Mass Transfer Operation II
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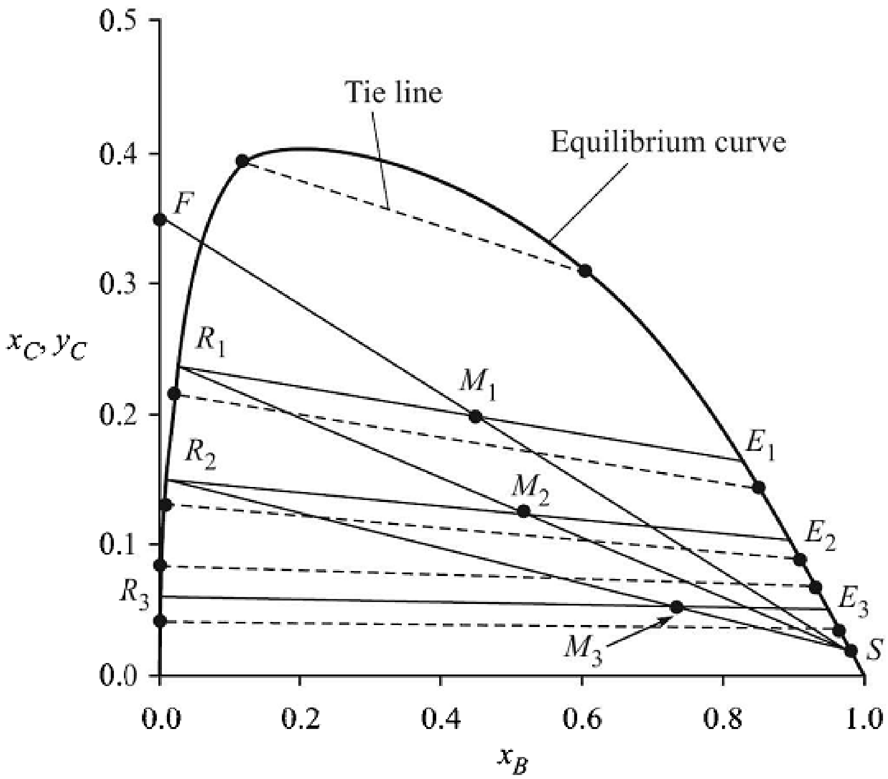
Lecture - 13
Liquid-Liquid Extraction
Design Calculation of Multistage Operation

Problem 3: 1000kg of an aqueous solution containing 35 mass % trimethylamine(TMA) and 65 mass % water, is to be extracted using benzene as solvent. A three stage cross current extractor scheme is suggested. The amounts of solvent (98% benzene, 2% TMA) to be used in successive stages are 815kg, 950kg and 2625kg. Determine the fraction of the solute removed if the stages are ideal. The composition of the raffinate and the extract (two phases) as well as the tie line data are given below (water: A, benzene: B, and TMA: C).

Water rich phase	x_B	0.004	0.006	0.01	0.02	0.03	0.036	0.07	
	x_C	0.05	0.10	0.15	0.20	0.35	0.3	0.35	
Benzene rich phase	y_B	0.95	0.90	0.84	0.78	0.71	0.63	0.50	0.26
	y_C	0.05	0.10	0.15	0.20	0.25	0.30	0.35	0.40

Tie line data	x_c	0.04	0.083	0.13	0.215	0.393
	y_C	0.035	0.068	0.09	0.145	0.31

Solution:



The problem is solved using right triangular co-ordinates.

F = 1000kg, $x_F = 0.35$

Stage I: $S_1 = 815$kg, $y_S = 0.02$

$M_1 = 1000 + 815 = 1815$kg

$X_{M1} = (Fx_F + S_1y_S)/(F + S_1) = (1000\times0.35 + 815\times0.02)/(1815) = 0.202$

The point F (x_B=0, x_F=0.35) and S (y_B=0.98, y_C=0.02) are located on the diagram. Point M_1 is located on the line FS (x_{M1}=0.202). The tie line is drawn. Extremities of the tie line give raffinate and extract phase composition of stage I in equilibrium. From graph:

$x_1 = 0.24$, $y_1 = 0.166$

$M_1x_{M1} = R_1x_1 + E_1y_1$

$1815\times0.202 = R_1\times0.24 + (1815 - R_1)\times0.166$

$R_1 = 882.97$kg

$E_1 = 932.03$kg

Stage 2: Raffinate from stage I(R_1) is fed to stage 2

$S_2 = 950$kg, $y_S = 0.02$

$M_2 = R_1 + S_1 = (882.97+950)$kg $= 1832.97$kg

$M_2x_{M2} = R_1x_1 + S_2y_S$

$1832.97\ x_{M2} = 882.97\times0.24 + 950\times0.02$

$X_{M2} = 0.126$

The composition of the solvent to the second stage being the same as that to the first, it is represented by the same point S. Line R_1S is joined and point M_2 ($x_{M2} = 0.126$) is located. The tie line R_2E_2 is drawn through the point M_2.

From graph $x_2 = 0.15$, $y_2 = 0.105$

$M_2x_{M2} = R_2x_2 + E_2y_2$

$1832.97\times0.126 = R_2\times0.15 + (1832.97 – R_2)\times0.105$

$R_2 = 855.39$kg and $E_2 = 977.58$kg

Stage 3: Stage 3 receives $R_2 = 855.39$kg as feed

$S_3 = 2625$kg

$M_3 = (855.39 + 2625)$kg $= 3480.39$kg

$M_3x_{M3} = R_2x_2 + S_3y_S$

$3480.39\ x_{M3} = 855.39\times0.15 + 2625\times0.02$

$X_{M3} = 0.052$

Locate M_3 ($x_{M3} = 0.052$) on R_2S and draw tie-line through $M_3$to meet the equilibrium curve at the point R_3 and E_3. $X_3=0.06$ and $y_3 = 0.051$

$M_3x_{M3} = R_3x_3 + E_3y_3$

$3480.39\times0.052 = R_3\times0.0596 + (3480.39 – R_3)\times0.0506$

$R_3 = 541.39$kg and $E_3 = 2939$kg

Total amount of trimethylamine (TMA) leaving the stages $= E_1y_1 + E_2y_2 + E_3y_3$

$= 932.03\times0.166 + 977.58\times0.105 + 2939\times0.051$

$= 407.3$kg

Total amount of TMA entering with solvent $= (S_1 + S_2 + S_3)\ y_S$

$= (815 + 950 + 2625) \times 0.02$

$= 87.8$kg

Net amount of TMA removed $= (407.3 – 87.8)$ kg

$=319.5$kg

Total amount of solute in the feed = 1000 × 0.35 = 350kg

Fraction of TMA removed = (319.5/350)×100 = 91.3%

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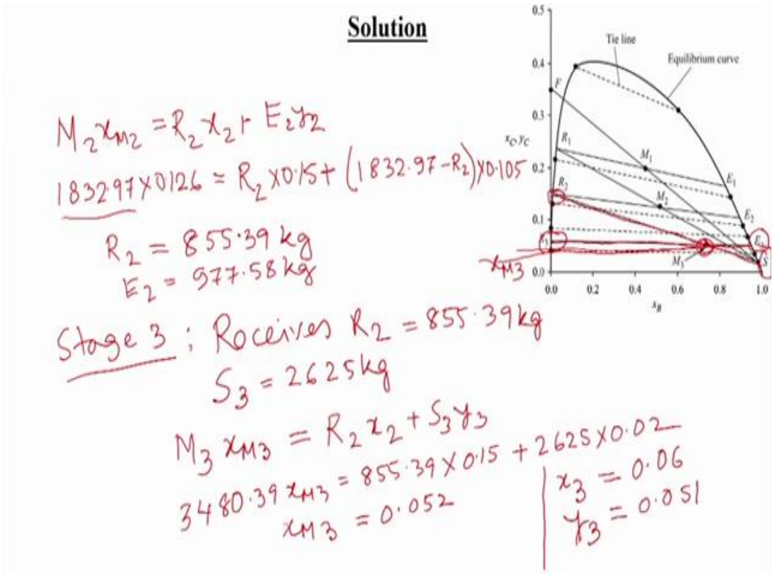

Like we can say, M 2 x m 2 is equal to R 2 x 2 plus E 2 y 2. So we have this M 2 we have this 1832.97 and x m 2 we have find, found out that 0.126 that is equal to we can say this one R 2, that is unknown to us, we will be getting this one into x 2 we have this one 0.15 plus E 2 will be like this, the total is 1832.97, so the we can say this one 1832.97 minus R 2 into y 2 is equal to 0.105 into say 0.105. So from here actually we will be getting R 2, that is equal to we will be getting this 855.39 kg, so it is R 2 is equal to 855.39 kg, okay.

Then we will be getting E 2 is equal to, say from this total, 1832.97 minus 855.39, that is coming out as 977.58 kg, okay. So this, this M 2, we can say this one, whatever the R 2 we have this one now is the feed to stage 3, so we can again now for stage 3 we will be joining this S and R 2 so we will be, for stage three we can say, for stage 3 we can say this one, it receives, receives R 2 okay, and so that is we have this one 855.39 kg and S 3, that is given in the problem that is equal to 2625 kg, okay. That is equal to say whatever we got it, this one 855.39 kg and S 3 is equal to this one.

So now, we will be getting this one, from this we can say, we can say this one whenever we will see this component balance for this system R 3, say R 2 and S and to say we will be getting this M 3, we will be getting this one, just by doing the component balance M 3, that you can say this mixture, that will be summation of R 2 and S 3, so that is M 3, x m 3 need to get is equal to we can say R 2 and into x 2 plus, we can say this one S 3 and y 3. Okay.

So M 3 that is nothing but the we can say this one, summation of these two R 2 and S 3 that is coming out as 3480.39 into x m 3 is equal to R 2 is equal to 855.39 into x 2 equal to 0.15 plus S 3 is equal to 2625 and the solute concentration in the extracting solvent is already given as 2 percent, so it is given as 0.02. So from here we will actually be getting x m 3 is equal to say point, it is 0.052. So whenever we have this, we have got this x m 3 like this one, 0.052 so now we have this point M, M 3, so we have this R 2 and S and we have x m 3 so if we extend this one, from this we can say x and y c, x axis.

So when this touches this R 2 axis from there we will be getting the point M 3. So here also this point, in this mixture is M 3, it will be divided into 2 different parts like this one will be R 3 and another will be E 3. So from here actually, we can say this one, if we read this one, R 3 and E 3, so we will be getting, say, we can say, the x 3 and y 3 so that we will be getting from this graph, that we are getting this x 3 that is obtained as very small value, like this, 0.06 this value and y 3 that is again lower that is you can say 0.051. So now, we have this, x 3 and y 3 we have, now we will be doing this component balance, from there we will be getting R 2, sorry, R 3 we will be getting from there.

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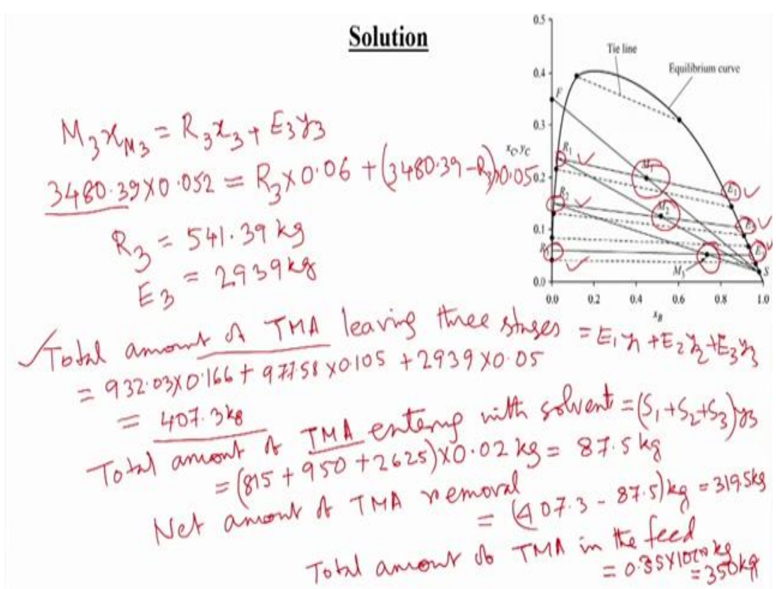

So we will be doing this say, component balance like this, say M 3 into say, we can say this x M 3 is equal to we have this R 3 into X 3 plus E 3 into y 3, okay. So now M 3 we have this one, already we have this one, so 3480.39 into x M 3 actually we have got this one as a 0.052 that is equal to R 3 we need to find out, R 3 into x 3 that we have got, which is very very small value 0.06 and say we can say, and this E 3 means nothing but this total minus R 3 that is 3480.39 minus R 3 into 0.05.

So from here actually we will be getting R 3 is equal to we will be getting, by this, manipulating this equation, we will be getting this 541.39 kg and say E 3 we will be getting as, say the rest of this, means 3480.39 minus 541.39 that is coming out as 2939 kg, okay. So now, we have this total x 1, x 2, so we have this one x 1, y 1, x 2, y 2, and x 3, y 3 and R 1, R 2, R 3 and E 1, E 2 and E 3.

So we have all these parameters like this and also we have M 1, M 2 and M 3 means x M 1, x M 2, and x M 3. So we have now everything with us now and also we know that the total amount of extracting solvent, how much amount of solvent is added and how much amount of the component is a tri methylamine, which is also enter inside this extracting system.

So we know that also, okay, so now we will be finding that how much amount of we can say this one tri methylamine that is leaving the stages means from the different stages like this, so we can say this one, total amount of tri methylamine that is leaving the 3 stages. Now we can say, will be like this, will be equal to, will say this one, E 1 say y 1 plus E 2 y 2 plus E 3 y 3, so that will be equal to say this one, E 1 is equal to say 932.03 into 0.166 plus E 2 is equal to 977.58 into 0.105 plus E 3, whatever we got on this one 2939 and y 3 is equal to 0.05 okay.

So that, this total amount is coming out as 407.3 kg, okay. So we can say this one, this is one point. Now, say how much amount of tri methylamine is entering inside the 3 extractors with the extracting solvent. So that is also we need to consider, but whenever we will be calculating the extraction efficiency, we need to deduct that amount from the total amount of this one, tri methylamine, that is leaving, from there we need to deduct that amount.

So total amount of tri methylamine, say we can see this one entering, so with solvent. So there are 3 different solvents, so we can say this one will be equal to say, we can say, S 1 plus S 2 plus S 3 into we can say this on for y s because for all the cases the solvent, solute concentration in the solvent is same. So it will be like this, so we have this that these are given 815, in the problem these are given, S 1 is 815, S 2 is 950, S 3 that is given as 2625 kg. And the concentration of the solute in the this one, extracting solvent is 2 percent, so into 0.02 kg so now we can say this one, this amount, this one, this small amount, that is 87.5 kg. This one what is called tri methylamine has entered.

But we say, we have seen that total 407.3 kg tri methylamine is extracted. So we can say effectively we can say on this one, so now we can say this one net amount of tri methylamine, that is we can say this one, removal, so we will be equal to 407.3 minus 87.5 kg. So that is coming out as 319.5 kg, okay. So we can say this one, now how much amount of the tri methylamine was there in the total feed. So we can say this one, we had 1000 kg feed, so we can say, we had total amount of, we can say, total amount of tri methylamine, so we can say this one in the feed, so that is equal to we can say this one, 35 percent which is 0.35 into 1000 kg, so that is 350 kg.

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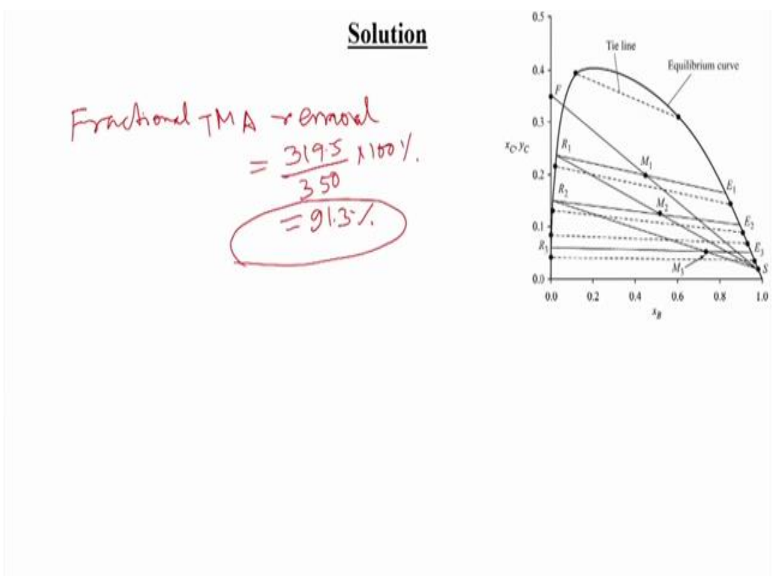

So out of this 350 kg, now we have, we can say 319.5 kg, so these are separated. So we can say this one, fractional, say tri methylamine removal so will be like this, 319.5 divided by 350 into 100 percentage. So that is coming out as 91.3 percent. So we can say, this is the fractional tri methylamine removal. Okay. So 91.3 percent tri methylamine is removed, okay. So that was the question actually for this, this is the question for this problem, okay.

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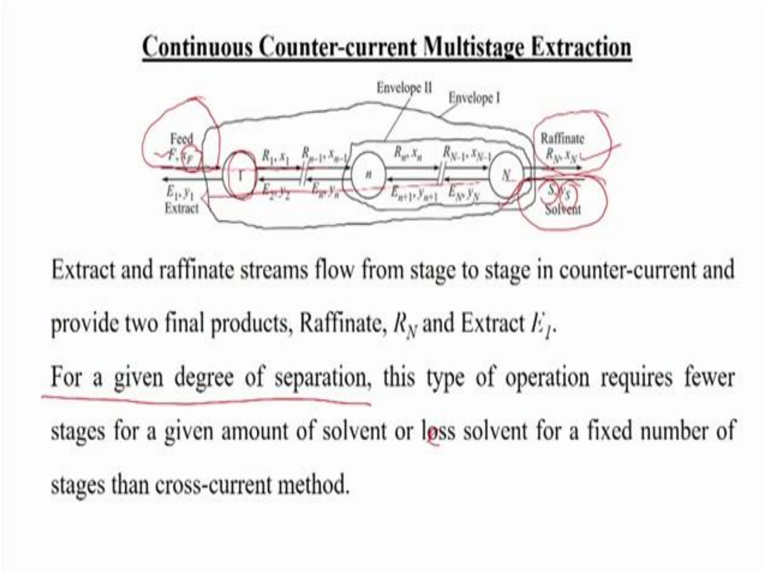

Now we will be discussing about the continuous counter current multistage extraction process. So in this case actually, the feed is entering from one side of this we can say system and we say suppose feed flow rate is F and the solute concentration as x f and the raffinate is exiting from the other side of this, we can say this one extraction unit. So that is we can say this solid concentration as x N, and flow rate of the raffinate is R N and in that side, we can say this one, from where the raffinate, last raffinate is actually exiting, there the solvent actually is added.

So that with the solvent flow rate is S and solute concentration as y S, that y S may be 0 in some cases, and that is why it is counter currently these are flowing. So we can say this one, the solvent, extracting solvent will flow in this direction, and the ultimate, the final extract will be leaving from the first extraction stage, where the feed was added and the feed will be moving in this direction.

Like this, whatever we have this one, tried in this counter current cross current extraction, here we say both the extract and raffinates are moving in the cross current direction. And say, this, sorry, counter current direction, but the thing is that the solvent is not added in all the stages individually. So solvent is added in the last stage of the extraction unit. So we can say this one Nth stage solvent is added and the solvent actually is carried forward.

So we can say this one, extract and raffinate stream, flow from the stage to stage in the counter current mode, and provide two final products, like we can say this raffinate, that is exiting from the final stage of the extraction unit and extract that is coming from the first stage of the extraction unit. So for a given degree of separation, we can say this one, this type of operation, requires this less number of stages for the given amount of solvent or we can say this one this less solvent, we can say this one, less solvent for a fixed number of stages than cross current method like this, whatever we have discussed previously, is compared to the cross current extraction process. The counter- current extraction process is, we can say economic.

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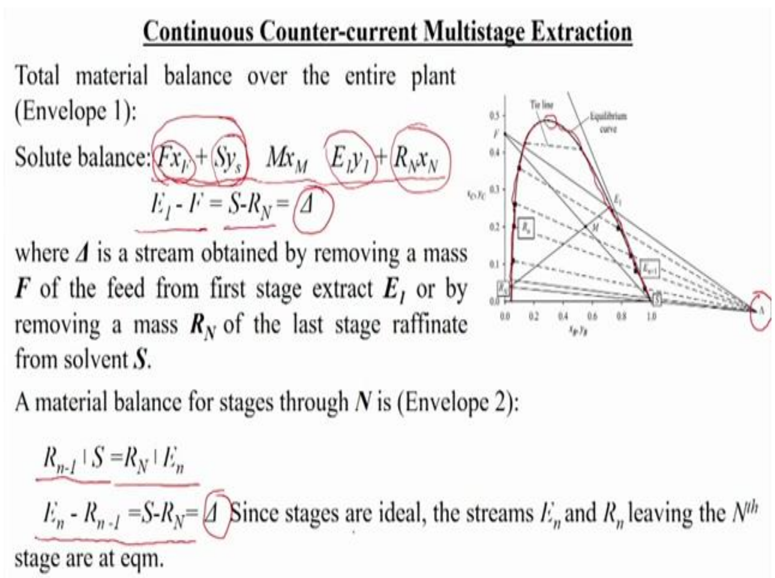

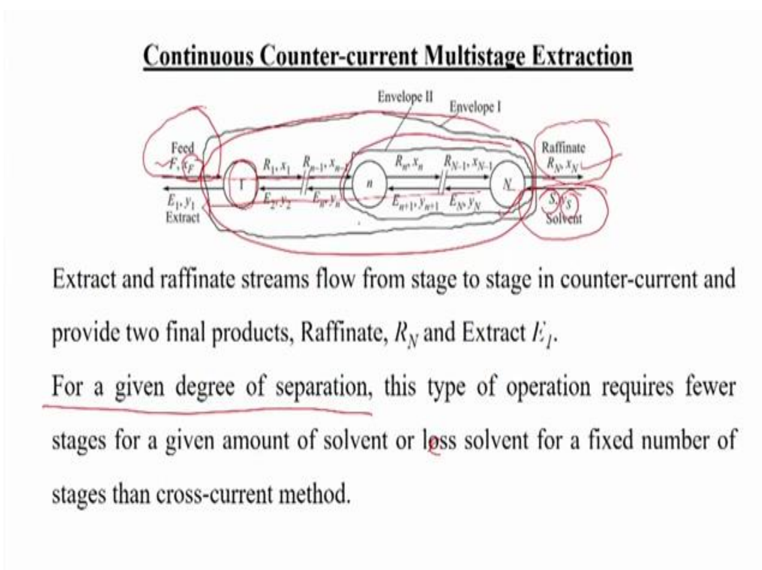

So, like this one, here we will be the discussing the step by step procedure also, for the design of the multistage counter current extraction process. Before that we need to understand the we can say process. So in that case also, we have this, we can say, this binary zone like this, this is the raffinate R N and suppose the tie plate point is somewhere here and this is the extract R N, okay, and it is a rectangular triangular diagram.

Now we can say this, we will be doing this for the entire plan, for the entire system, say we can say this one, the feed is entering at the first stage and raffinate is exiting from the last stage, where is in the last stage, say solvent is added and from the first stage extract is taken out. So we will be, what we will be doing this one, if we do the material balance, then we can say this one, F x f plus S y S, is equal to we can say this one, M x M that is nothing but this E 1 y 1 plus R N x N.

So that is we can say this one, whatever in this first stage, this we can say this one, this we can say F x F, S y S in the last stage and then we can say this one, extract is coming out from the first stage with E 1 y 1 and raffinate is exiting from the last stage that is with the solute amount will be R N into x N.

So whenever we will be doing this suppose if we say this one, E 1 minus F means in any of the stages if we say what is the resultant of the, we can say this one flow rate, then that is the nothing but we can say E 1 minus F, that is then in case of this we can say S minus R N so we will be getting always the common factor like this so that we, now, design, or we can this one as the delta.

So this is nothing but the delta is a stream that is obtained by the removing a mass F of the feed from the first stage and this extract E 1 or by removing we can say this one mass R N of the last stage raffinate from the solvent S. Or we can say this one in any stage, this one, whatever is entering and what is exiting, what is the resultant of this one, so we can say this one, if we do the now do the material balance in the envelope 2 like this in the envelope like this, so there actually we will be getting, like this, R n minus 1 plus S is equal to R N plus E n. So from here we will be getting, the extract minus raffinate, that will be giving like this S minus R N. So this is also delta.

So if we do this one, repeatedly, for all the stages also, we will be getting one difference, we can say this one, extract minus raffinate so that is, this the delta. So that delta actually we will be getting here, how to get this one, we will be discussing later in this. So since the stages are ideal, so the streams E n and R n leaving the nth stage are in equilibrium. So we are assuming that all the stages we can say this one, attain equilibrium before leaving the this one, the extraction unit. Okay.

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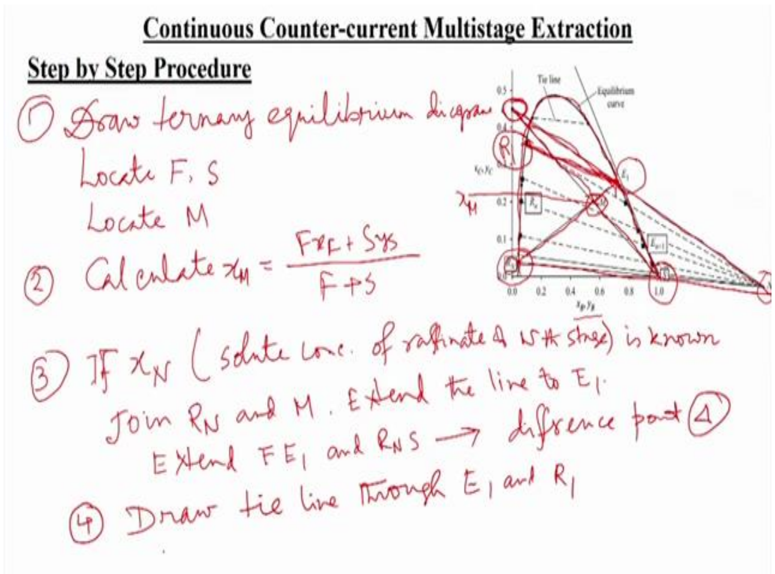

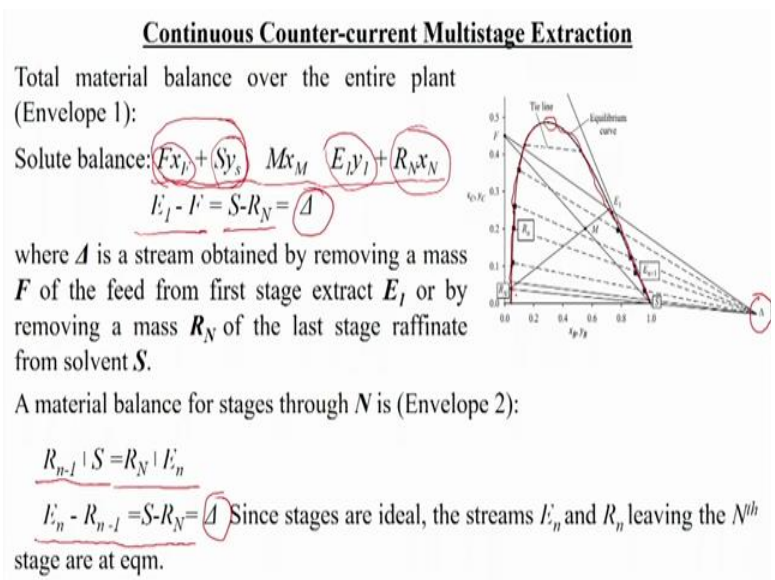

So we can say, we will start with step by step calculation, procedure for this, we can say multistage counter current extraction unit. This is very simple and easy to understand also. So we will be doing this one. Now we need to draw this one, what I actually told earlier. We need to say if we know the, we can say this extracting solvent, carrier solvent and solute miscibility then we have this ternary diagram.

So we will be now drawing this one from the we can see the equilibrium data point will be getting this we can say binary phase zone by drawing this we can see equilibrium line. So this one we can see this one suppose this is the plate point so left hand will be a raffinate term and right hand side will be the extract term. This is common to every extraction stage like this. So now we have to draw this ternary equilibrium diagram. So first step will be like this we need to draw this ternary equilibrium diagram.

So this diagram may be of many types what we discussed this one it may be right angular triangular diagram, it may be or equilateral triangular diagram or it may be solvent-free bases also, so whatever will be this 1 but for most of the this 1 calculations we prefer this right angular triangular diagram for convenience we will also be discussing this one while we solve the problem. This is a very convenient way of this one drawing this right angular triangular diagram. Okay.

Then we need to locate we can say this point feed point and as well as this solvent point, so this feed point that that is composition is already given and how much amount of solute is there? Suppose in this case for suppose around 45 percent solute is present in the feed so it is like this X c will be 0.45 whereas this we can say this one X b will be 0 and so this is the feed point.

And the solvent point we can say this one that is S will be almost we can say this one pure or small amount of solute may present based on that actually it will be placing near to this we can say this one in the Xv and Y v axis. Ok, if it is 100 percent pure then it will be touching this y-axis is equal to 0 and if Ys is not 0, then it will shift towards we can say this one is following this equilibrium diagram. So may be somewhere here like this okay.

Now we need to get this one this we need to add this F and S so we will be adding this one. Now, we will getting this point M, so M will be located and now we need to say locate M that is the mixture point. So this mixture means if we know M then from the component balance we will be getting suppose this X m so from there will be getting this M. So now, we need to calculate this, the second step will be like this we need to calculate X m from this component balance like this, so we will be doing this F x f plus S y s by F plus S.

So if we know all these because you see this X f and Y s these are already given in the problem means this one what is the feed we know this one and what is a solute concentration in the feed and the solvent flow rate and then we can say this and total amount of solute plus solvent we will be getting now this X m. Okay.

So then the third stage will be like this, if the solute concentration of the raffinate of the final stage is known like this we can say this one if X N, solute concentration of raffinate that is leaving the last stage actually raffinate of nth stage or we can say this one that is the final stage is known. Suppose this is given as this known parameter then we can say this one say R n and M are joined like this, so we can say that if it is given then we can say this one whatever the we can say this one in the raffinate whatever the solute concentration.

Suppose X n is given so it will be like this somewhere else, then we can say this one we have this RN is known, suppose then X is known then we can say this one we will be adding this RN and joining M, then we will be extending to the extract arm then will be getting this E 1, ok so we will be getting that so now we will be joining suppose RN and say M then extend this one, extend the line to E 1. That is now in the we can say in this extract arm. Okay.

Now, we have this one F is available we know then E 1, so we know that this the difference point actually that we found out that suppose E 1 minus F or S minus R N that is giving this Delta, so we can say this one whenever we are have this F and E 1 we have this 1 so we can say this one E 1 minus F or we will be getting now say S minus R n suppose we have this S and RN so from there we will be getting this same delta.

Like this now we have this E 1 and F suppose we will be joining 1 line we have RN and S, so from this 1 we can say S minus R N or E 1 minus F where it will be meeting so that is that is nothing but delta. Okay so from there actually we will be getting this we can say this from there we will be getting this difference point. So if we extend say if we extend F E 1 and R N S then we will be going to this difference point like difference point so this Delta, so we will be getting this data. So for all these stages also if we extend this one it will meet a particular point that is called say the difference point that is delta, okay.

Now the Fourth Stage will be like this say draw the tie line that is just like this one whatever we have done in the other we can say this one extraction processes, so we need to draw tie line this one through E 1 and R 1 like this, so we will be doing this one. So we have now this say E 1 we have this if we get this we can say this one a tie line, so we will be getting R 1 and so we will be drawing this one like this from here we will be drawing this (equilibrium line) tie line.

So here we will be getting this R 1, so this is the tie line then we will be getting this R1 here okay. So we can say this one whenever this R 1 we have this 1 it is nothing but whatever raffinate is obtained from the stage 1 that is nothing but the feed to the stage 2 ok and now we have this Delta so if we do this one if we repeat this one or we can say this one say this stage 5 will be like this.

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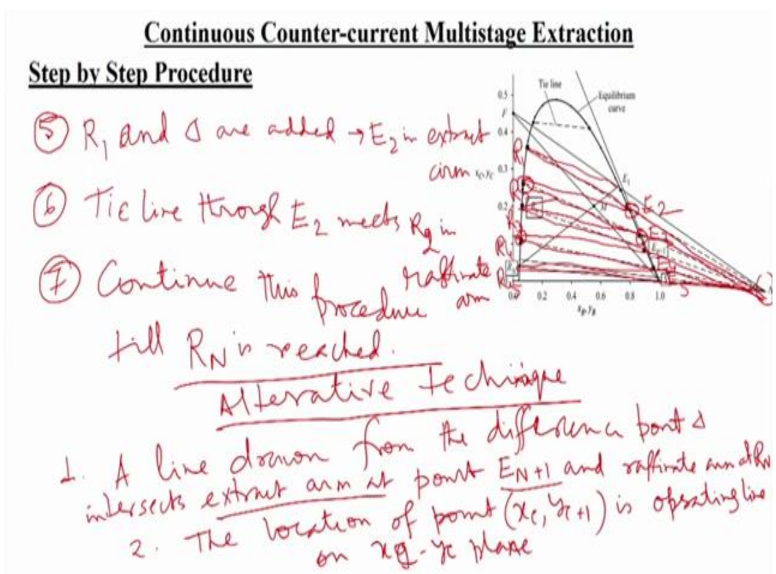

Now we have this Delta point we have suppose this is R 1 we have this one because this is the tie line from E 1 and then we can say this R 1 and delta. So these two are added. Now we will be getting this E 2 here, so then we will be obtaining this then these are added will be getting E 2 in the extract arm, okay. So that will be we can say this one. Nsow again from this we can say this we need to draw this suppose this stage 6 will be from E 2 again we will be drawing the tie line and it will be like R 2. So now what we will be doing this we can say tie line through this E 2 meets R 2 in raffinate arm. Okay.

So this is R 2 that is feed to the stage 3. So now we will be continuing this one suppose this again you will be adding this R 2 and Delta, so here it will be meeting E 3, then we have again this we can say tie line we will be getting this R 3 and that way we will be actually continuing and then we will be getting say one time we will be reaching like this so whenever we have this R 3 we will be joining Delta, then we will be getting now say E 4, then from there we have this we can say tie line, there we will be getting say R 4, so that way again will be drawing this tie line and then we will be getting E 5 and then suppose we have this again tie line, we will be getting suppose R 5, then we will be adding this 1.

So one time we will reach actually we will be now we can say this we will be now continuing this one, continue this procedure till RN is obtained or reached. So this is one route actually this is we can say this is very convenient route I think we are able to this one understand the procedure for the stage calculation in the counter current extraction process. So another alternative technique is there like this alternative technique.

So what we will be doing a line drawn from the difference point delta intersects extract arm at point actually EN plus 1 and at raffinate arm, its extract arm that is in N plus 1 and raffinate, raffinate arm at RN. Okay. Now we can say this one, then second stage will be the location point like this we can say this Xc, and Yc plus 1 that is nothing but the is operating line, that is on suppose this is Xc and Yc plane, we will be drawing this one Xc, Yc plane also we will be drawing this one.

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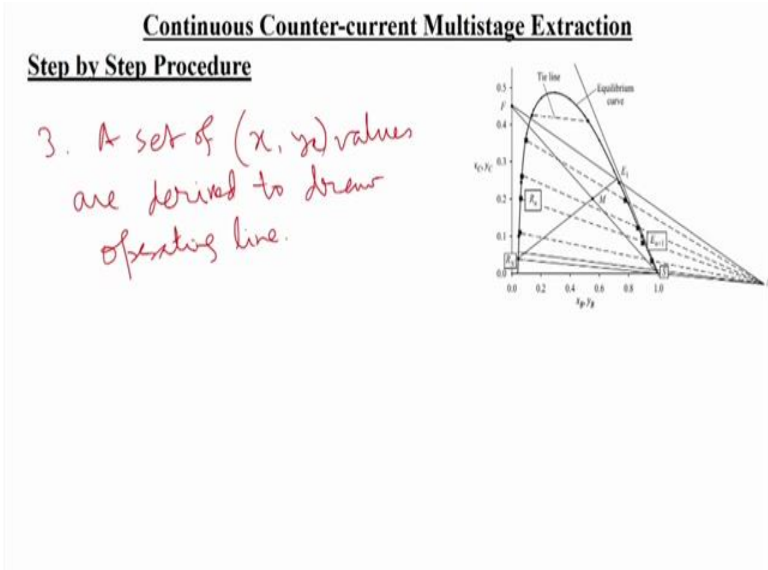

Then we can say then a set of Xc and Yc values are derived to draw operating line. So that also we can do this 1 so we can say this in using this alternative technique also we will be doing this we can say counter current or multistage extraction system.

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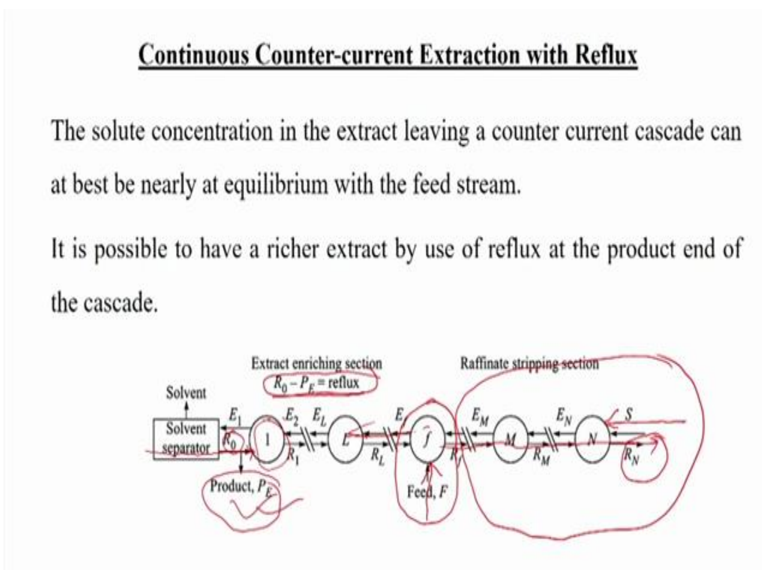


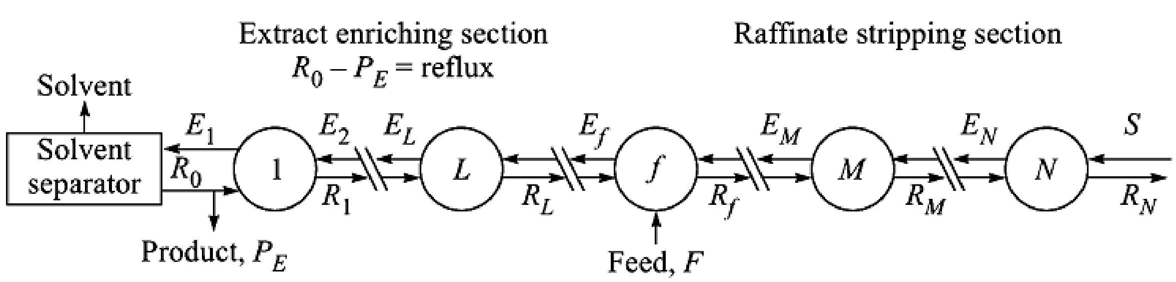
Now, we have this another system that is we can say this one continuous counter current extraction with some reflex this is we can say this one the solute concentration in the extract leaving a counter current cascade can based to nearly at equilibrium with the feed stream, so it is possible to have a richer extract by the use of the Reflux at the product end of the cascade.

This system is similar to that of the distillation unit, say this is the feed point location so feed is adding here in the fth stage and then say this 1 we can remember that make this equivalent like this say this raffinate from the f stage we will follow this route and then we will be getting the final raffinate from the last stage or the nth stage where we can say that the extracting solvent was added. Like this is a counter current operation but feed is not added at this stage 1.

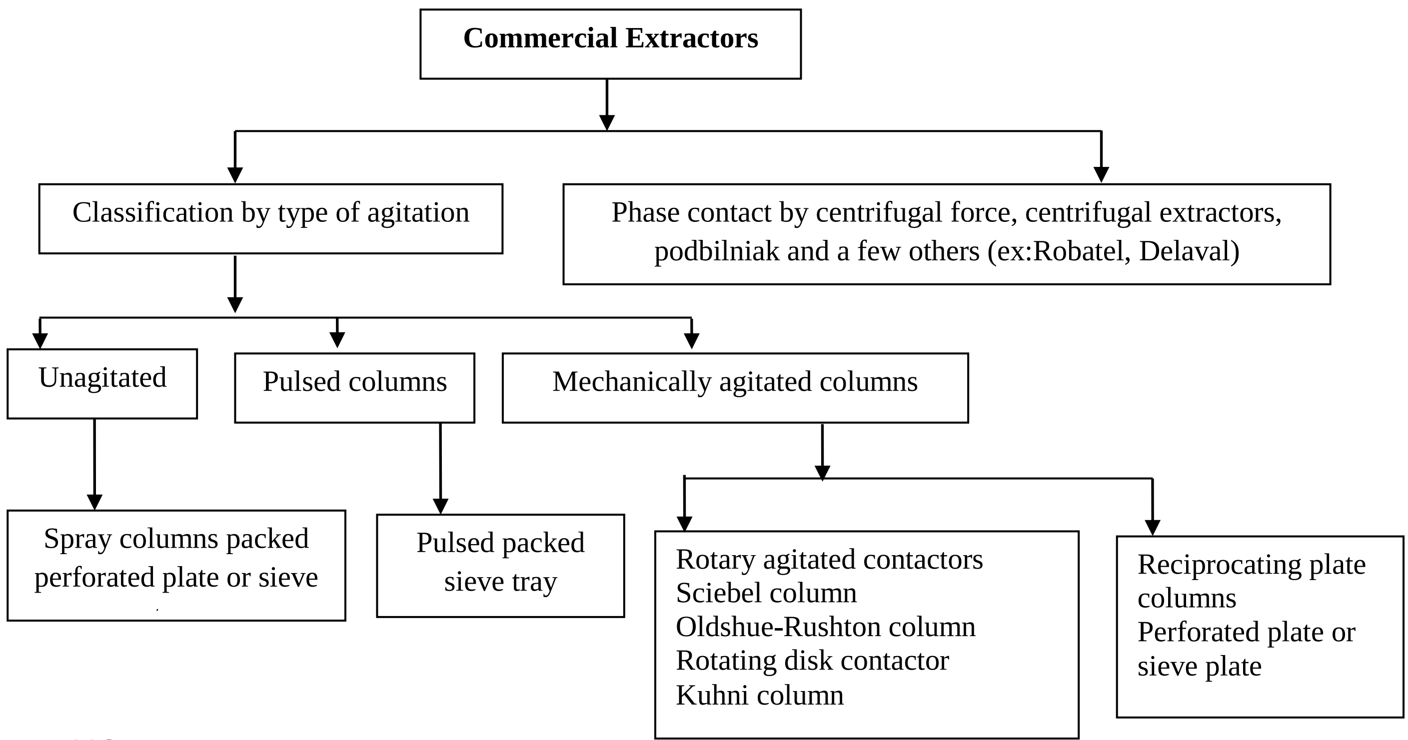
This is added to some extend in between like this, so to increase this we can say extraction efficiency also. And suppose this extracting solvent is added in the nth stage so this part is common so we can say this one that raffinate stripping section is common and the feed is added in between this we can say this one stages and after that say again say suppose this extract will be following this route and then say whatever the we can say this one another feed or we can say this one whatever R 0 we can say that is added in the first stage.

That is nothing but another raffinate actually that is coming or we can say this one (())(38:23) some of the part is actually is taken out as the product. If the situation demands like this so if we get the time also we can explain this one so in that case we can say this one the reflux will be like this R 0 minus this PE whatever the product actually we are extracting then the difference is entering here like this one this is the whatever reflux is added here.

So that is coming as the feed to the stage 1, okay. So this is R 0 minus PE that is we can say this one because R 0 we have obtained from the separator of any of the system like this where the extracting solvent actually is separated and some amount of this we can say this is extracting say what is called that career solvent or we can say this anyway if it is a feed some amount of it is taken out so we can say this one this is nothing but the reflux



Liquid-Liquid Extraction Equipment



**Source:

1. Reissinger , KH, and schroter.J selection criteria for liquid liquid extraction ,Chem Engineering 6(1978).
2. Lo,Tc, Baird , MHI and hangen ,C(Edition) Hand book of solvent extraction ,John Willey, NewYork,1983.

Equipment:

1. Mixer-settler (a) Mixer (b)Settler (c)Tower extraction .
2. Centrifugal extraction.
3. Unagitated extraction columns.

   a) Spray towers.

   b) Packed extraction columns

   c) Sieve tray columns

   d) Pulsed columns.

4. Rotary agitated extraction columns:

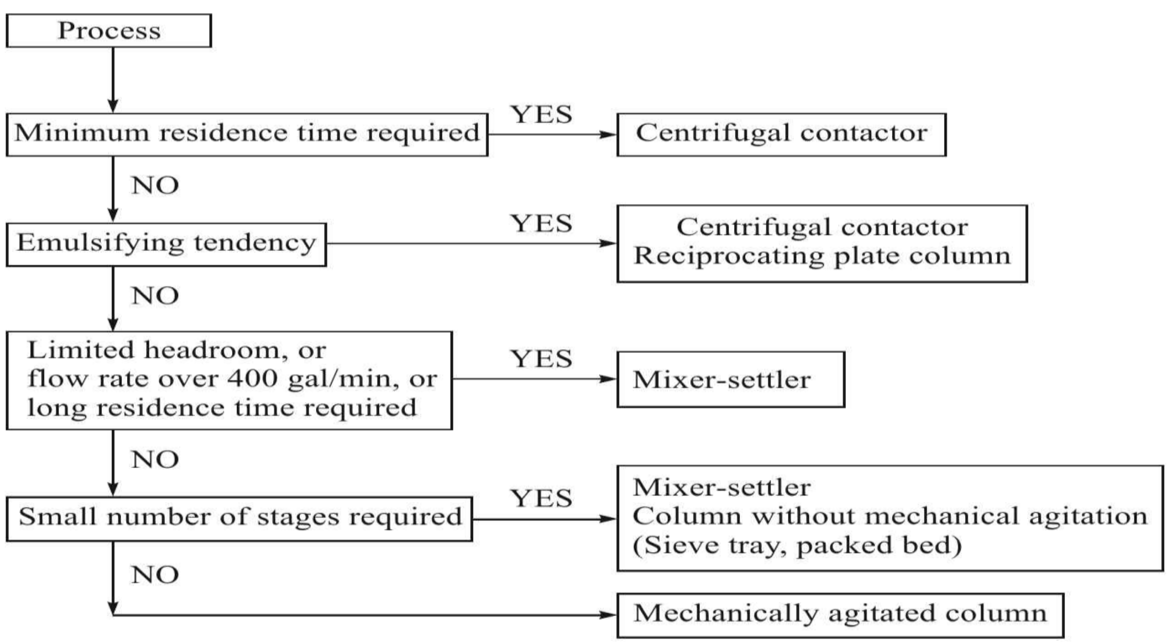
   a) Scheibel column

b) Oldshu- Rushton column.
c) Kunhi extractors.
d) Karr column.
e) Rotating disc contactor(RDC)

Selection of Extraction:

Factors:

1. Fluid properties.
2. Throughputs and phase ratio.
3. Settling characteristics of liquid-liquid dispersion.
4. Residence time in the extractors.
5. Number of theoretical stages required.
6. Presence of suspended solids.
7. Available space (floor area and height).
8. Cost and maintenance of equipment.



So thank you.

In the next class will be discussing about the design calculation of multi stage counter-current extraction.