Principles and Practices of Process Equipment and Plant Design
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Module - 02
Lecture - 38
Liquid-liquid extraction (L4)

Well so, good day to you all. So, we continue with our discussions on liquid- liquid extraction and this is the last lecture which we are going to have on liquid liquid extractions. So, therefore, if you remember in the last class we had started the continuous operations - the extractor under a continuous operation.

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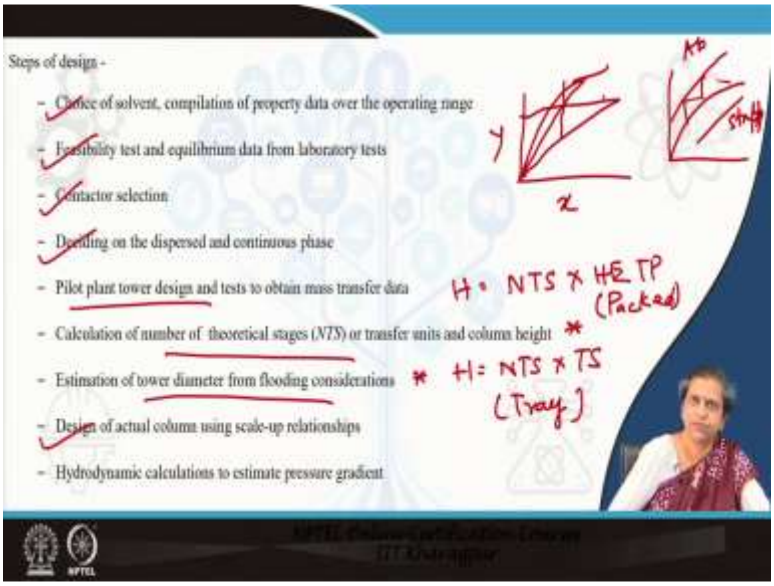

Steps of design -

- Choice of solvent, compilation of property data over the operating range
- Feasibility test and equilibrium data from laboratory tests
- Contactor selection
- Deciding on the dispersed and continuous phase
- Pilot plant tower design and tests to obtain mass transfer data
- Calculation of number of theoretical stages (*NTS*) or transfer units and column height *
- Estimation of tower diameter from flooding considerations *
- Design of actual column using scale-up relationships
- Hydrodynamic calculations to estimate pressure gradient

H = NTS × HETP
(Packed)

H = NTS × TS
(Tray)

So, in the continuous operation the steps of design had been mentioned to you and we had also completed or rather we had already discussed the criteria for choosing the solvent and compiling their property data over the entire operating range that you would like to take.

Operating range includes the fit composition and the raffinate composition to which you would have to go and corresponding to that the concentration of the solute in the fresh solvent or the solvent which is being used to start with and the final composition of the extract solution. So, this comprises the entire operating range over which the extractor has to operate for one particular operation.

So, therefore, the criteria of selection of solvent is the same; it has been discussed in the very beginning of the extraction class in the first lecture on extraction. Once it is done, then you need to do a feasibility test and you have to collect equilibrium data from the laboratory.

Now this particular exercise is quite important as we will see in one of the illustration problems that will be discussing as I proceed further. Next we have supposed to decide upon the contactor selection that I had also mentioned and you will be remembering that when we will be opting for a mixer settler for most of the cases particularly when the throughput is high we would like to go for a continuous operation, among the continuous operation we can select a packed bed, we can select a sieve tray tower.

And suppose, the number of theoretical stages are very high or else the interfacial tension is say moderate to high as we had already discussed we would like to go for any type of an agitated contactor. Now, regarding the agitated contactor also I had mentioned and I had shown you there are different types of agitated contactors, I did not go into the details of all these things, we had just discussed simply the basic structure of a rotating disc contactor.

But if in any case any of you are interested to know about the Cybil contractor, the pulse tray contactor or maybe the car reciprocating contactor, you put up your doubts in the forum or contact us, we would be clarifying the doubts accordingly.

Well, after the contactor selection you have to decide on the dispersed phase and the continuous phase. This also in the last class we had discussed about it, that the main factors are interfacial tension, relative viscosity and the flow rates, and I had also mentioned that the higher flow rate is dispersed for the packed bed and the sieve tray, and the lower flow rate is dispersed for all other types of tray.

And also I mentioned that normally although for gas liquid and vapor liquid contacting particularly for vapor liquid contacting in distillation we often prefer the bubble cap tray, but in this case due to low throughput, low efficiency of the bubble cap tray we do not go for the bubble cap tray in this particular case.

Now, after this the next thing which is very important is the pilot plant design to obtain the mass transfer data, for mixer settler if you remember I had derived the basic equation for the holding time, we found that the holding time was a function of mass transfer coefficient and interfacial area.

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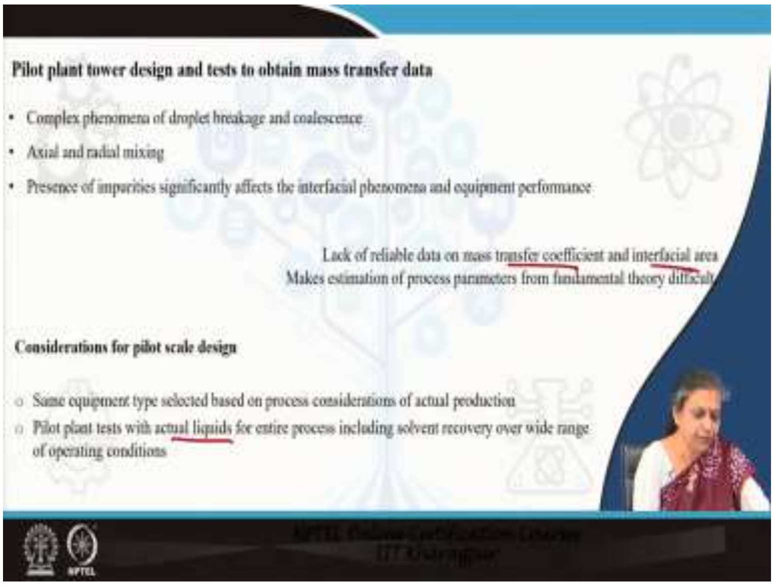

Pilot plant tower design and tests to obtain mass transfer data

- Complex phenomena of droplet breakage and coalescence
- Axial and radial mixing
- Presence of impurities significantly affects the interfacial phenomena and equipment performance

Lack of reliable data on mass transfer coefficient and interfacial area
Makes estimation of process parameters from fundamental theory difficult

Considerations for pilot scale design

- Same equipment type selected based on process considerations of actual production
- Pilot plant tests with actual liquids for entire process including solvent recovery over wide range of operating conditions

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And I had also mentioned that just because in this case there are large number of uncertainties, why? Because extractor involves the complex phenomena of droplet breakage and droplet coalescence which is itself not an extremely predicted phenomenon, other things are - there are axial mixing and the radial mixing.

Then see, we cannot avoid any sort of impurities whenever there are any sort of impurities it significantly affects the interfacial phenomena as well as the equipment performance. Quite naturally due to all these things there is a lack of reliable data on both mass transfer coefficient as well as interfacial area.

As a result, the estimation of process parameters from the fundamental theory is from the basic mass transfer equations. The equations which you have learnt in your mass transfer class that is extremely difficult. And so naturally, what we would like to do in order to obtain data on the mass transfer coefficient as well as on the interfacial area, generally we perform the tests in the pilot plant.

Now, when we perform the tests in the pilot plant, two things are important that we should remember, first thing is we have selected the contactor earlier itself. So, therefore, the pilot plant tests have to be conducted in the same equipment type based on the process considerations of the actual production.

If we plan that well for this particular case we would have to go for a packed tower contactor, the pilot plant data will also be collected in the packed tower data and the other important thing is the selection of solvent. Frequently we find that we select a solvent and for the pilot plant test we operate or we perform these tests with a synthetic fluid which has the same properties as that of the actual solvent, this often results in unsatisfactory results.

So, therefore, it is important for you to consider that the pilot plant tests are also performed with the actual liquids and when we are performing pilot plant test we should be performing both the extraction as well as the solvent recovery over a wide range of operating conditions.

So, it is just to emphasize upon you how important the solvent recovery part is for the extraction process, we cannot think of the extraction process in isolation which I have mentioned earlier.

So, even for generating data for the pilot plant we just need data on the mass transfer coefficient and interfacial area, which has nothing to do with the solvent recovery, but then also it is advisable that the pilot plant tests are performed over the entire process including both the solvent including both the extraction as well as the solvent recovery over a wide range of operating conditions.

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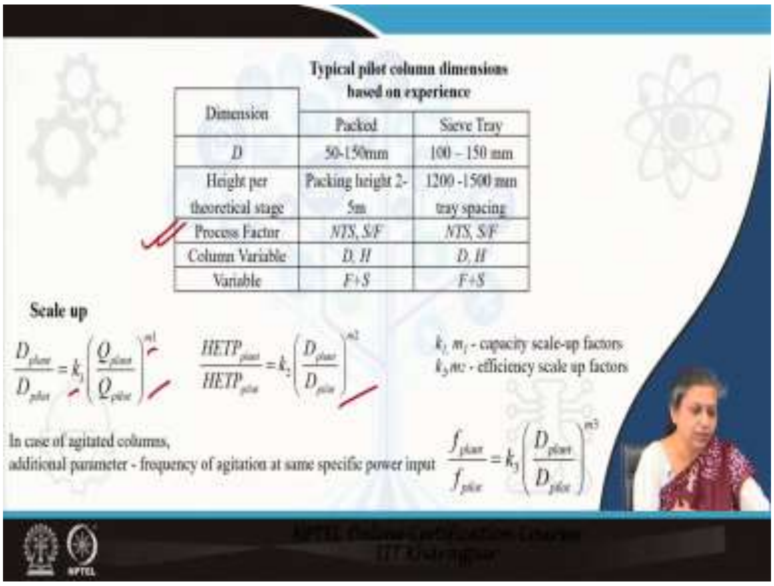

Typical pilot column dimensions based on experience

Dimension	Packed	Sieve Tray
D	50-150mm	100 – 150 mm
Height per theoretical stage	Packing height 2-5m	1200 -1500 mm tray spacing
Process Factor	NTS, S/F	NTS, S/F
Column Variable	D, H	D, H
Variable	$F+S$	$F+S$

Scale up

$$\frac{D_{plant}}{D_{pilot}} = k_1\left(\frac{Q_{plant}}{Q_{pilot}}\right)^{m1} \qquad \frac{HETP_{plant}}{HETP_{pilot}} = k_2\left(\frac{D_{plant}}{D_{pilot}}\right)^{m2}$$

k_1, m_1 - capacity scale-up factors
k_2 m_2 - efficiency scale up factors

In case of agitated columns,
additional parameter - frequency of agitation at same specific power input

$$\frac{f_{plant}}{f_{pilot}} = k_3\left(\frac{D_{plant}}{D_{pilot}}\right)^{m3}$$

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Now, once the pilot plant tests have been conducted, you have obtained the data, next you need to have some particular scale-up laws by basis of which you are supposed to scale up the pilot plant data to the actual plant conditions, we assume that the mass transfer characteristics are same in the pilot plant as well as in the actual plant. Based on them what are the things that we should be scaling up? Quite naturally the throughput will be higher in the actual plant as compared to the pilot plant.

So, therefore, accordingly the diameter and the HETP of the plant has to be decided, on other words diameter and the column length. So, these two are the parameters which have to be decided based on the pilot plant data and therefore, normally these are the two equations which are used where k 1 and m 1 are decided based on the particular contactor that you are selecting, k 1 and m 1 naturally refer to the capacity scale up factors and k 2 and m 2 refer to the efficiency scale up factors.

And one more thing just you should be remembering that when we are going for agitated columns then there is an additional parameter which should be considered that is the frequency of agitation at the same power input. This is the almost similar if you remember when we were discussing scalar for mixer settlers there also we had scaled up assuming the same power input.

So, in this case also the we assume this and accordingly the frequency of agitation is decided here, this has got an additional advantage that it gives us one more parameter that we can vary in order to optimize the operation. In this particular table if you see just for the packed and sieve because we are going to deal with these two, I have put down the typical dimensions of a pilot plant, these dimensions have been decided based on experience which have been gathered in different industries which use extractors for different purposes.

Now, here I have put down the typical dimensions and also the different process factors and the column variables as well as the variables. So, all these things have been put up. There is identical plant column, there is a pilot column dimension which are available for agitated contactors as well. Since we will not be dealing much with agitated contactors I have not included them in this particular table.

So, therefore, the for next thing that you have done. So, we have already discussed the pilot plant tower design and also the design of the actual column using the scale up relationships. The two important things which are left now to be discussed are the calculation of the number of theoretical stages.

And we can find out the column height H which will be equal to NTS into HETP for a packed column or it is going to be H equals to NTS into tray spacing for a sieve tray column. So, accordingly we can find out the H.

And the other thing which is left is the tower diameter from flooding considerations. Now regarding tower diameter let me mention that you are already aware how the tower diameter is decided for sieve tray and packed columns in the case of vapor liquid and gas liquid contacting.

These are based on the flooding considerations or these are designed based on the maximum mass flux of the vapor under flooding conditions or the volumetric flux of the vapor under flooding conditions. We will find that in this case it is slightly different, we will find what is different and why the difference has to be taken into account.

So, in this class primarily we are going to discuss the calculation of the number of theoretical stages and the estimation of tower diameter from flooding considerations these two we are going to decide. Now regarding the number of theoretical stages I would also like to remind you that when we had discussed gas liquid, vapor liquid contacting what did we do - we had constructed the equilibrium curve.

We had constructed the operating curve and then we had drawn the two curves if you remember in this particular way we had this line. So, therefore, for the distillation this was the curve if this was the Q curve and accordingly we had drawn the steps between the operating curve lines as well as the equilibrium curve and we had found out the number of stages.

In case of absorption also if you remember so therefore, this was the equilibrium curve, this was the line in the case of absorption, this will be the line in case of stripping and accordingly we had marked of steps and found out the number of stages. In this case also we can do this under very special circumstances.

Generally, the process is not so straightforward and you must have guessed the reason for it. The primary reason is the miscibility of the feed solvent and the extraction solvent between each other as a result of which for most of the cases we have to refer to ternary plots or rectangular plots.

And so, naturally just because of that although the concept is the same, the concept is based on the basic overall mass balance and the component balance, the same equations we are going to use, but just because of this particular miscibility and just because we have to go for ternary plots the situation becomes slightly difficult, I should say it is not difficult, slightly different in this particular case.

I will explain the logic behind the particular technique that how we have developed it and then I will be giving you a series of steps following which you can find out the number of theoretical stages.

And remember in your assessment what you will be given, maybe the entire process you will be not be requiring, maybe we would be telling you to formulate the operating line equation or to find the mixture point delta etc. So, you will be given such type of piecemeal questions from the estimation of the number of theoretical stages.

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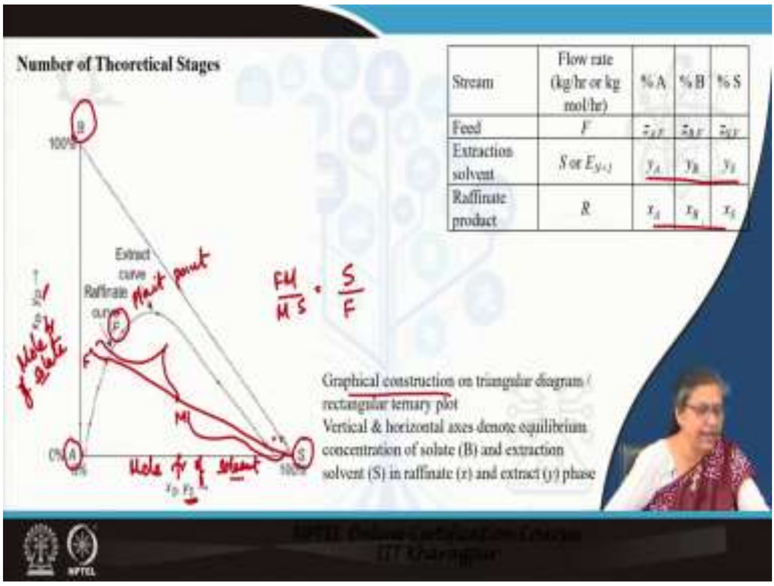

Number of Theoretical Stages

Stream	Flow rate (kg/hr or kg mol/hr)	% A	% B	% S
Feed	F	$z_{A,F}$	$z_{B,F}$	$z_{S,F}$
Extraction solvent	S or E_{N+1}	y_A	y_B	y_S
Raffinate product	R	x_A	x_B	x_S

$$\frac{FM}{MS} = \frac{S}{F}$$

Graphical construction on triangular diagram / rectangular ternary plot
Vertical & horizontal axes denote equilibrium concentration of solute (B) and extraction solvent (S) in raffinate (x) and extract (y) phase

So, now with this particular background let us go to find out the number of theoretical stages. So, therefore, mostly as I have mentioned that we have either a rectangular plot for most of the cases, we take a rectangular plot.

What happens in this particular rectangular ternary plot, whatever we have done it we can do the same thing on a triangular plot as well. In a rectangular plot what we have on the y axis we plot the composition or the mole fraction of the solute in the raffinate or the extract.

So, in this particular case we plot the mole fraction of solute, for my case B is the solute, S is the solvent, A is the feed solvent and on the x axis we plot the mole fraction of the solvent. Now, x S referred to the mole fractions in the raffinate phase, y S referred to the mole fraction in the extract phase.

So, therefore, when I plot x B versus x S, it means I am plotting the mole fraction of the solute to the mole fraction of the solvent in the raffinate phase and this is the curve that you generate from here. And similarly when I plot y B as a function of y S it means that I am plotting the mole fraction of the solute in the extract phase versus the mole fraction of the solvent in the extract phase to give me the extract curve.

So, therefore, the mole fractions of the solute and the solvent in the raffinate phase are given in the raffinate curve, the mole fraction of the solute and the solvent in the extract phase produce the extract curve both the curves meet at the plate point which marks the limit of immiscibility between the two liquids.

So, therefore, this is the zone of immiscibility you already know beyond the plot plate point whatever is there this entire region in this region A, B and S they are miscible in all proportions, as a result of which if you have to perform solvent extraction then in that case you have to perform this or rather you have to limit your operations within this particular immiscible dome.

Now, in this particular immiscible dome you can very well locate say the point F, now if suppose we do not have any amount of solvent in the feed then maybe the feed composition will be located somewhere here, but suppose there is some amount of solvent in the feed then naturally the point will not be located on the straight line connecting the feed solvent and the solute or the straight line connecting A and B it will be inside the triangle.

Say maybe it will be somewhere here under that condition same thing you need to locate - the solvent point. If you are dealing with completely pure solvent rather solvent completely free of solute and as well as A which generally does not happen because always we will be having some amount of recycled and recovered solvent, but it can happen when the feed solvent and the extraction solvent are completely immiscible.

So, that whatever small amount of A is there in S we can neglect it, whatever small amount of B is there in S we can neglect it, if we can neglect it then we can assume that the solvent is 100 percent pure or in other words the solvent starts or rather the solvent point is this particular point.

For most of the cases the solvent point will be somewhere here or if suppose it does not contain A, but it contains B then in that case it can be somewhere here. So, therefore, we can locate the solvent point as well.

Now, if you remember that I had already mentioned last time when we were when we were discussing regarding the ternary plots while discussing the equilibrium considerations if you join say this particular point to S then the mixture point is going to lie on the straight line joining F and S, more number of solvent the mixture point keeps on shifting here, lesser amount of solvent the mixture point is towards here.

So, therefore, the mixture point is somewhere it lies somewhere on the F S line and it depends upon the relative proportion of the feed solution and the solvent solution by the inverse lever rule on other words FM divided by MS this gives you the amount of solvent and divided by the amount of feed both of them taken in kg moles per hour or kg per hour does not matter.

So, therefore, the amount of solvent that decides the location of the point M. Now this finding the number of theoretical stages it will be based on the graphical construction which we are going to discuss and I will be discussing it in a rectangular ternary plot as I have told you, but the same thing is applicable for a triangular diagram as well.

This I have already told you what you need to remember is that all your compositions in the extraction solvent are denoted by y S and in the raffinate solvent they are denoted in the raffinate they are denoted by x S this is what you need to remember in this particular case.

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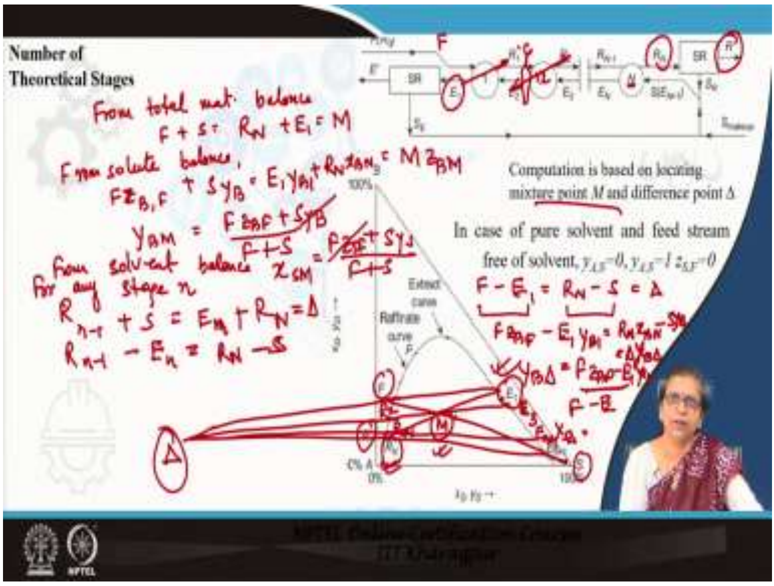

Number of Theoretical Stages

From total mat. balance

$F + S = R_N + E_1 = M$

From solute balance,

$F z_{B,F} + S y_B = E_1 y_{A1} + R_N x_{AN_S} = M z_{BM}$

$y_{AM} = \frac{F z_{AF} + S y_B}{F + S}$

From solvent balance $x_{SM} = \frac{F z_{SF} + S y_S}{F + S}$

For any stage n

$R_{n-1} + S = E_n + R_N = \Delta$

$R_{n-1} - E_n = R_N - S$

Computation is based on locating mixture point M and difference point Δ

In case of pure solvent and feed stream free of solvent, $y_{A,S}=0, y_{A,S}=1\ z_{S,F}=0$

$F - E_1 = R_N - S = \Delta$

$F z_{AF} - E_1 y_{A1} = R_N z_{AN} - S y_{AS} = \Delta y_{A\Delta}$

$y_{B\Delta} = \frac{F z_{AF} - E_1 y_{A1}}{F - E_1}$

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So, now see just like we are done for absorption and distillation in this particular case also what we do we perform this particular stage calculation based on the overall material balance and the component material balance. So, from the I have drawn the diagram here this is F and it goes on this the from the last stage N th stage R N comes in and from here S enters which is nothing, but the extract entering from the N plus 1 th stage and what comes out is E 1.

So, therefore, suppose I do the total material balance what will I get from the total material balance, I will get F plus S if you can see F entering, S entering will be equal to R N going out from here plus E 1 going out from here this is equal to M is not it. So, therefore, what does it mean it means that suppose my F is here and suppose my E N plus 1 is here if I join then M is going to be a point.

And on that and that point will also mark the intersection between the final raffinate composition say the final raffinate composition is somewhere here and suppose the E 1 is here then if I join the two then in that case M is going to mark the point of intersection between F S and R N E 1.

And so therefore, what are the coordinates of M, to find out the coordinates of M we can do the solute balance. From solute balance we are going to get the y coordinate from solvent balance we are going to get the x coordinate is not it. So, for from this should be from not for.

So, from solute balance or the balance of component B we can locate M in what particular way, we can write it down as FZ B, F where B is our solute plus S y B will be equal to E 1 y B 1 plus R N x B N is not it.

So, and this is equal to M into Z B M actually since we are doing the solute this should be actually y BM. So, from there we can find out y B M or z B M in whatever way you tell it this is nothing, but F Z B F plus S y B divided by F plus S, if you are taking completely pure solvent then in that case this disappears off.

In the same way from the solvent balance if we take from the solvent balance in the same way we can find out my x S M. This is also going to be the same thing it will be F Z SF plus S y S divided by F plus S is not it and for most of the cases suppose we do not have any solvent in the feed then this disappears off and this gives us the x coordinate of the point. So, therefore, we know that M also lies on the E 1 R N line as well.

Now, we can just rearrange the saw the total material balance and the solvent material balance equation, just how I rearrange it say F minus E 1 if I write it down this gives me equal to R N minus S and this side I denote it as a difference point delta. What is this delta you tell me, it is the difference of the net flow from any particular stage this is the net flow out from the first stage, this is the net flow out from the last stage.

So, from there similarly I can perform a solute balance also, if I perform a solute balance just the same balance I can write it down in the same way which is going to give me F Z B F minus E 1 y B 1 will be equal to R N x B N minus S y B which will be equal to delta x B delta. In fact, it should be actually y B delta not x because we are we shall be getting the y coordinate from here.

So, from there what do we get, from here if you find we are going to get y B delta it will be equal to say F Z B F minus E 1 y S 1 divided by F minus E and in the same way you can find out the x S delta as well. Now, we find that instead of doing for the entire plant we can write down such equation for any particular individual stages, say instead of writing it for the last stage N we can write it for any particular intermediate stage say for example, this is the stage N say for any particular stage we can write it down.

When we write it down for any particular stage we find that we get something like for any stage n what do we find R n minus 1 is entering plus S equals to E N plus R N when we are writing from stage N to the last stage and this is equals to delta. So, from here also what do we get, we get R n minus S equals to R n minus 1 minus E n will be equal to R N minus S which is nothing, but equal to this.

So, therefore, we find that whether we take up any particular stage for any stage we find that the net flow out of this stage should cross through one particular point which is the delta point and this delta point therefore, it should comprise of all the or it should be a point from where all the lines for which mark the net outflow from any particular stage should be located is not it.

So, therefore, from here what do we get, we get that if suppose we have a point say F E 1 suppose I join f E 1 and I extended this side. I join R N minus S or if it is E N minus 1 and I extend it here and I find that it is it joins at a point delta. So, therefore, it means that from delta if I take a point which is connecting R 2 say the raffinate which is going out of R 2 and if I extend it then it should this point should be E 3 in the same way if I extend it and I come to some particular point R N.

And I extend it then this should give me the point E N if I extend it if it is R N then this should give me E N plus 1 where N is any particular number of stages. So, therefore, there are two things on which the entire construction is based, it is based on locating the mixture point once you have located the mixture point you know F, you know S the R is usually specified, but in this case you have to remember generally this R prime after the solvent recovery is specified.

So, therefore, since it is depleted of solvent this R prime is going to lie on the y axis. So, therefore, how to find out R N in this particular case, when you join the R prime with the S or the E N plus 1 depending upon the concentration of the solute then the point where this R prime meets this raffinate curve gives you R N.

So, once you have noted down R N then you know, F you know, S you know, F you know, you know R prime from where you know R N by joining R prime and S. So, once you know this. So, if here the point M is known to you then simply drawing a line from R N to M to the extract curve gives you the E 1 point. Moment you know the E 1 point what you do E 1 F you extend it backward and you extend S R N point backward you get the point delta.

Once this point delta is done then what you do from E 1 you can draw a tie line, when you draw a tie line from E 1 what do you get, you get R 1 from because you know that this E. You know R 1 because R 1 and E 1 these two are in equilibrium for every particular stage, we know that the exiting streams are in equilibrium.

So, therefore, from E 1 you get R 1, then from the delta point if you are extending it you are going to get E 2, from E 2 you draw the tie line you will get R 2, from the delta point you extend to R 2 and then extend it you get E 3.

You can keep on continuing this particular feature and the number of times you are you have you are doing this gives you the number of stages you keep on continue till you reach the point R N here. This is the basic thing which you are supposed to do for the particular composition or rather for constructing the number of stages here.

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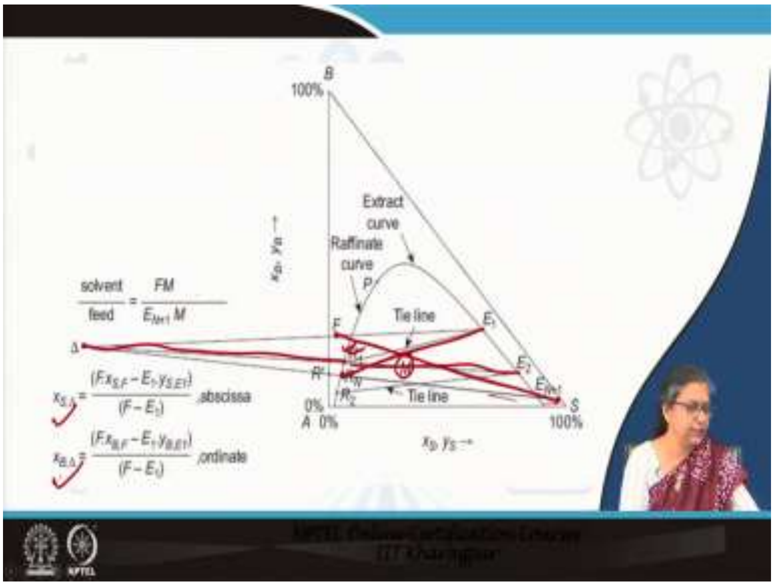

$$\frac{\text{solvent}}{\text{feed}} = \frac{FM}{E_{N+1}M}$$

$$x_{S,\Delta} = \frac{(F x_{S,F} - E_1 y_{S,E1})}{(F - E_1)} \text{, abscissa}$$

$$x_{B,\Delta} = \frac{(F x_{B,F} - E_1 y_{B,E1})}{(F - E_1)} \text{, ordinate}$$

The whole thing is already done to you very clearly what we do from R prime we have joined, I repeat once more you have located R N here once you have located R N the F point is here the E N plus 1 point because we have not taken pure solvent in this case. So, therefore, you join this particular line and then if you know M then you join M to here you get E 1 you join F E 1 you get and you join these two you get the delta point.

Then from even you have dropped up tie line you get R 1 from delta to R 1 extend it you get E 2, from E 2 again you draw a tie line you get R 2 in this particular case we find R 2 is less than R N. So, therefore, we stop the construction here. So, this is the thing that you need to do. In fact, you can also geometrically you can find out x S delta x B delta just the way I have told you.

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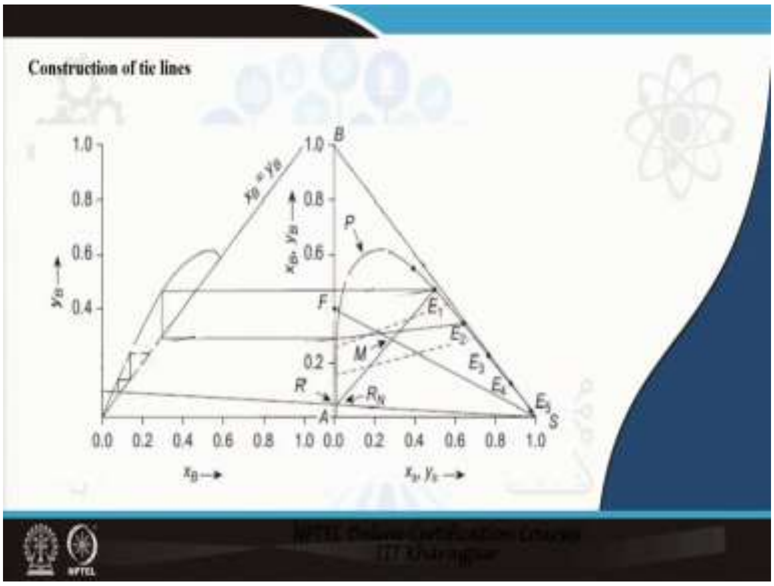

Construction of tie lines

So, therefore, once you have done this, so you know how to find out the number of stages. The only thing that is remaining now for you to think is how to construct the tie lines, this is the next thing which we will be doing in the next class.

Thank you so much.