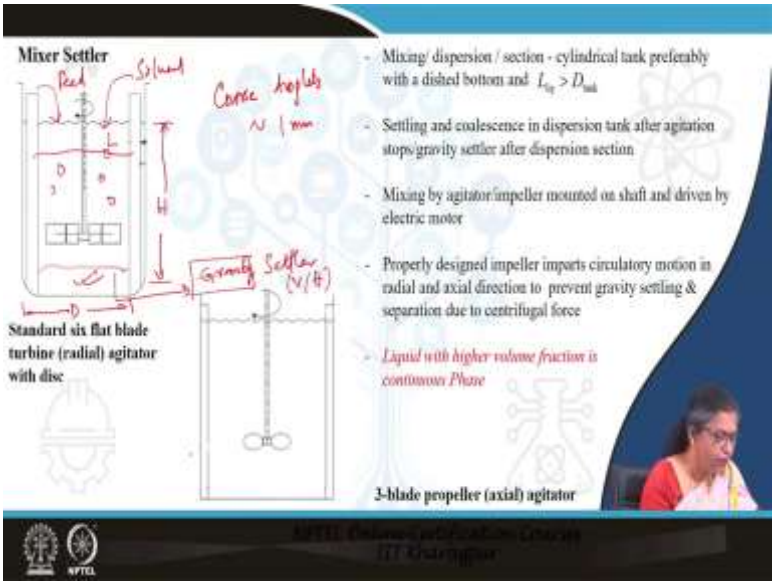

You have maybe a vessel and or a cylindrical tank in that normally we have a cylindrical tank with a dished bottom. And usually what we do is - the depth of the liquid, say, is taken as H. This is generally equal to the diameter of the tank.

Now, in this particular tank what you do? We introduce the two liquids, we introduce the feed and we introduce the solvent and then they are agitated here. This agitation takes place either by means of an impeller or a propeller and which is powered by an electric motor. After they are agitated, naturally what happens?

You have given two particular phases here. Out of these usually one phase gets dispersed as droplets in the other phase. Generally, we find that the liquid which has a lower volume fraction among the two that forms the dispersed phase. If feed is in a lower proportion that gets dispersed if the solvent which is generally the case is the lower proportion then that gets dispersed.

Generally, we prefer the solvent to get dispersed so that greater amount of the solute can diffuse from the feed into the solvent. So, once then by means of the agitator or the impeller which start that the two fluids and naturally the one of the fluids with the lower volume fraction that gets dispersed. And then the churning goes on, mass transfer happens. And after the extraction has taken place, we stop the operation and we need to separate the two phases - the extract and the raffinate phase.

Now quite often this mixer and the separation they occur in the same particular tank; this can happen that after the mixing is over the settling happens. It can also happen that after the operation, from here we take the mixture into a gravity settler and in this particular gravity settler the two phases they get separated as per the gravity. This gravity settler it can be either a vertical or a horizontal tank. And generally we try to ensure that the inlet velocity is not very high such that there are not a lot of interfacial disturbances which facilitates the separation.

Now when we are designing this mixer settler what are the things that we have to consider? For the mixing part, first thing is this is if this is a batch process, then we need to know what will be the volume based on what is the time of contact and the volume of the two. If it is a continuous process the feed and solvent are entering and the extract and raffinate are being withdrawn out. We need to know the residence time of the mixture in order to effect a proper extraction or a proper separation of the solute from the feed.

So, based on this particular time the dimension of the mixer is decided. And what about the settler? If the settling has to occur here itself, then for the settling we need to consider the droplet size; because for settling what is required? There were large number of droplets which were formed, all these droplets they need to coalesce. After coalescing if the dispersed phase was the lighter phase then in that case we will have the droplets coalesce and they form interface on the top. If it is the heavier phase, then it forms, it

settles down and forms an interface at the bottom after that both of them are separated out.

So therefore, for the mixing phase we need to design the total holding capacity and for a proper mixer, the design of the agitator is very important. These two things we need to consider. And for the settling part, we need to consider the droplet size. Definitely as I have mentioned we would not like to go for very fine droplets because although it gives a proper mass transfer, they take a very long settling time; the smaller the droplet let the greater is the settling time.

So, normally we would or like to opt for extraction purpose, we would like to opt for coarse droplet whose sizes are near about 1 millimeter. And based on that the settler is designed.

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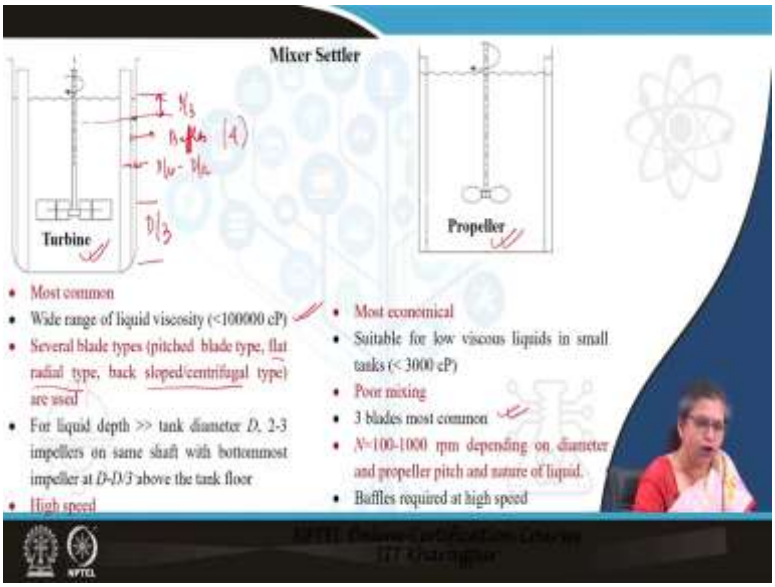

Now, regarding the agitator part, normally as I have told you, we go for a turbine or we go for a propeller, most common is turbine, quite naturally it can be used for a wide range of liquid viscosity, several types of designs are possible. We can have the pitched blade type where the blades are inclined at 45 degrees and they can give both axial as well as radial dispersion.

We can also have a back sloped or a centrifugal type which is good for handling difficult dispersions. And we can also have a flat radial type which is the case in this particular

case where the blades they are connected to a disc. And when we have a very large depth, then in that case instead of one turbine we can have a number of multiple turbines.

Under that case usually what we find, lowest turbine is at a height of say D by 3. The turbine at the top is at a height of D by 3 from the top and mostly the other turbines they are D-D by 3 and they are equally spaced.

Propeller we do not opt very frequently, the only good part about it is that it is cheap and when we opt that, 3 blades are most common. The other thing also which we need to remember is that whenever we are churning a liquid quite naturally vortexes are formed we would like to eliminate the vortices. Now if we have to eliminate the vortices what we have? We have the baffles. What are this baffles they are thin strips of metals which are fastened on or welded on the tank wall.

Generally, we go for not more than 4 baffles, we have found that 4 baffles are sufficient. This thin strips of wall and I will be giving you the design parameters that more or less their height is equal to the liquid depth and their thickness is about say D by 10 to D by 12; where D is the diameter.

And so, these baffles they are more or less equally spaced around the circumference, usually we have found that 4 baffles are sufficient. So, therefore, these baffles are also there in order to eliminate the vorticity part. So, therefore, you know that when we are going for mixer settler device or equipment for extraction, we first need to size the vessel then we need to design the impeller and we also need to decide the dimension of the baffles.

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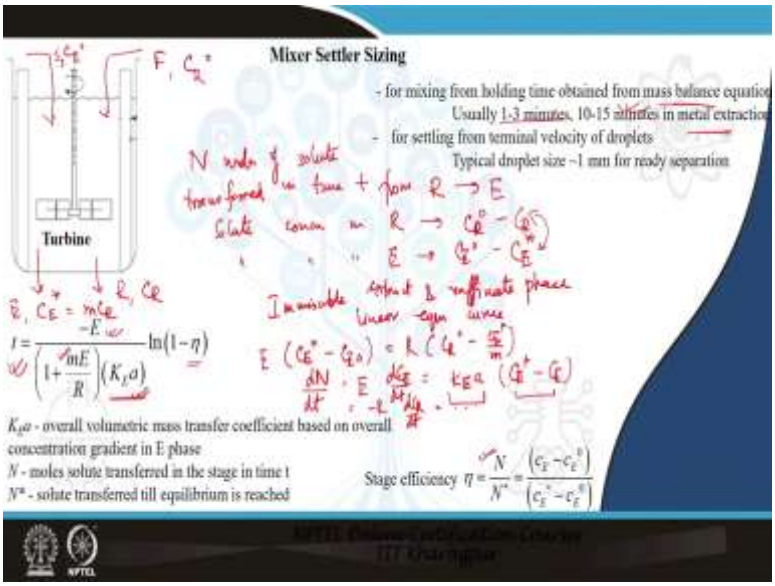

Let us go one by one. Initially suppose we go for the settling as I have told you that the settling is a function of the holding time; the time which is required for the mass transfer. So therefore, the holding time we can obtained from the mass balance equations. So, in this particular case if you would like to find the holding time. So, therefore, what we think?

Suppose we introduce the solvent, the solvent is S. The concentration of the solute in the solvent that is say C E 0 and the extract which comes out that has a solute concentration of C E. The feed comes in it has say the solute concentration of C R 0, the raffinate it comes out it has a concentration of C R; we assume that the solvent and the feed liquid they are completely immiscible with one another.

Then we are expressing these concentrations in terms of solute free bases, then R becomes equal to F, S becomes equal to E. And we find that the concentration it changes in the solvent or in the extract the concentration changes from C E 0 to C E, in the raffinate the concentration changes from C R 0 to C R and generally this C E this is in equilibrium with C R.

So, therefore, if we can assume a linear equilibrium curve under that conditions C E star will be equal to m C R. Now during this holding time if we can assume that N moles of solute are transferred in time t from the raffinate R to the extract E.

So, with this N moles of solute transfer what happened? This solute concentration in raffinate it changed from C R 0 to C R. And the solute concentration in the extract that changed from C E 0 to C E star I would like to put, because under ideal conditions this is going to be in equilibrium with this.

And I assume that, since I have already assumed that the solute free flow rate it is same for both these solvent as well as the feed. So therefore, I can assume that they are the C R and C E star they are connected by a linear equilibrium curve within the operating range fine.

So under this condition when we can assume that immiscible extract and raffinate phase, and also a linear equilibrium curve. Under these conditions if you write down the mass balance we are going to get E equals to C E star minus C E 0 which is equals to R into C R 0 minus C E star by m.

Now during a differential time t d t the total amount of solute that has been transferred that is naturally equal to d C E d t which is equal to from mass transfer considerations K E a C E star minus C E. If suppose instead of considering the extract you would have considered the raffinate then in that case also we could have written it down and we could have expressed in terms of K R a etc.

So therefore, from this particular equation we find that the rate of transfer of solute from the raffinate to the extract phase, that can be expressed in terms of K E a, which is the overall volumetric mass transfer coefficient based on the overall concentration gradient in the extract phase.

So, once we can integrate this we can separate the variables, we can integrate this particular equation, we get an expression of holding term holding time in terms of this expression. We have obtained it in terms of the extract flow rate the equilibrium, the slope of the equilibrium curve, then E by R and the overall mass transfer coefficient. And we have expressed this in terms of a stage efficiency where the stage efficiency defines the actual amount of solute which has been transferred and the amount of solvent which would have been transferred had equilibrium been obtained under this conditions.

So, using this expression we can find the holding time, from this particular holding time we can find out the; once we know the flow rate we can find the total amount or the

minimum amount of volume that has to be provided in this particular tank. Now here that is something very important that I would like to tell you.

See so, for finding the holding time we need data on the overall volumetric mass transfer coefficient which demands the data on mass transfer coefficient as well as interfacial area. Now, this is not usually readily available. And again we have to remember that whatever solvent you take and whatever feed you take therefore, there will be some amount of contaminants, some amount of surface active agent, something will be there with every such sort of contamination the mass transfer coefficient is going to change drastically.

So, normally we do not have data on this and just like I had mentioned in absorption in this case also it is the same. Very frequently we generate data on mass transfer an interfacial area from the pilot plant and then we use some empirical relationships for scaling up. The scaling up is usually done based on geometrical similarity.

So, this is one thing that you need to remember whenever we are discussing any sort of extractor, it is very important that we discuss the scale of relationships as well. So, anyhow. So, this is the way we find out the holding time. Usually it is important to remember that generally the holding time of 1 2 3 minutes are sufficient. Only when we go for metal extraction for which mixture settlers are quite often used due to the extractive reaction, the holding time can be much more larger about 10 to 15 minutes.

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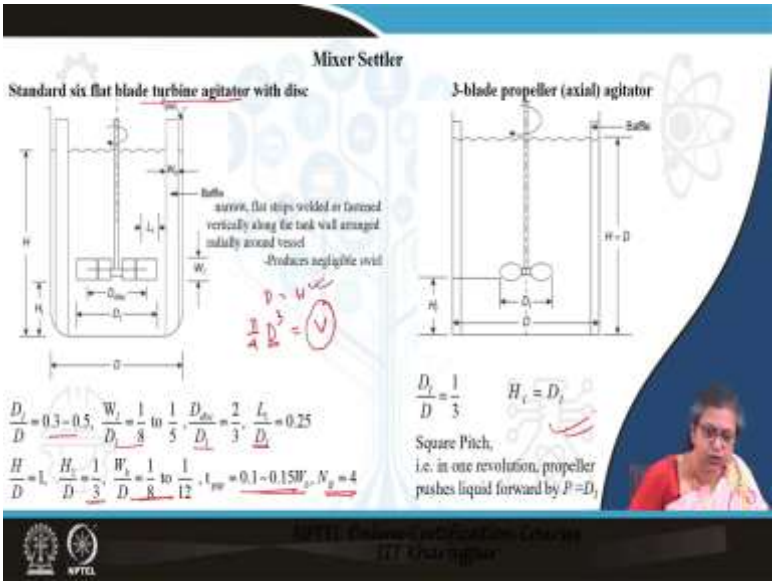

Well, once the volume has been decided. So, generally we take D equals to H as a result of which the volume is given by pi by 4 D cube equals to the volume from above. Once the volume is decided we can find out the diameter of the column. Once the diameter of the column is fixed, we will be doing a problem we will find, we can find out the height of the vessel. Once the height is decided then we can go for the impeller design.

Usually these are more or less thumb rule types and more or less the diameter of the impeller there are some fixed particular ratios for the diameter of the impeller, the width of the impeller, the diameter of the of the disc and also the length of the impeller. And the height from the liquid bottom where the impeller is kept; as I have told you that this same H 1 is maintained if the impel if there are multiple impellers, the top impeller from the liquid interface is also maintained at the same height. The baffle width, it is around I had mentioned D by 10 to D by 12 it is about D by 8 to D by 12 and generally the gap is also mentioned. And the number of blades that we have that is equal to 4.

So, generally we adopt the turbine agitator and if we adopt the 3 blade propeller agitator also the designs the design parameters, the parameter the geometric parameters are specified in here itself. Well, once we have decided the diameter and the height of the column we have completely designed the turbine agitator.

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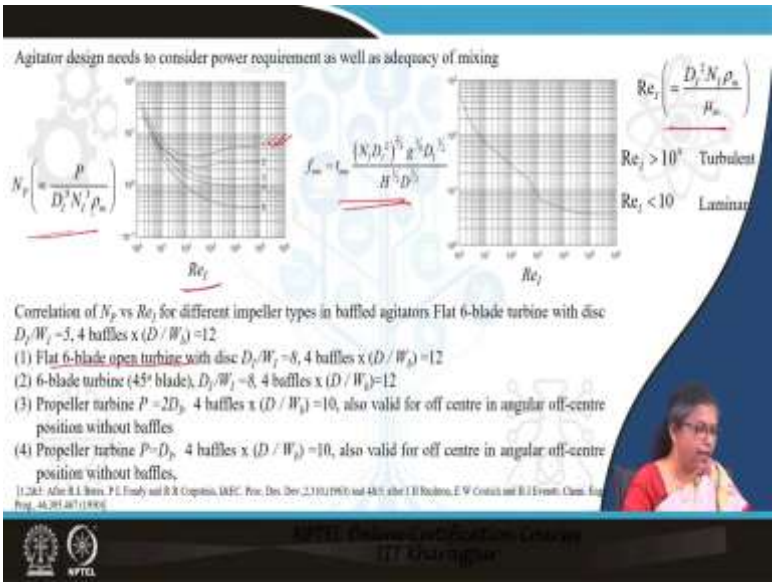

The next thing that we need to know as I have already told you that it is powered by an electric motor. So therefore, we need to know the power requirement and we also need to

know how good a mixing that it is doing which we quantify by means of a dimension this mixing number.

Here, you please remember that if the agitator demands a higher power it is not necessary that it that it will promote a greater amount of mixing. They are not completely related because it can happen that we have a higher power a very fine emulsion is forming, but the mixing may not be so very good. So therefore, if you find that more or less the power requirement and the adequacy of mixing they are obtained from some empirical relationships which are based on certain dimensionless numbers.

For example, the power requirement is obtained from a power number and the adequacy of mixing is obtained from a dimensionless mixing number. And both of these they are plotted as a function of the impeller Reynolds number which is given as a function of the number of blades, the impeller diameter and the mixer properties of the liquid inside this.

Here, if you find in the power number versus Re I plot you will find a number of graphs, these graphs are for different-different sort of turbine impeller and propeller turbines. Normally we would we would be opting for the flat six blade open turbine and so, we will be; we will be referring to the graph one.

Now one thing you have to remember not only in design, but everywhere in chemical engineering if you are going for any empirical correlations then it is very important that you know the units of each and every parameter that you are using here.

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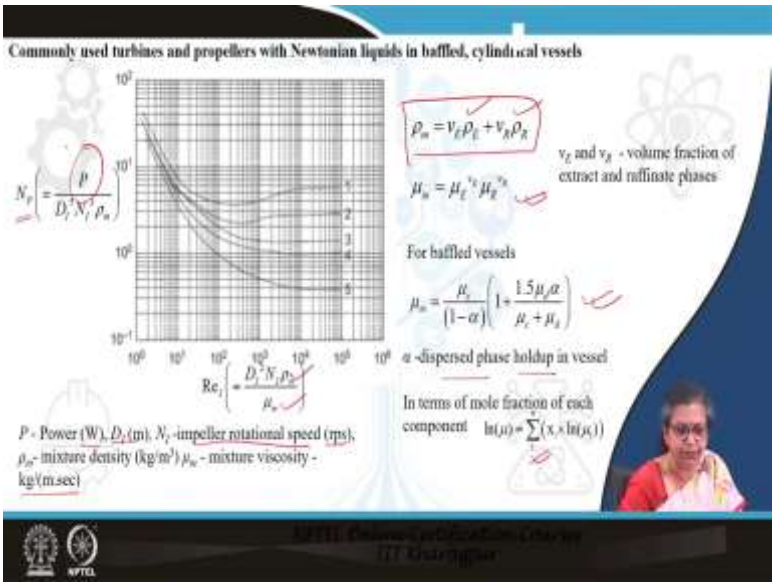

So, therefore, you have to remember that when we are plotting this particular graph, this is applicable for a power number where power is expressed in watt, the impeller diameter in m; it is the impeller rotational speed - it is in revolutions per second, mixture density kg per meter cube, mixture viscosity kg per meter second. These are all expressed in terms of SI units.

Now if you have to find out the power number, you need to know this rho m and mu m both for Re I as well as for N p. Finding out rho m it is not very difficult you can just find it out from the volume fraction of the extract and the raffinate phases, if you know the densities you can find out the mixture density.

Mixture density for any particular mixture it is not difficult, but finding out a mixture viscosity it can often be challenging. And that is the reason you will find that here I have expressed mixture viscosity in three different relationships. Where the first one expresses mixture viscosity in terms of volume fraction of extract and raffinate, the other one expresses it in terms of the holdup of the dispersed phase in the vessel, and the third one expresses the mixture in terms of mole fraction of each component.

Depending upon the data which is available we can select anyone the any one of them. Once we have calculated rho m and mu m we can find out Re I. Once we have found out R e I we go to the proper graph, we find out N p. Once we have found out N p we found out the power required.

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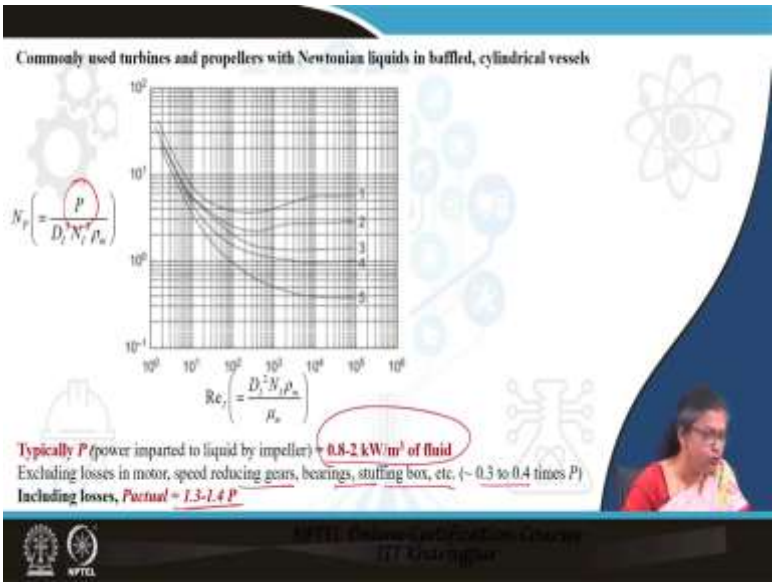

But remember one thing the power required which you have found out is actual the actual amount of power provided per meter cube of the fluid excluding the losses in motors, speed reducing, gears, bearing, bearings stuffing etcetera. All these losses they combine to form about 0.3 to 0.4 times the power.

So, therefore, from here the power which you are going to find out the actual power required will be around the Pactual is going to be around 1.3 to 1.4 times. This will give you the actual power which will be required to write the impeller.

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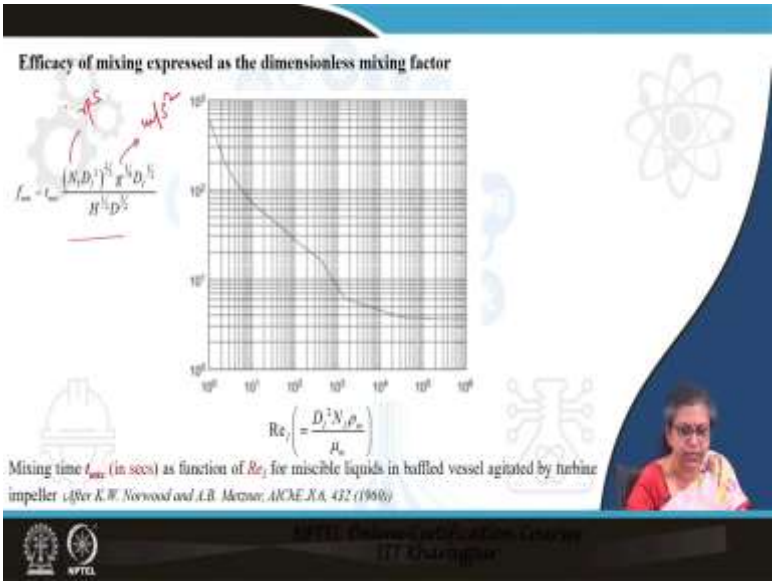

Well, regarding the efficacy of mixing again in this particular case you will find that more or less it is expressed in terms of certain para the geometric parameters. And again for all these parameters the SI units have to be maintained g is expressed in terms of meter per second square everything else is in meters and this is in terms of r p s. So, from here also we can find out the t mixing in seconds.

Well, one thing regarding the power I just forgotten to mention after you have calculated the power it is important for you to remember certain checks, which we do for all design. So, just check up whether the power more or less confirms to this particular value or not.

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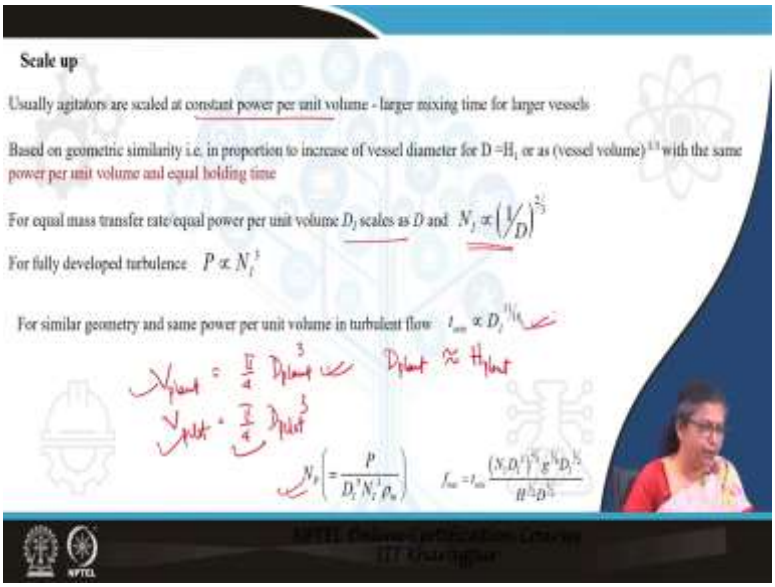

Well, once we have designed we have we have selected the vessel, we have designed the impeller, we have calculated the power now generally as I have told you in order to find out the total volume or to find out the diameter of the of the column if you remember, we needed to find out the holding time to find out the holding time we needed some idea regarding the mass transfer coefficient and the interfacial area which is usually not available as I have told you.

So, generally for scale up what we assume, we assume that for the larger vessel and for the smaller vessel both of them are operated at constant power per unit volume. And then based on geometric similarity what we do? We assume that even for the larger plant also it is equal to pi by 4 D plant cube and for the smaller plant in the pilot plant scale also it is equal to pi by 4 D pilot cube.

So, therefore, if we know this, we know this, we know this, then we can find the diameter of the actual plant. Once we know the D plant as I have told you usually we assume this to be equal to the height of the vessel in the actual plant. Once we have done it then we come to the impeller design. More or less the impeller design is that D I it scales as D and the r p s the revolutions they scale in this particular by this particular relationship. And when the we have fully developed turbulence, the power it can be scaled if you see if the power number is the same then the power can be scaled as N I cube. And the t mixing again from here if you can see that t mixing scales by this relationship with the impeller diameter.

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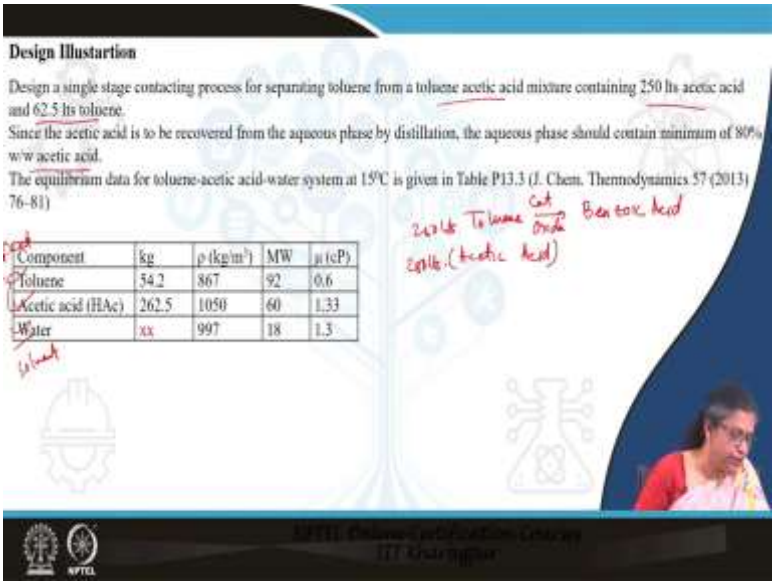

So therefore, this completes the entire discussion on the design of a mixer settler design. We have just considered the mixer part, if the settler has to be there as I have told you; the settler has to be designed or the time has to be decided based on the time of settling or the time of rise of the disperse droplets to form a separate phase.

Now, whatever I have discussed we would like to go for a illustration problem in order to demonstrate the design of the mixer settler that I have told you. What do I have here? I have got a toluene, acetic acid mixture. It contains 250 litres acetic acid and say 62.5 meters of toluene. Now generally this does not look very good that 62.5 litres etcetera I will tell you the genesis of the problem.

The genesis is very frequently what we do we use toluene and we perform its catalytic oxidation to obtain benzoic acid. And for this particular reaction the toluene it is dissolved in acetic acid and cobalt acetate is taken as the catalyst. So, what we did? We started with 250 litres of toluene and 250 litres of acetic acid the reaction occurred and about 75 percent conversion of toluene took place.

So, then we were left up with a benzoic acid, the unreacted toluene and the acetic acid. This was cooled by putting it in cold water, the benzoic acid crystals are separate out. And we have this particular the mixture where we have toluene as I have told you 75 percent is reacted, so the remaining amount of toluene is 62.5 litres and it has 250 litres of acetic acid. So, therefore, we would like to separate the acetic acid from the toluene so, that this toluene can be reused again.

What do we do? We know that acetic acid is highly soluble in water. So therefore, we add water to this mixture and we try to separate acetic acid. There is something that you have to remember before you are going to do this problem, these are practical aspects which you need to remember.

Water and acetic acid it forms an azeotrope at 80 percent weight per weight of acetic acid. So, you have to remember that when you are using water for extracting acetic acid the amount of acetic acid extracted has to be greater than 80 percent weight to weight.

Accordingly, we have to design the extractor such that in the solvent recovery, when the extract and the raffinate they come to the solvent recovery unit, the water contains at least 80 percent weight to weight of acetic acid. How do we go about the problem? I the all the input data etcetera are given. So, you have this comprises of the feed and you are going to give water here for doing. How much water will you give?

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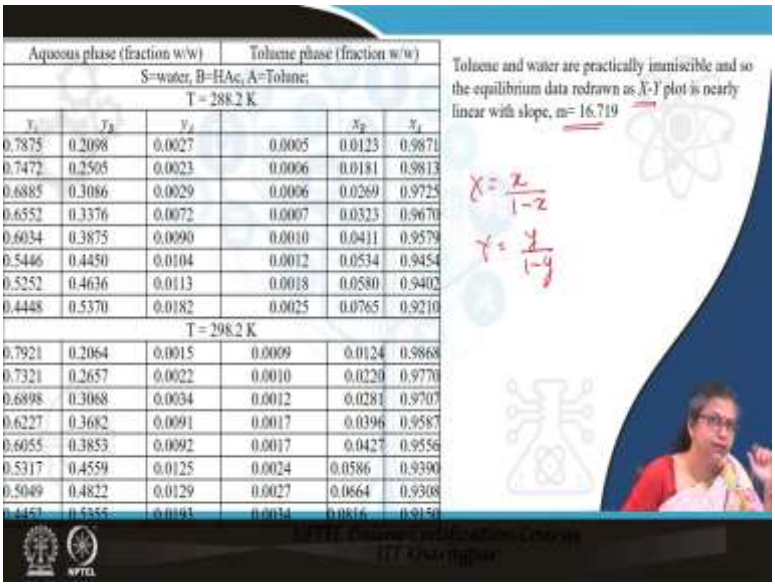

We have seen that I will show you the equilibrium data if you see you will find that more or less toluene and water they are practically immiscible. So therefore, I have not drawn it here, if you can represent the equilibrium data in terms of mole ratios where you already know the mole ratio is defined by this. The mole ratio of acetic acid in the toluene and the water phase, you will get a linear graph with m equals to 16.719. So, for this case we get a linear operating curve.

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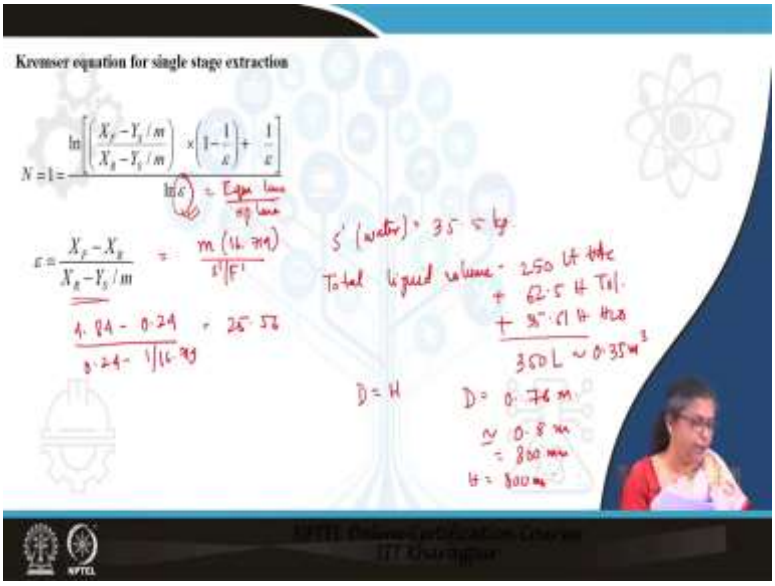

And as a result we find that we can use the Kremser equation if you remember which was which was discussed with you in the class of absorption we will also be going into some discussions on it. We can use this particular Kremser equation. There it was defined in terms of an absorption factor which we replaced by means of an extraction factor here and naturally the extraction factor is the slope of the operating to the equilibrium, it is the slope of the equilibrium line to the operating line.

So, what we do? We use this particular equation to find out epsilon. This is, once its simplified for n equals to 1 we get this. Once we know epsilon we know this epsilon what is this, this is equal to the slope of the operator of the equilibrium line; the slope I had already mentioned it is 16 points 16.719 divided by the slope of the operating curve.

What is the slope of the operating curve? It is naturally equal to S prime by F prime where both of this is the moles water and this is the moles toluene. So, we know the moles toluene. So, we can find out the moles water from this particular equation.

So, then we can find out what is the amount of water which will be required in order to effect the separation. And then we can find out whether in the final mixture of water acetic acid; what is the proportion of acetic acid and water just in order to check that the acetic acid is higher than 80 percent weight to weight. So, from here if we can substitute all the values see X F if you can take you can just find it out X F is equals to 4.84, the final X R was 0.24 and this become 0.24 minus 1 by 16.719 from where we get the extraction factor is 25.56.

Then if you substitute it in this particular equation, we know that the total amount of feed that I have already shown you here, the total amount of feed it is toluene is 54.2 kgs. So, therefore, if you are substituting it here, we get the total amount of water that will be required S prime which is nothing but the water in this particular case that will be around equal to say about 35.5 kgs of water we will be requiring in this particular case.

So, therefore, what is the total liquid volume in the vessel in that particular case? The total liquid volume will be 250 litres of acetic acid plus 62.5 litres of toluene plus 35.5 997 was the density. So, it is around 35.61. So therefore, this much litres of water. So, totally you get around 350 litres is the total liquid volume which gives you around 3.35 meter cube of the vessel volume. Its actually 348.1 I have just rounded it off.

Now from this if you consider D equals to H then you get the diameter as 0.76 meters based on the course we make it 0.88 meters or it is equal to 800 millimeters which also gives you H equals to 800 millimeters.

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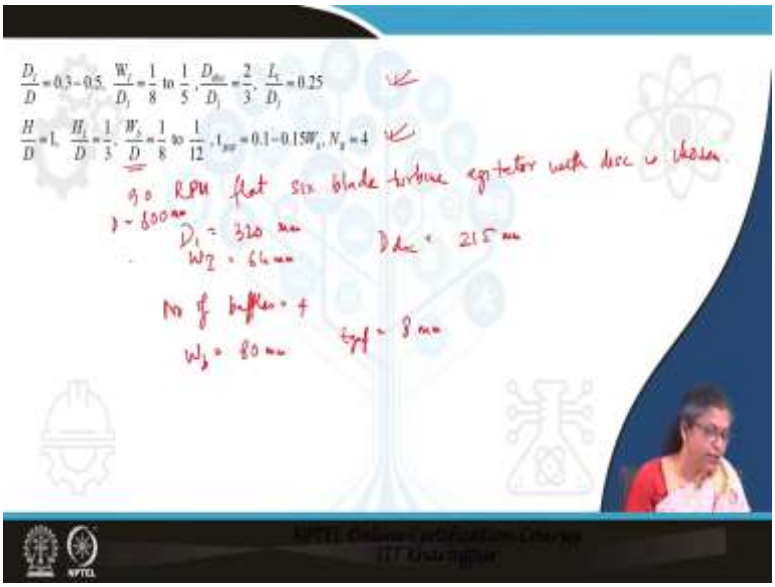

Now once the vessel dimensions are fixed then we go for designing the impeller. If for this particular case we assume a standard 90 RPM flat 6 blade turbine agitator with a disc for that we have all the data this is used. So therefore, we select this particular turbine. For this turbine we have already mentioned all the geometric parameters they are already mentioned in this particular case.

So, based on that you can calculate D I, you can for D equals to 800 millimeters you can calculate D I you will be getting as 320 millimeters W I you will be getting as 64 millimeters. In this particular way you can keep on calculating number of baffles that is equal to 4. D disc will be equal to say about 215 millimeters roughly and then the w b the width of the baffle will be 80 millimeters. Just I am simply substituting everything in this particular equation the gap will be around 8 millimeters etcetera.

So, once you have designed you know the vessel dimensions you have designed the impeller.

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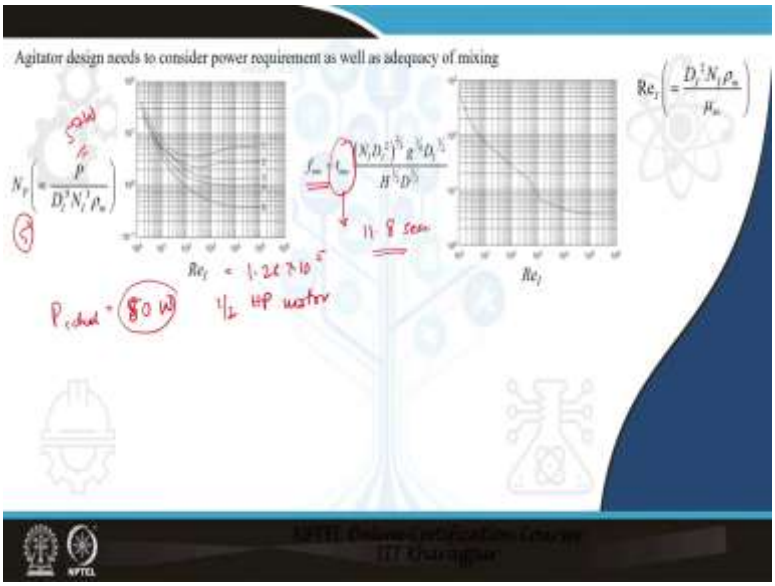

Next what you need to know? You need to find out the power required. So, from this particular case you have to find out Re I. Re I for the present case you can check up I have got something like 1.26 into 10 to the power 5, from there you can find out N p roughly around 5 means, it was roughly I found out it was around 5 from there I found out P. This P was equal to 57 watts and therefore, the actual power I found out P actual that was roughly equal to say around 80 watts.

We know that the; that this is quite a low power, so half horse power motor would have been sufficient for this. And this is quite expected because the mixture volume is also not high and then the system viscosity is also not high. So, therefore, the power requirement is very low. And so therefore, just a half horse power motor will be sufficient to drive this.

Next we try to find out f mix. Accordingly, we have found out of we have substituted all the parameters and then from Re I we have found out f mix, from f mix we have found out the t mix, we have found out t mix to be roughly equal to 11.8 seconds you can check it up for yourself. We find that the mixing time is also not very high. So therefore, the design is fine.

Now if you want to go for a larger scale quite naturally you will have a larger volume. According to the volume the diameter of the vessel has to be designed, once the diameter

is designed the height, the impeller, the power the power required will be kept the same. So therefore, everything else can be designed accordingly.

So, this was just the simplest type of extractor which is more or less by a single stage contact we could take it up. We have adopted a single stage contacting device because in this particular case the selectivity of water was very high. So therefore, single stage was sufficient.

For most of the cases we will find that a single stage will not be sufficient we need multiple stages. Multiple stages can be provided by a number of mixer settlers in series or more commonly by a continuous operation device a packed column or a sieve tower. But the design of packed column and sieve tower for extraction and for distillation or absorption are completely different.

So, in the next class what we do? We take up the continuous operation devices and we try to find out or rather we try to discuss the how to find out the number of theoretical stages, how to find out the diameter of the column etcetera. We will find that the basic theory is the same, but since there are certain intrinsic difficulties or complexities when there are two liquids instead of a vapour liquid mixture, the design equations will be more involved at the same time slightly more interesting.

So, we discuss them in the next class.

Thank you so much.