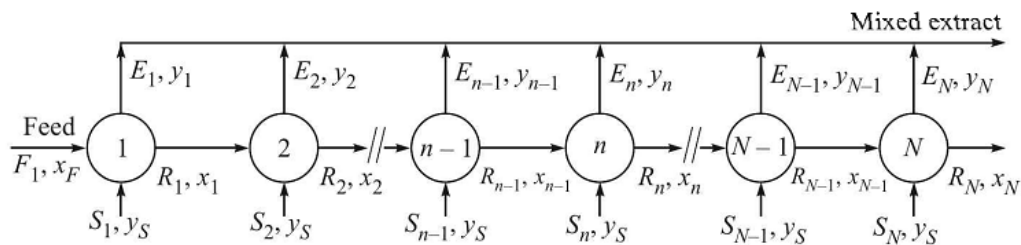


Mass Transfer Operation II
Professor Chandan Das
Department of Chemical Engineering
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Lecture - 12
Liquid-Liquid Extraction

Design Calculation of Multistage Operation

Welcome back to mass transfer operation II, we were discussing on Liquid-Liquid Extraction, in the last class we discussed the design calculation of single stage operation, now we will be discussing on design calculation of the multistage operation.

Multistage cross current extraction:



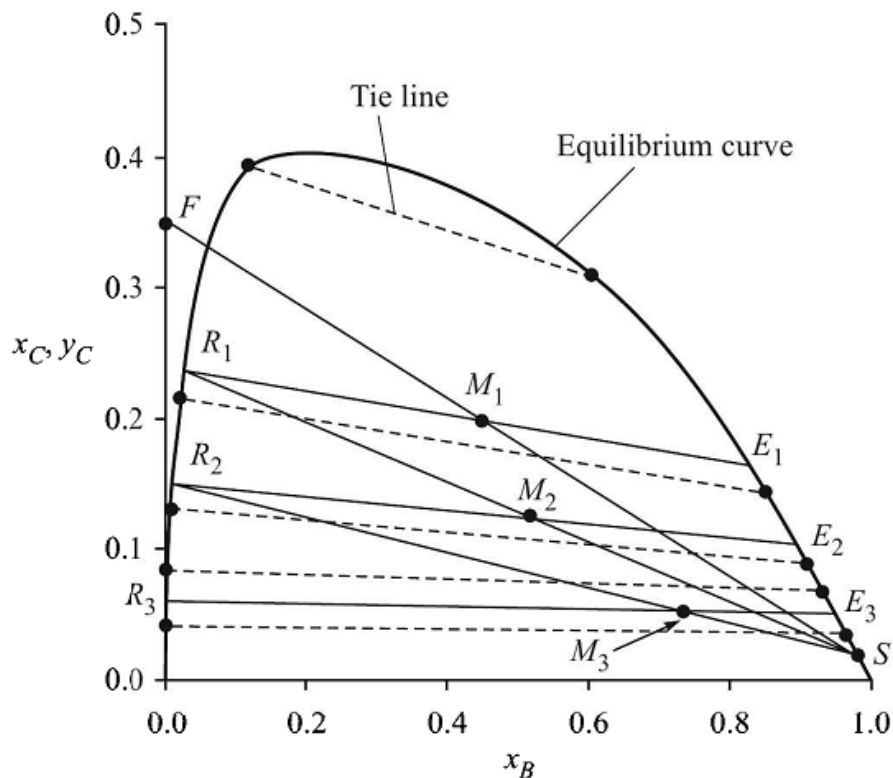
For any stage n;

Total mass balance: $R_{n-1} + S_n = M_n = E_n + R_n$ (1)

Mass balance of C: $R_{n-1}x_{n-1} + S_n y_s = M_n x_m = E_n y_n + R_n x_n$ (2)

The principle of multistage extraction calculation is similar to that of single stage extraction unit. If the flow ratios and concentrations of the streams R_{n-1} and S_n are known, point M_n can be located on the line joining R_{n-1} and S_n . Again if tie line through the point M_n is drawn, its terminals give the points R_n and E_n .

Step by step procedure:



(1) Stage 1 receives the fresh feed which does not contain any solvent.

Putting $n = 1$ in eqn. (1) we get for stage 1

$$R_0 + S_1 = M_1 = E_1 + R_1 \Rightarrow F + S_1 = M_1 = E_1 + R_1 \quad \text{---} \rightarrow \quad (3)$$

The point F is located on the x-y plane. Point S_1 is also located. The point M_1 is also located on the line joining f and S_1 by using lever arm rule or by calculating value of x_{M1} from eqn.(2). Tie line through the point M_1 is drawn and the point E_1 and R_1 are located at its terminals.

(2) For stage 2, solvent rate is S_2 and feed input rate is R_1 (first stage raffinate). Point S_2 is located on the graph. Now S_2 and R_1 are joined and the mixture of phases M_2 is located on the line. Again the terminals of the tie line through M_2 give points E_2 and R_2 .

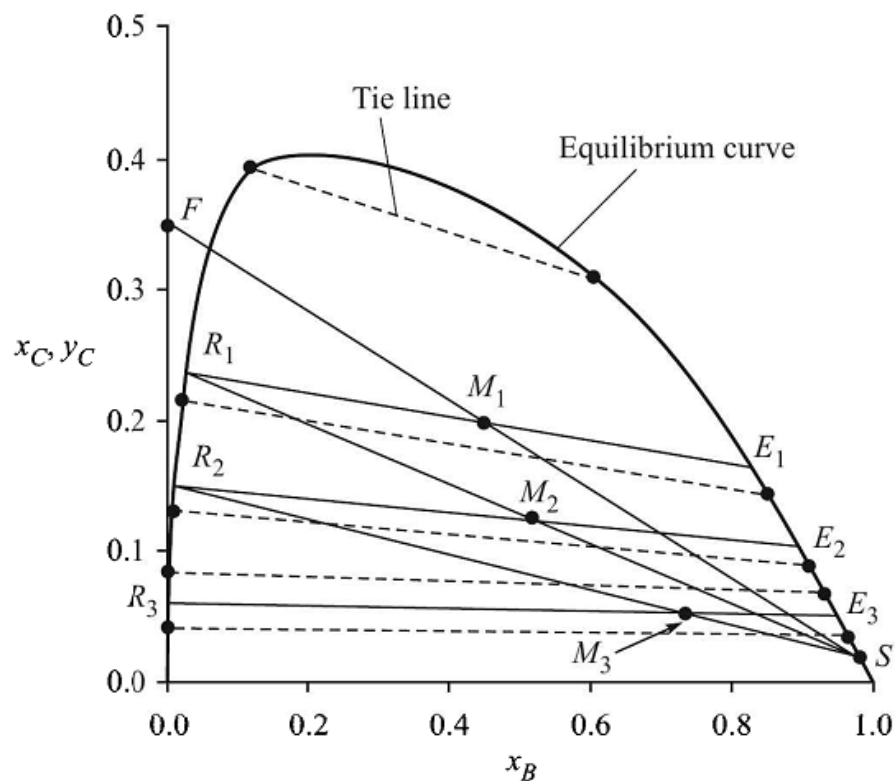
(3) The above steps are repeated for remaining stages. We have assumed that solvent concentration and input rate are known.

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.

$$F = 1000\text{kg}, \quad x_F = 0.35$$

$$\text{Stage I: } S_1 = 815\text{kg}, \quad y_S = 0.02$$

$$M_1 = 1000 + 815 = 1815\text{kg}$$

$$X_{M1} = (F x_F + S_1 y_S) / (F + S_1) = (1000 \times 0.35 + 815 \times 0.02) / (1815) = 0.202$$

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:

$$x_1 = 0.24, y_1 = 0.166$$

$$M_1 x_{M1} = R_1 x_1 + E_1 y_1$$

$$1815 \times 0.202 = R_1 \times 0.24 + (1815 - R_1) \times 0.166$$

$$R_1 = 882.97 \text{ kg}$$

$$E_1 = 932.03 \text{ kg}$$

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

$$S_2 = 950 \text{ kg}, \quad y_s = 0.02$$

$$M_2 = R_1 + S_1 = (882.97 + 950) \text{ kg} = 1832.97 \text{ kg}$$

$$M_2 x_{M2} = R_1 x_1 + S_2 y_s$$

$$1832.97 x_{M2} = 882.97 \times 0.24 + 950 \times 0.02$$

$$x_{M2} = 0.126$$

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 .

$$\text{From graph } x_2 = 0.15, y_2 = 0.105$$

$$M_2 x_{M2} = R_2 x_2 + E_2 y_2$$

$$1832.97 \times 0.126 = R_2 \times 0.15 + (1832.97 - R_2) \times 0.105$$

$$R_2 = 855.39 \text{ kg and } E_2 = 977.58 \text{ kg}$$

Stage 3: Stage 3 receives $R_2 = 855.39 \text{ kg}$ as feed

$$S_3 = 2625 \text{ kg}$$

$$M_3 = (855.39 + 2625) \text{ kg} = 3480.39 \text{ kg}$$

$$M_3 x_{M3} = R_2 x_2 + S_3 y_s$$

$$3480.39 x_{M3} = 855.39 \times 0.15 + 2625 \times 0.02$$

$$x_{M3} = 0.052$$

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 $y_3 = 0.051$

$$M_3 x_{M3} = R_3 x_3 + E_3 y_3$$

$$3480.39 \times 0.052 = R_3 \times 0.0596 + (3480.39 - R_3) \times 0.0506$$

$$R_3 = 541.39 \text{ kg and } E_3 = 2939 \text{ kg}$$

$$\begin{aligned}
 \text{Total amount of trimethylamine (TMA) leaving the stages} &= E_1 y_1 + E_2 y_2 + E_3 y_3 \\
 &= 932.03 \times 0.166 + 977.58 \times 0.105 + 2939 \times 0.051 \\
 &= 407.3 \text{ kg}
 \end{aligned}$$

$$\begin{aligned}
 \text{Total amount of TMA entering with solvent} &= (S_1 + S_2 + S_3) y_s \\
 &= (815 + 950 + 2625) \times 0.02 \\
 &= 87.8 \text{ kg}
 \end{aligned}$$

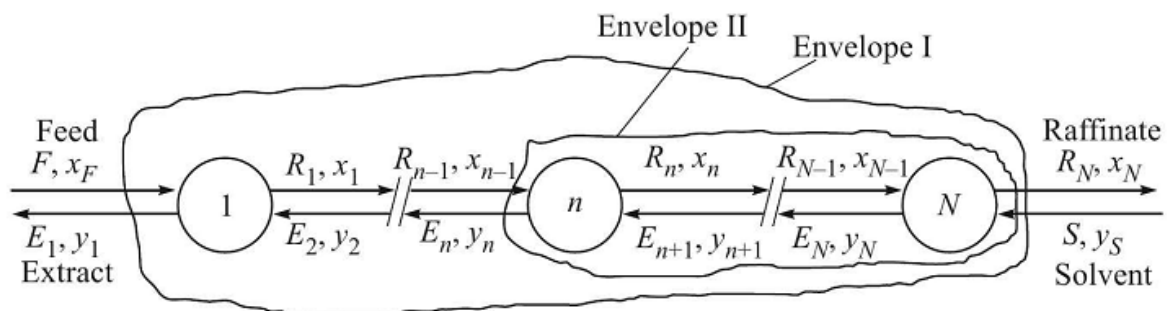
$$\begin{aligned}
 \text{Net amount of TMA removed} &= (407.3 - 87.8) \text{ kg} \\
 &= 319.5 \text{ kg}
 \end{aligned}$$

$$\text{Total amount of solute in the feed} = 1000 \times 0.35 = 350 \text{ kg}$$

$$\text{Fraction of TMA removed} = (319.5/350) \times 100 = 91.3\%$$

Differential Extractions:

Continuous countercurrent Multistage Extraction



Extract and raffinate streams flow from stage to stage in countercurrent and provide two final products, Raffinate, R_{NP} and extract E_1 . For a given degree of separation, this type of operation requires fewer stages for a given amount of solvent or less solvent for a fixed number of stages than cross current method.

Total material balance over the entire plant (Envelope 1):

$$\text{Solute balance: } Fx_f + Sy_s = Mx_M = E_1 y_1 + R_{NP} x_{NP}$$

$$E_1 - F = S - R_{NP} = \Delta$$

Where Δ is a stream obtained by removing a mass F of the feed from first stage extract E_1 or by removing a mass R_{NP} of the last stage raffinate from solvent S .

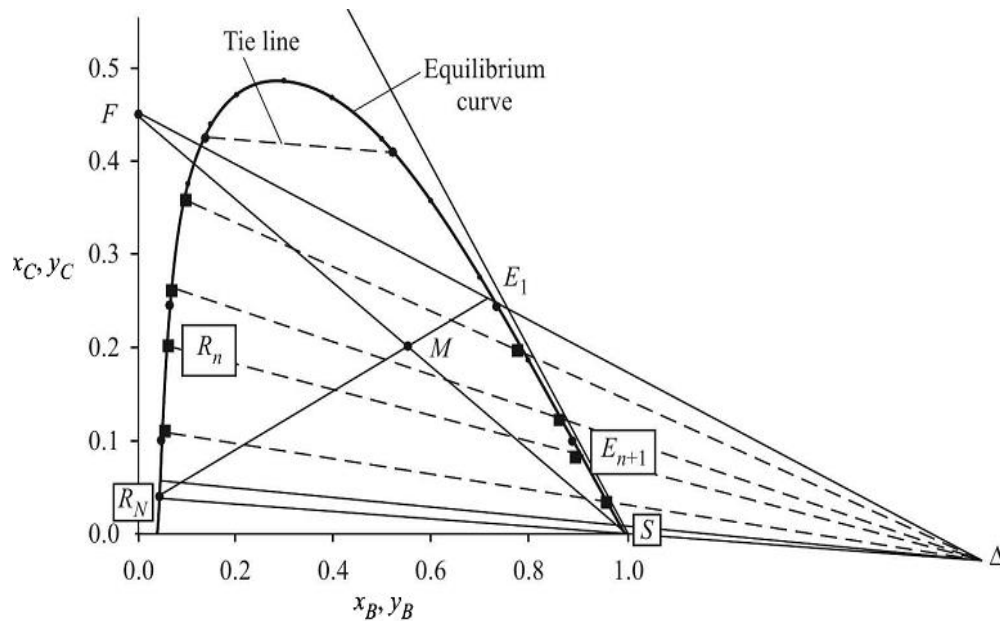
A material balance for stages through N_P is (Envelope 2):

$$R_{S-1} + S = R_N + E_S$$

$$E_S - R_{S-1} = S - R_N = \Delta.$$

Since stages are ideal. The streams E_S and R_S leaving the S^{th} stage are at equilibrium.

A step by step procedure:



Now, will be discussing about the continuous counter multistage extraction process, so in this case actually the feed is entering from one side of this we can say system and with 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 solute 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 actually is 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 this are flowing, so we can say this one the extracting solvent will flow in this direction and the ultimately this final extract will be leaving from the first extraction stage, where the feed was added.

And the feed will be this moving in this direction like this whatever we have this one tried in this counter current for cross current extraction, here we say both the extract and raffinate 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 this solvent is actually is carried forward, so we can say this one extract and raffinate stream flow from this 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 numbers 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 few can say stages then cross current method like this whatever we have discussed previously, so compared to the cross current extraction process the counter current extraction process is we can say this economic.

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Continuous Counter-current Multistage Extraction

Total material balance over the entire plant (Envelope 1):

$$\text{Solute balance: } Fx_F + Sy_s = Mx_M = E_1y_1 + R_Nx_N$$

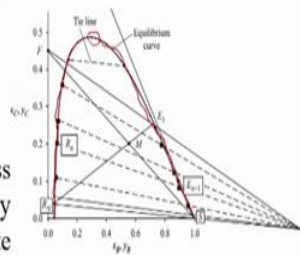
$$E_1 - F = S - R_N = \Delta$$

where Δ is a stream obtained by removing a mass F of the feed from first stage extract E_1 or by removing a mass R_N of the last stage raffinate from solvent S .

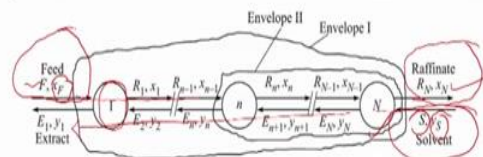
A material balance for stages through N is (Envelope 2):

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

$E_n - R_{n-1} = S - R_N = \Delta$ Since stages are ideal, the streams E_n and R_n leaving the N^{th} stage are at eqm.



Continuous Counter-current Multistage Extraction



Extract and raffinate streams flow from stage to stage in counter-current and provide two final products, Raffinate, R_N and Extract E_1 .

For a given degree of separation, this type of operation requires fewer stages for a given amount of solvent or less solvent for a fixed number of stages than cross-current method.

So, like this one here will be 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 say we have this we can say this binary zone like this, this is the raffinate arm and suppose the (47:57) point is somewhere here and then this is the extractor, and say it is a right angular triangular diagram now we can see this will be doing for the entire plant like this the 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 in the last stage say solvent is added and from the first stage extract is this one taken out, so will be what will be doing this one if

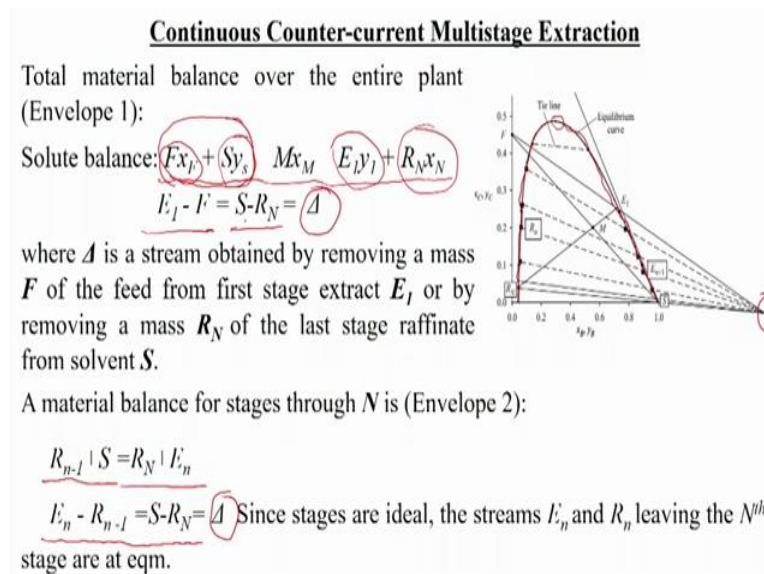
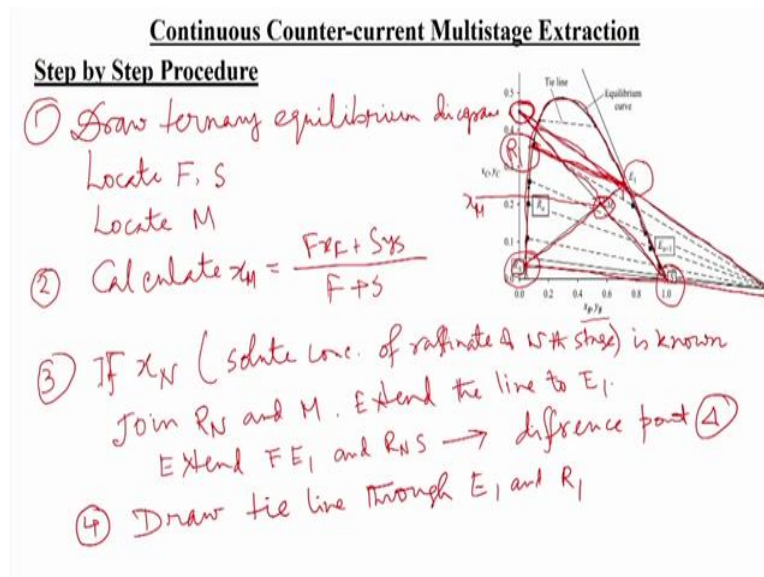
we do the material balance then we can say this one FxF plus SyS is equal to we can say this MxM that is nothing but this $E1y1$ plus $RNxN$.

So that is we can say this one this whatever this in the first stage that is x_F that is SyS in the last stage and we can say this one extract is coming out from the first stage $E1y1$ and raffinate is exiting from the last stage that is with this solute amount will be RN into x_N , so whenever we will be doing this suppose if we say this one $E1$ minus F means in any of the stages if we say what is the resultant of the flow rate then that is nothing but we can say this one $E1$ minus F that is then in case of this we can say S minus RN , so will be getting always the common factor like this, so that we now design or we can say this one as the Δ so this is nothing but this Δ is a stream obtained by removing a mass F of the feed from the first stage and this extract $E1$ or by removing we can say this one mass RN of the last stage raffinate from the solvent S , or we can say this in any stage if we say 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 the material balance in the envelope 2 like this in the small envelop like this so there actually we will be getting like this Rn minus 1 plus S is equal to Rn plus En , so from here will be also getting extract minus raffinate that will be giving like this S minus Rn , so this is also Δ so if we do this one repeatedly for all the stages also will be getting one difference that is we can say this one say extract minus raffinate so that is we can say this is the Δ .

So, that Δ will be actually will be getting here how to get this one will be discussing in detail like this, so since these stages are ideal so the streams En and Rn living the N th stage are in equilibrium. So, we are assuming that all the stages this one we can say this one attend equilibrium before living the, this one extraction unit.

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So, we can say will start this stage by step by step calculation procedure for this we can say multistage counter current extraction unit this is very simple easy to understand also and so we will be doing this one, now we need to draw this one what I told actually 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, will be now drawing this one from this we can say equilibrium data point will be getting this we can say binary phase zone by drawing this we can say equilibrium line so this one we can say this one so we can say this one suppose this is the plate point so left hand side will be raffinate arm and right hand side will be the extract arm 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 any types say what we discussed this one it may be right angular triangular diagram, it may be equilibrium triangular diagram it may be solvent free vases also, so whatever will be this one but for most of the this one calculations we prefer this right angular triangular diagram for convenience will be also discussing also this one when we will solve the problem, this is a very convenient way of this one of drawing this right angular triangular diagram.

Then, we need to locate we can say this feed point as well as the solvent point, so this feed point that is composition is already given how much amount of the solute is there suppose in this case suppose around 45 percent solute is present in the feed, so it is like this x_F will be 0.45, whereas this we can say this one as x_B will be 0, so this is the feed point and this solvent point this we can say this one that is S will be almost say we can say this one pure or some amount of the small amount of the solute may be present based on that actually it will be placing near to this we can say this one in the x_P and y_B axis.

If it is 100 percent pure then it will be touching this y axis is equal to 0 and if the y_S is not 0 then it will shift toward we can say this one if this following this equilibrium diagram, so may be somewhere here like this, now we need to get this one this we need to add this AF and S so we will be adding this one. Now will be getting this point M , so M will be located now say we need to locate M that is the mixture point, so this mixture means if we know M then from the component balance will be getting suppose this x_M .

So from there will be getting this M , so this now we need to calculate this this second step will be like this, we need to calculate x_M from this component balance like this we will be doing this Fx_F plus Sy_S by F plus S , so if we know all this 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 the solute concentration in the feed and this solvent flow rate and then we can say this one total amount of heat plus solvent, so we will be getting now this x_M .

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 that is we can say this one solute concentration concentration of raffinate that is leaving the last stage actually raffinate of N th stage or we can say this one or N th stage or that is the final stage is known suppose this is given as this known parameter.

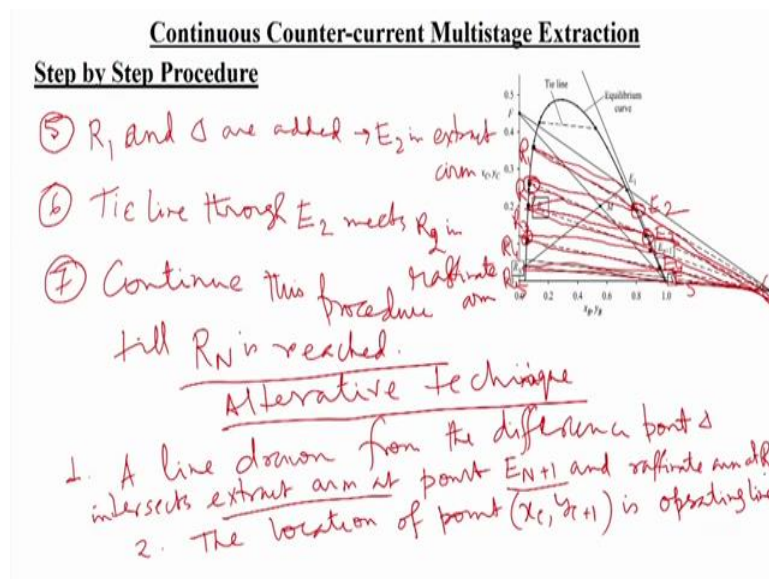
Then we can say this one say RN and M are joint like this that is if it is given then we can say this one in the raffinate whatever the solute concentration, suppose x_N is given so it will be like this somewhere else then we can say this one we have this RN is known suppose this x_N is known then we will be adding this RN and joining M then we will be extending to the extract arm then will be getting this E1. So now we will be joining join suppose RN and say and M then extend this one extend the line to E1 that is now in the we can say this extract arm, now we have this one F is available.

We know then E1 say we know that this the different points actually that we found out that suppose E1 minus F or S minus RN that is giving this delta, so we can say this one whenever we have this F and E1 we have this one, so we can say this one E1 minus F or we will be getting now say S minus RN suppose we have this S and RN so from there will be getting this same delta like this now we have E1 and F, suppose we will be joining one line we have RN and S, so from this we can say this one S minus RN or E1 minus F, were it be meeting so that is nothing but delta.

So from there actually will be getting this we can say from there will be getting this difference point, so if we extend F E1 and RN S then will be going to this difference point like difference point so this delta, so will be getting this delta so for all the stages also if we extend this one it will meet a particular point that is called say difference is difference point is that is delta.

Now, the 4 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 E1 and R1 like this will be doing this one, so we have now this one say we can say E1 we have this if we get this we can say this one tie line so will be getting R1, now drawing this one like this from here will be drawing this equilibrium line tie line, here will be getting this R1, so this is the tie line then will be getting this R1 here whenever this R1 we have this one it is nothing but the whatever the raffinate is obtained from the stage 1 that is nothing but the feed to the stage 2 and now we have this delta so if we do this one if we repeat this one we can say this one say the stage 5 will be like this.

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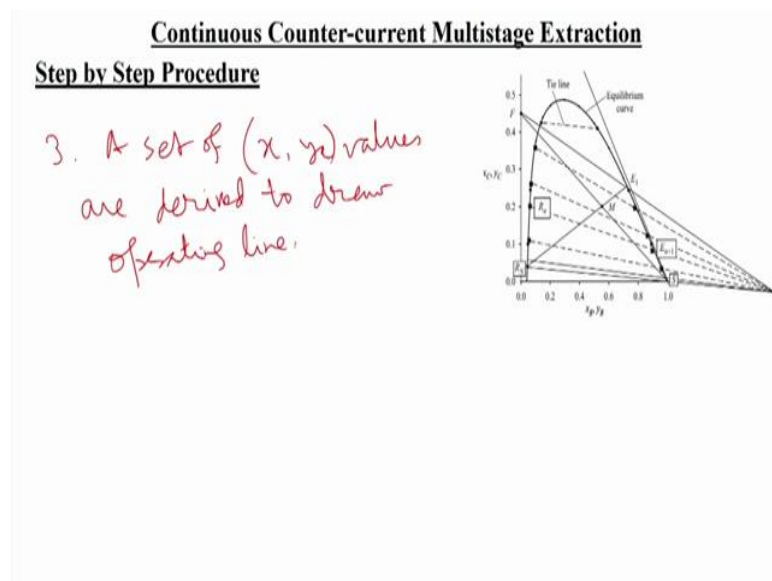
Now, we have this delta point we have suppose this is R_1 we have this one because this is the tie line from E_1 and then we can say this R_1 and Δ so these two are added, now we will be getting this E_2 here. So, then we will be obtaining this then this are added then will be getting E_2 in the extract arm so that will be we can say this one now again this 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 will be doing this we can say tie line through this E_2 meets R_2 in raffinate arm, so this is R_2 that is the feed to the stage 3.

So now we will be continue this one suppose this again this we will be adding this R_2 and Δ 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 continue and then we will be getting say one time we will be reaching like this whenever we have this R_3 will be joining with Δ then will be getting say 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 we will be again 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 say suppose R_5 then will be adding this one.

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

So another alternative technique is there like this we can say this alternative technique, so what will be doing a line drawn from the difference point delta intersects extract arm at point actually E_{N+1} and at raffinate arm this extract term that is in $N+1$ and raffinate raffinate arm at R_N , now we can say this one, then second stage will be the location point like this we can say this one x_c and y_c plus 1 that is nothing but the is operating line, that is on suppose this x_c and y_c plane we will be drawing this one x_c , y_c plane also we will be drawing this one then we can say.

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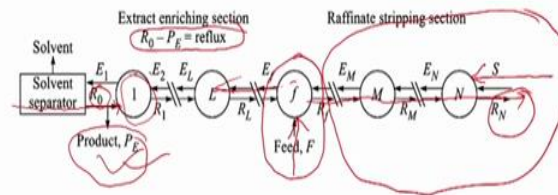
Then, a set of x_c and y_c values are derived to draw operating line, so that also we can do this one so we can say this using this alternative technique also will be doing this we can say counter current multistage extraction system.

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Continuous Counter-current Extraction with Reflux

The solute concentration in the extract leaving a counter current cascade can at best be nearly at equilibrium with the feed stream.

It is possible to have a richer extract by use of reflux at the product end of the cascade.



Now, we have this another system that is we can say this one continuous current extraction with some reflux, this is we can say this one the solute concentration in the extract leaving the counter current cascade can based be nearly at equilibrium with the feed stream, so it is possible to have a richer extract by the use of 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 f, fth stage and then say this one we can remember that we can this equilibrate equivalent make this equivalent like this, say this raffinate from this fth stage we will follow this route means a and then we will be getting the final raffinate from the last stage or Nth stage, where the we can say this one extracting solvent was added.

Like this, this is counter current operation but feed is not added at the stage 1 this is added to some extent in between like this in between like this, so to increase this we can say extraction efficiency also and this suppose 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 the feed is added in between this we can say this one stages and after that say again say suppose this extract we 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 is that is coming or we can say this one first stage.

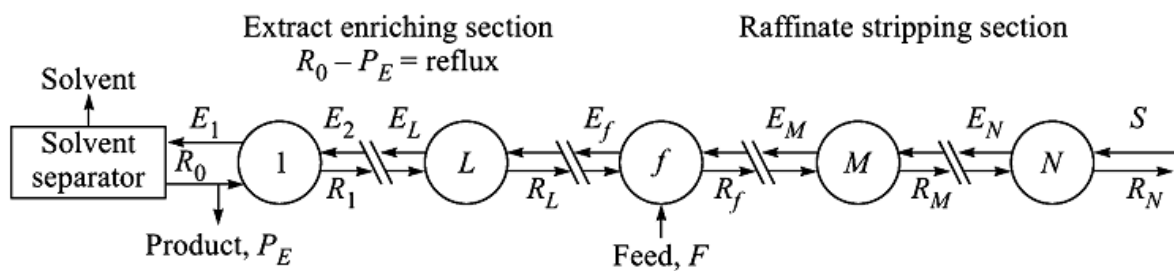
Some of the part actually is taken out as the product if the situation demands like this say if we get the time also we can explain this one, so in that case we can say this one the reflux will be like this R_0 minus this P whatever the product actually we are extracting then the

difference is entering here like this one this is the we can say this one whatever the reflux is added here.

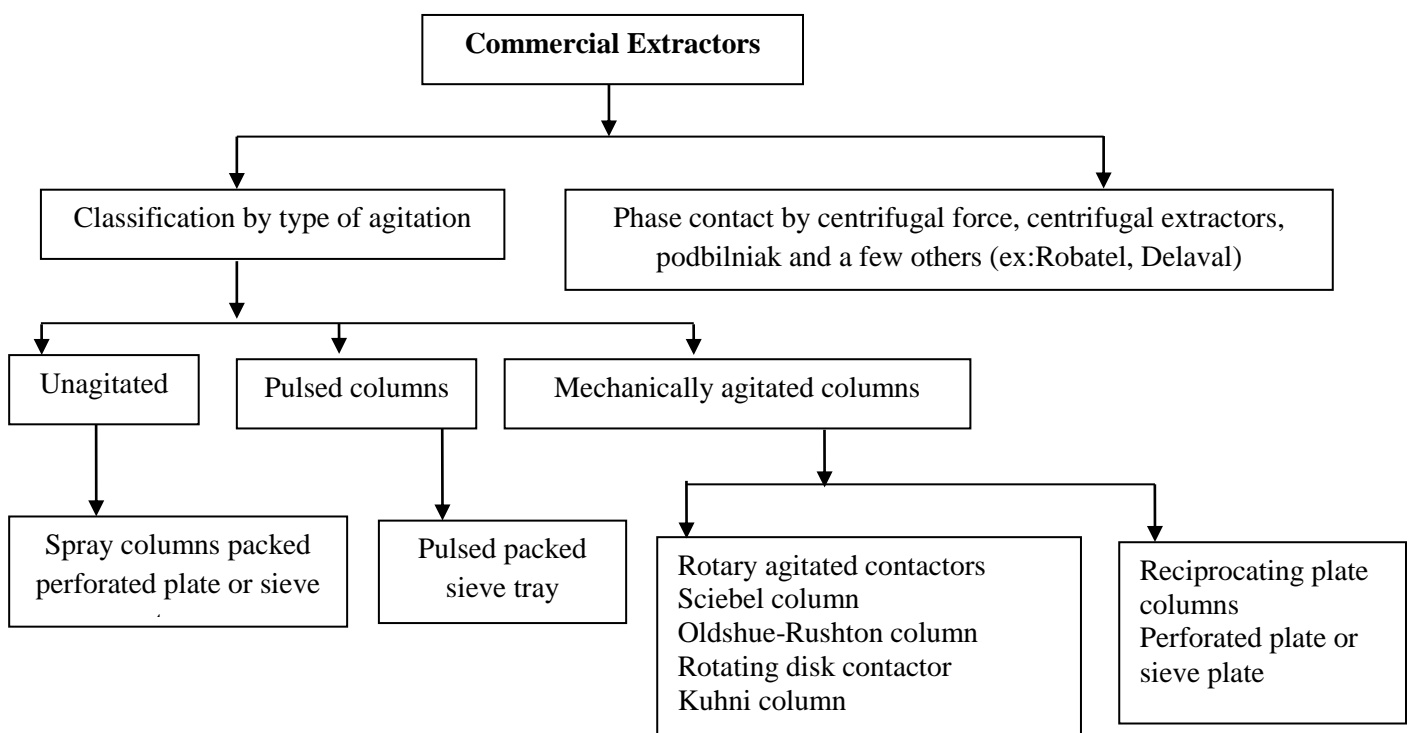
So that is coming as the feed to the stage 1, so this is R_0 minus P that is we can say this one because R_0 we have obtained from this separator from any of the system like this were the extracting solvent actually is separated and some amount of this we can say this extracting say, what is called that carrier solvent or we can say this one anyway if it is a feed some amount of is taken out so we can say this one this is nothing but the reflux.

Continuous counter current Extraction with Reflux:

The solute concentration in the extract leaving a counter current cascade can at best be nearly at equilibrium with the feed stream. It is possible to have a richer extract by use of reflux at the product end of the cascade.



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)

So, thank you. In the next class, we will be discussing about the design calculation of multistage counter current extraction.