Mass Transfer Operation II
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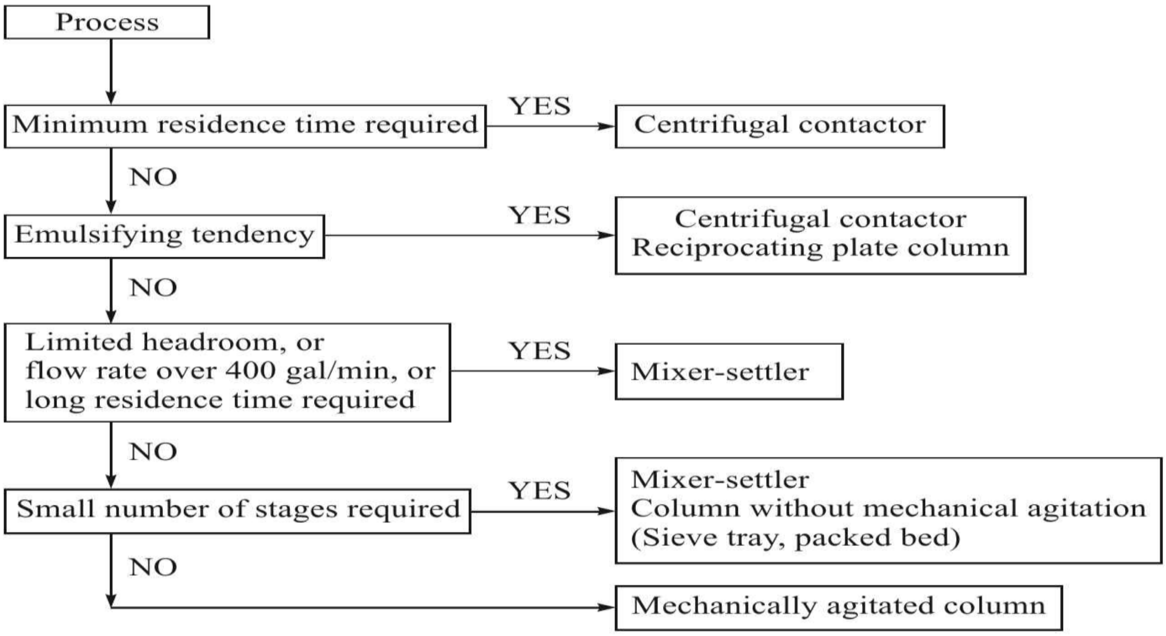
Lecture - 14
Design calculation of multistage counter-current extraction

Welcome back to Mass Transfer Operations II. We were discussing on liquid-liquid extraction. Now, we will be discussing on the design calculation of multistage counter-current extraction.

Important factors that govern the selection of an extractor:

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.

Simple guidelines for extractor selection



Minimum solvent rate determination

An infinite number of stages are required.

To determine minimum solvent flow rate, S_m we need to follow the following steps:

1. Points *F, S* and R_N are located.
2. R_N and *S* are joined.
3. A tie line through *F* is drawn and extended to R_N-*S* extension.
4. As per lever rule: $\frac{F}{S_m} = \frac{M_m S_m}{F M_m}$

<u>**Multistage countercurrent extraction**</u>**:**

Problem 4: An aqueous solution of acetic acid (35% acid) is to be extracted with pure di-isopropylether in a countercurrent cascade at a rate of 2000kg/hr. A solvent rate of 2970 kg/hr is suggested. The raffinate stream must not contain more than 10% of acid in it. Determine the number of ideal stages required and minimum solvent rate for the job. The liquid-liquid equilibrium data at 20°c are given below:

Water layer (raffinate) ,mass %			Ether layer(extract),mass%		
X_a	X_B	X_C	Y_A	Y_B	Y_C
0.981	0.012	0.0069	0.005	0.993	0.0018
0.971	0.015	0.0141	0.007	0.989	0.0037
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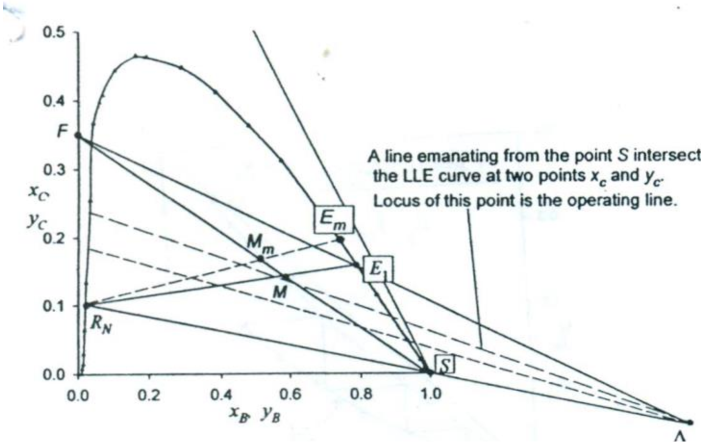
0.589	0.044	0.367	0.069	0.715	0.216
0.415	0.106	0.443	0.108	0.581	0.311
0.371	0.165	0.464	0.151	0.487	0.362
0.19	0.38	0.43	0.19	0.38	0.43

Before that, actually, we will be, we need to discuss about the important factors that govern the selection of an extractor. The first will be like this, fluid properties. So what type of fluids we are handling for the extraction like whether it is very high viscous liquid or low viscous liquid or we can say, whether the concentration of the solute is very high or low. And what type of extracting solvent we will be using which is compatible with the feed solvent feed.



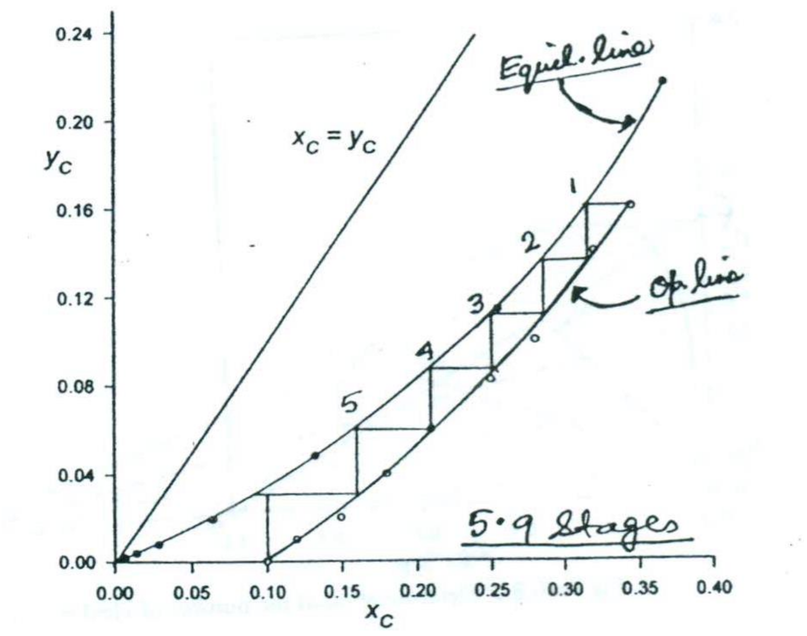
A line emanating from the point S intersect the LLE curve at two points x_c and y_c. Locus of this point is the operating line.

F=2000kg/hr ; x_F=0.35,   F(x_B, x_C=0,0.35)

S=2970kg/hr; y_s=0,   S(y_B, y_c=1,0)

$$x_M = \frac{2000*0.35+2970*0}{2000+2970} = 0.141.$$

Locate M on the line FS. Join R_{NP} M and extend to meet extract arm of LLE curve at E_1. Join and extend FE_1 and R_{NP} S to get the difference point, Δ. Draw a set of lines from Δ to intersect the LLE curve at a set of points.



Operating line:

x_C	0.1	0.12	0.15	0.18	0.21	0.25	0.28	0.32	3.4
y_C	0	0.01	0.02	0.04	0.06	0.082	0.10	0.14	0.16

The equilibrium line is drawn as x_C Vs y_C.

The opsating line is drawn by joining the above points.

Steps are drawn between equilibrium and opsating line to find out number of ideal stages, N=5.9.

To obtain minimum liquid rate, Δ_m can be obtained by drawing Tie line (FE_M) through F. Join R_{NP} and E_M . The line intersects FS at M_M.

$$\frac{F}{S_m} = \frac{M_m \Delta_m}{F \Delta_m}$$

$$S_m = 2000 * \frac{5.6}{5.55} kg / h = 2018 kg / hr.$$

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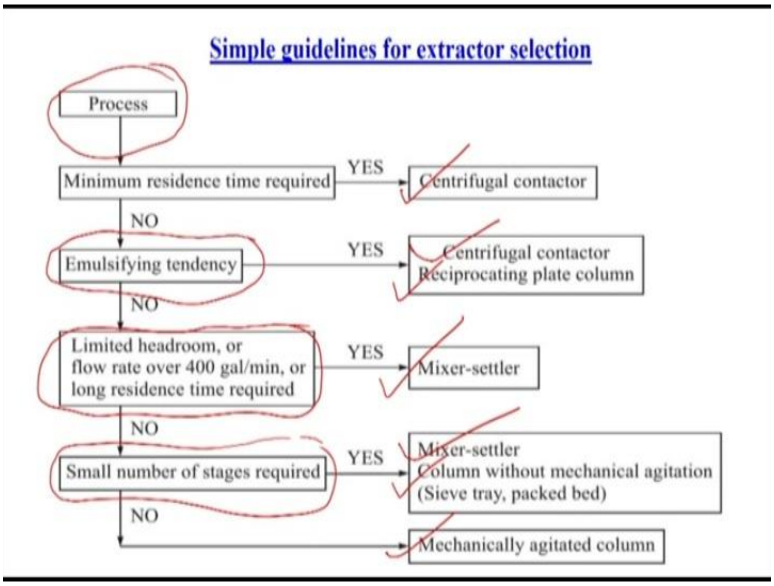

Simple guidelines for extractor selection

Process

Minimum residence time required — YES → Centrifugal contactor

NO

Emulsifying tendency — YES → Centrifugal contactor
Reciprocating plate column

NO

Limited headroom, or flow rate over 400 gal/min, or long residence time required — YES → Mixer-settler

NO

Small number of stages required — YES → Mixer-settler
Column without mechanical agitation (Sieve tray, packed bed)

NO → Mechanically agitated column

And based on that actuallysay there is one thumb rule type, we can say this one for the selection of the extractor. Suppose, the, we have a particular process means where the material is to be separated using one carrier-extracting solvent. So there, the minimum residence time required, say if it is a target, then if it yes then we shall go for centrifugal contractor. If we say that minimum residence time requirement is not mandatory then we will be, we will find this next criteria like emulsifying tendency. If emulsifying tendency is present then we shall go for this, either centrifugal contractor or reciprocating plate column.

So, if the emulsifying tendency is not present there then we can say this one, whether this limited headroom or we can say this one, flow rate over 400 gallon per minute is there, or we can say this one, long residence time requirement is there. Because we started with this, whether this long residence time is there. Then, if it is yes, then we can say this one we can go for mixer-settler.

If that headroom availability is more or we can say this flow rate is, we can say this one, below 400 gallon per minute or we can say this one, residence time requirement is not so high like this, then we can say, we will be asking for that, whether this small number of stages are required. If it is yes, then we shall go for mixer-settler. Or we can say this one, we can go for this column without mechanical agitation like this, like sieve tray or packed bed like this. And if you can say small number of stages is not required at all, then we can say this one, we can go for mechanically agitated column for the effective separation using this extraction process.

So, these are not the exhaustive, we can say, procedures. There are maybe so many other factors that we discussed earlier also, we need to consider those. But these are the, whatever we are now showing this one, it is the thumb rule for this selection of the particular extract or for a particular application.

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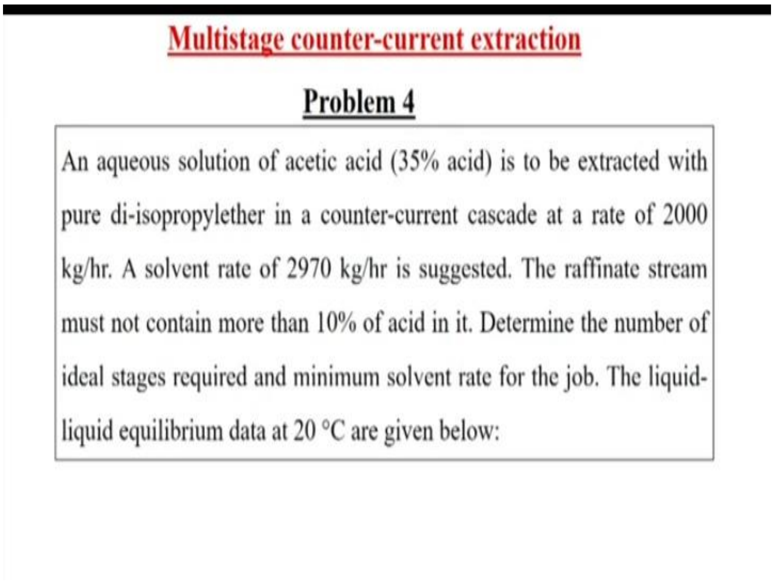

Multistage counter-current extraction

Problem 4

An aqueous solution of acetic acid (35% acid) is to be extracted with pure di-isopropylether in a counter-current cascade at a rate of 2000 kg/hr. A solvent rate of 2970 kg/hr is suggested. The raffinate stream must not contain more than 10% of acid in it. Determine the number of ideal stages required and minimum solvent rate for the job. The liquid-liquid equilibrium data at 20 °C are given below:

And now we will be solving one problem on the multistage counter-current extraction. The problem is very straightforward. An aqueous solution of acetic acid, where 35 percent acid is present, so we can say Xf is equal to point 35 is to be extracted with pure di-isopropylether in a counter-current cascade at a rate of 2000 kg per hour.

A solvent rate of 2970 kg per hour is suggested. The raffinate stream must not contain more than 10 percent of acid in it. So, we can say this one, whenever the last stage it will, when this we say feed will leave last stage there, say we can, we can say Xn that is from the last stage will be 0.1, ok. So, now we need to determine the number of ideal stages required and minimum solvent rate for the job. The liquid-liquid equilibrium data is given in the next slide.

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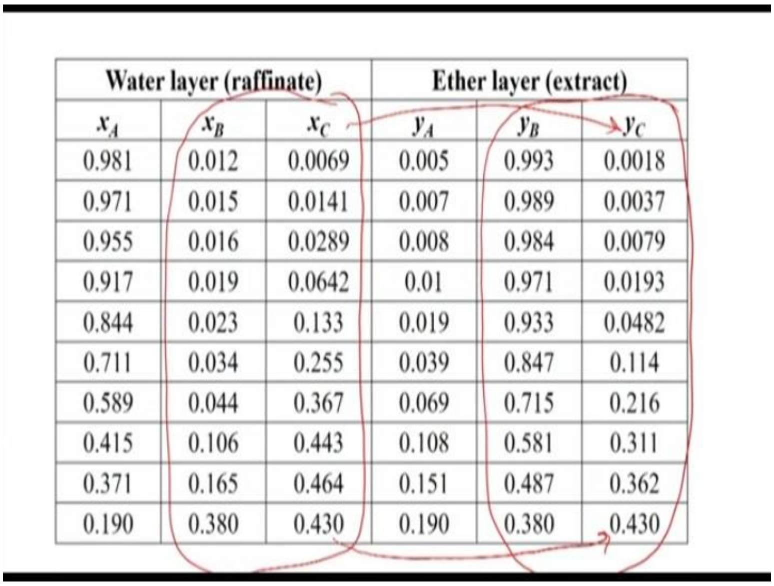

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0.371	0.165	0.464	0.151	0.487	0.362
0.190	0.380	0.430	0.190	0.380	0.430

Like this, in the raffinate phase and the extract phase, suppose this all this Xb versus Xc, these are given, so we can say this one of using this data points we will be able to get the raffinate arm and using this Yb versus Yc, we will be able to draw the extract arm. And all this Xc and Yc, these are all in equilibrium. So, we can say this one, the, this is actually plate point. So, this we can say Xc is equal to Yc, that is 0.43, that is the plate point. So, these informations are all available now.

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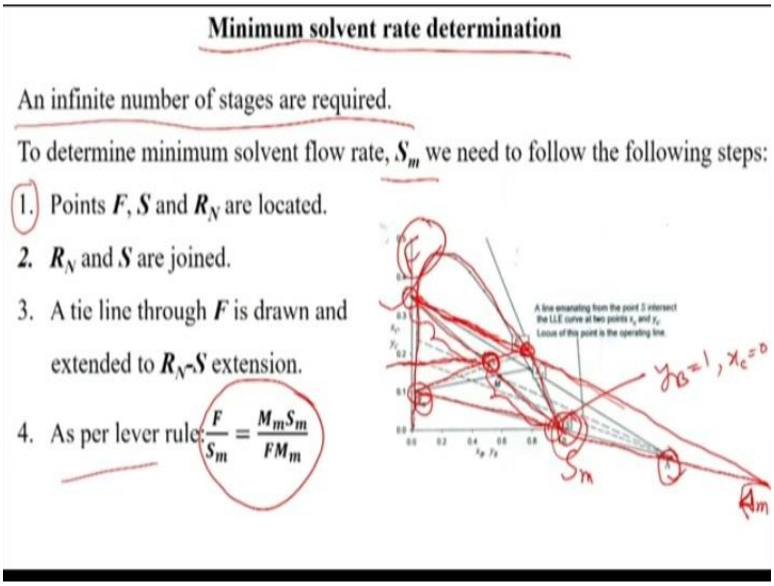

Minimum solvent rate determination

An infinite number of stages are required.

To determine minimum solvent flow rate, S_m we need to follow the following steps:

1. Points F, S and R_N are located.
2. R_N and S are joined.
3. A tie line through F is drawn and extended to R_N-S extension.
4. As per lever rule: $\frac{F}{S_m} = \frac{M_m S_m}{F M_m}$

Now, actually we need to discuss first these how to measure the minimum solvent rate. This is very easy to determine. Suppose this, an infinite number of stages are required if we say, if we use this minimum solvent flow rate, but at the same time, we can say this one, we can calculate that how much minimum amount of solvent we can use for a particular separation. So, for that to determine the solvent flow rate Sm, say minimum solvent flow rate, we need to do the following steps or we need to follow these steps like this.

The first step is, say we need to locate this point F, then point S and say Rn. So, for this case, suppose for this problem, Rn is given say we can say this one, that point 1. So, here actually this Rn is there, ok. Then, we need to, we can say, we need to add this F and S. Then Rn and S actually, this is added, joined. Say Rn and S, that is joined and it is extended like this. And a tie line through this F is drawn and extended to, we can say this one, Rn, however this we can say this one from this F is drawn and extended to RnS, ok.

Suppose on tie line that is drawn through this one, that is, where we can say this one, it will pass through Em. Then we need to extend this one, somewhere it will meet. We can say, here we will be getting delta m, ok. So, whatever the procedure we followed for this regular, we can say this one, counter-current extraction process. Here we need to get that say, we need to make one tie line, that is, through this F, that is we can say, we need to do this one very carefully. We need to find first that where we will be getting the tie line. Because you see, we have this Xc versus Yc and we need to find near this Xf value and then we need to get this one. Then we will be getting this Em. Through this Em, we need to extend this one.

In case of this regular extraction process, through E1, from F actually we extended and we got this delta. So whatever the other delta m or we can say minimum flow solvent flow rate we will be getting, that it will be there in the same line with Rn, S, this in that same line. But maybe, it is to some far away from this delta. So, it definitely it should be far away from this delta, ok.

So, as per the lever rule we can say this one, if we say, F this F and this say S minimum, because this S minimum also will be at the same point because you see, we have plotted here the solute concentration. That is why we have got this binary phase diagram. So, S and Sm, both will lie in the same. Here, in this particular problem, this S the location is at this, we can say this one where, Yb is equal to 1. So, here actually Yb is equal to 1 and Xc is equal to 0. Xc or Yc, that is equal to 0 because here there is no solute in the extracting solvent.

So this is Sm. That is why we can say this one whenever we will be, as per this lever rule, we can say this feed flow rate by the solvent flow rate minimum. Then will be like this feed means then this Xm by this one. This MmSm by this, we can say, this distance FMm, ok.

So, this Mm is actually obtained when we have drawn the tie line through F upto delta, then we need to draw this one line actually through this Rn and Em, ok. Then when it will cut a particular point in between this F and Sm that gives that mixture concentration in the minimum solvent flow rate, ok. So that that is why this Xm value is to some extent higher than the concentration of the solute in the mixture, where we can say this one, it is not this minimum solvent flow rate.

Or in other words, we can say this one, extracting solvent amount is small that is why, the solute concentration in the mixture is greater compared to the solute concentration in the regular mixture, ok.

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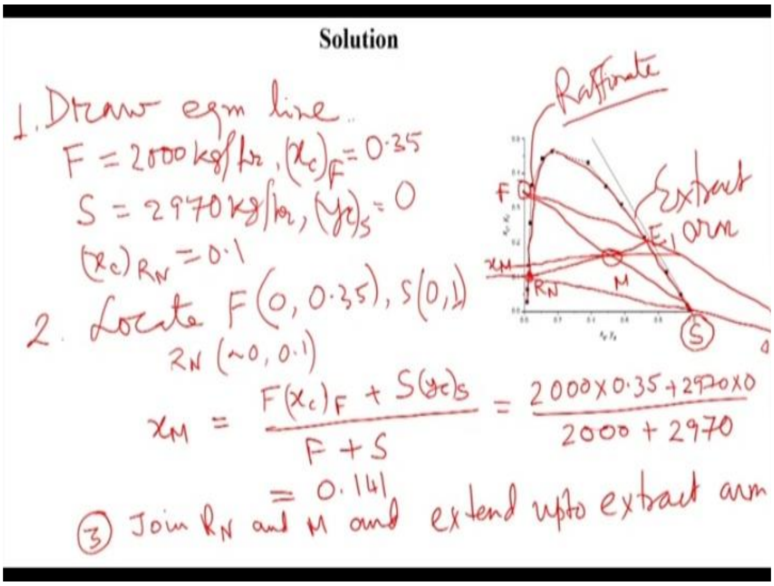


Solution

1. Draw eqm line

$F = 2000\ kg/hr,\ (x_c)_F = 0.35$

$S = 2970\ kg/hr,\ (y_c)_S = 0$

$(x_c)_{R_N} = 0.1$

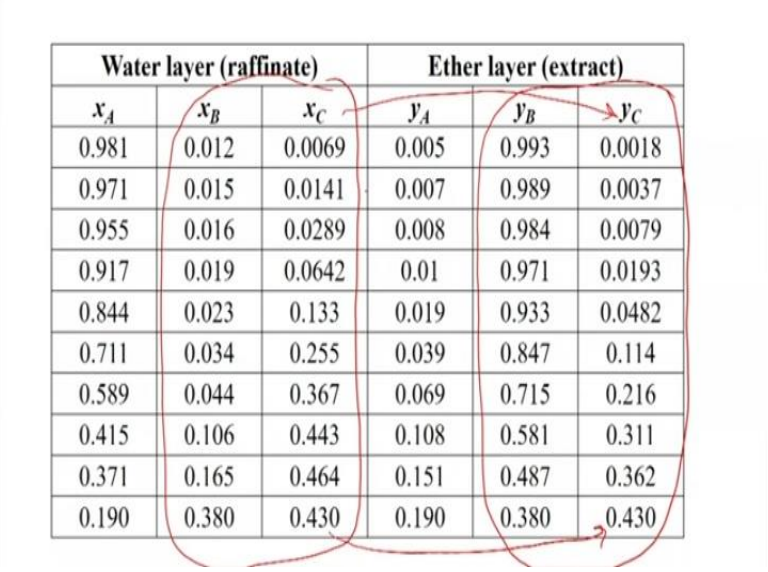
2. Locate $F(0, 0.35),\ S(0,1)$

$R_N(\sim 0, 0.1)$

$$x_M = \frac{F(x_c)_F + S(y_c)_S}{F+S} = \frac{2000 \times 0.35 + 2970 \times 0}{2000 + 2970}$$

$$= 0.141$$

③ Join R_N and M and extend upto extract arm



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0.371	0.165	0.464	0.151	0.487	0.362
0.190	0.380	0.430	0.190	0.380	0.430

So, now we will be solving this problem. So first we need to do this one. First we need to draw this raffinate arm. Say that is upto this one. So this is we can say this one. Raffinate arm and say this one is extract arm, ok.

So, first step is you can say this one we have all this so we need to draw equilibrium line using this equilibrium this one values, like this Xb versus Xc, that is for raffinate arm. And Yb versus Yc for extract arm.

So, that is done. Then, the second step will be like this, we whatever we have given in this problem, like feed flow rate is given 2000 kg per hour. And say, Xc in the F actually is given as 0.35, 35 percent this oil is there. And solvent is given as 2970 kg per hour and this Yc that is in the solvent that is given as 0 is a pure solvent, extracting solvent is pure, ok. And in the condition it is given that only 10 percent solute will be there in the raffinate.

So, we can say this one, Xc in the raffinate actually is we can say this Rn, that is, is equal to 0.1. That is why we here actually, this is, we can say this one Rn. Now, we have this, suppose for this we have feed point is 0.35 and S is here. So, we have this F, S and Rn. Now, say this suppose this if the first point is the we have to draw the equilibrium line. These are known, then we need to locate F, that is, we can say this one Xb is equal to 0 and Xc, this means that is equal to 0.35. X and Y-axis that F and then S will be like this, 0 and 1, because this is we can say this one Xc is 0 in the x-axis and (Yb) Yc, that is 1. So, this is purely pure solvent.

And Rn, that is we can say this one, Rn, that is nothing but say we can say, it would be somewhere else here. That is, some value maybe around nearby 0 and 0.1. So, that is why it is like this, it is lying in this, we can say this one, in the equilibrium line or raffinate (line) line, ok.

Now, we need to draw this, we can say F to S we need to draw a straight line and now we need to get the M point somewhere, M point is there so we need to get this one say Xm, like this one, Xm will be like this. F Xc F plus S Yc S divided by F plus S. So, it is like F is equal to 2000 into Xc F is equal to 0.35 plus S is equal to 2970 into 0, divided by 2000 plus 2970. So, it is coming out as, Xm is equal to 0.141. So, it is somewhere, it will be like this. Xm is equal to, this is Xm.

So, so whenever we have this M point and we have Rn then the third step will be like this, we need to say join Rn and M and extend up to extract arm. So then, if we join this M and then if we extend this one, we will be getting E1, ok. So, that is we will be getting this E1, ok. Now, if we draw this Rn and S and F, we can extend this one. Now, if we join this F, E1 and S join, extend this one, where it will meet, there we will be getting delta. That we have already discussed this one.

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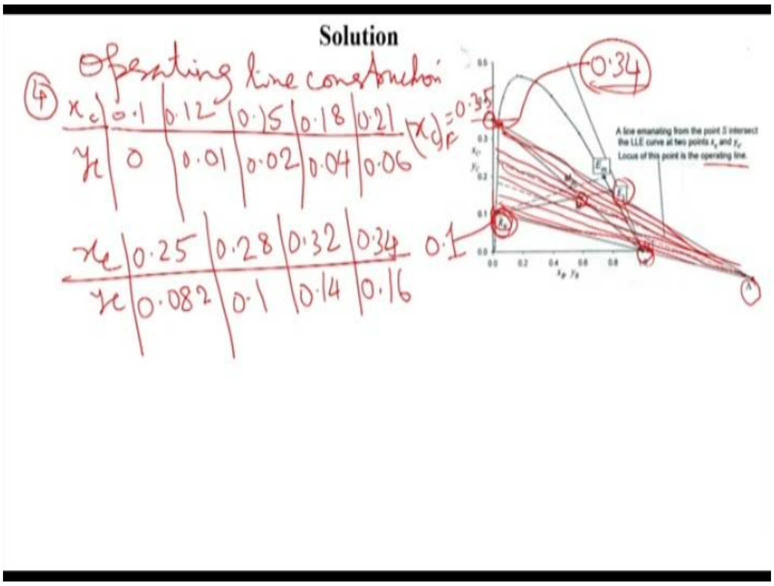

Solution

④ Operating line construction

x_c	0.1	0.12	0.15	0.18	0.21
y_c	0	0.01	0.02	0.04	0.06

$(x_c)_F = 0.35$

x_c	0.25	0.28	0.32	0.34
y_c	0.082	0.1	0.14	0.16

0.1

0.34

A line emanating from the point S intersect the LLE curve at two points x_c and y_c. Locus of this point is the operating line.

So, like this one, we will be getting all this. So, we can say like this one, M point is located like this one you see M is located and then Rn is located. F is located. S is located. Then, RnS extended to this, and F we can say E1 is extended to the so it now we have got this delta, ok.

Now, we need to do so this we can say this one, fourth step will be like this. We need to draw so many lines from this delta to this raffinate arm. Then, it will definitely cross this extract arm somewhere so, so many lines actually we need to draw [steath] straight lines. Because all these are we can say this one operating line in between this extract this one, what is called the last unit of the, we can say this one, counter-current extraction system where from where from this one n from nth we can say this one extraction unit, that is exiting and where this E1 means, where this, we can say this one, from the first unit we are taking the extract, ok.

So in-between the, we can say this one, all the, we can say this one, extraction units are located. From 1 to, this is, from 1 to Rn. So, we can say this one all these lines are corresponding to, we can say, operating line. So, we need to do one thing, we need to draw this operating line for this system. So, if these, say F is here, we can say this one, XcF is equal to, suppose 0.35. So, we can say this one, whenever the this point in the raffinate arm, that is, so that is why this value is around 0.34. And this is around 0.1. Because this is touching almost touching this Y-axis. Here, a small deviation is there from 0.3. That is why it is 0.34.

So, we can say this one that entire extraction units, all the extraction units are units are operating in between 0.1 to 0.34. So, we can get this one corresponding to x-value what will be the y-value. So from this all this see, we can say this one, we need to read this one whenever we will be drawing this one in any, this one, millimetre millimetre log paper. We will be getting all this value and say, we can try this one and we will be getting all this value. For the time being, I am writing this Xc and Yc values, for this operating line drawing. So, we can say this one, operating line construction.

So, for different Xc value, what will be the Yc? So, minimum Xc value we will start from this 0.1 because that will be the minimum value. So, for that whatever the Yc value for this one, Yc value is here you see if we see this one, Yc will be definitely leaving this one with 0. So, Yc will be 0 where we can say this one, where S point is there. Then, we can takes any point like this, like 0.12 then it will be 0.01. So, if we take 0.15, so it will be say 0.02. If it is 0.18, so it is 0.04. If it is 0.21, so it is 0.06. Then, Xc and Yc. If it is 0.25, the Yc value is 0.082, means if we read this one somewhere here it will be 0.25 like this one, corresponding to this. Yc value will be like here. That is 0.082, ok. 0.28 then it will be 0.1, ok. Then 0.32, it will be like 0.14. And the last point, that is, 0.34, so that is nothing but 0.16.

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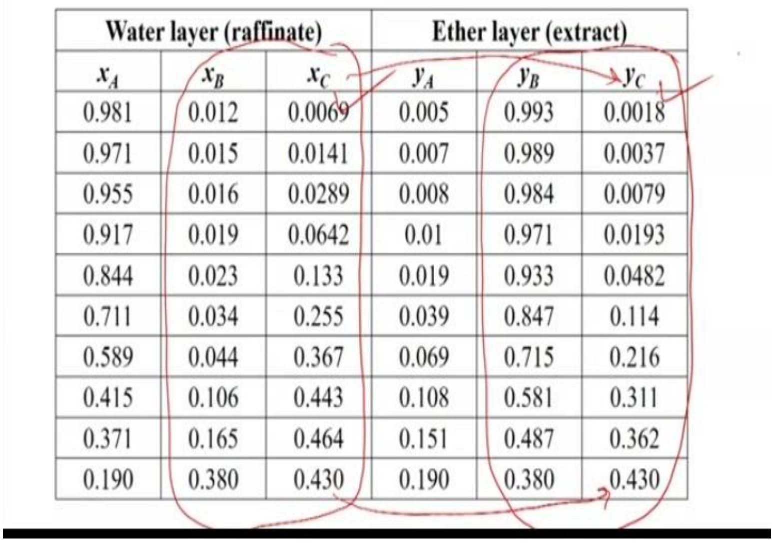

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So, now we have this operating line, so we will be constructing the operating line in the Xc-Yc graph, ok. And the equilibrium line that we can plot from here, for this Xc and Yc, we will be drawing the equilibrium curve.

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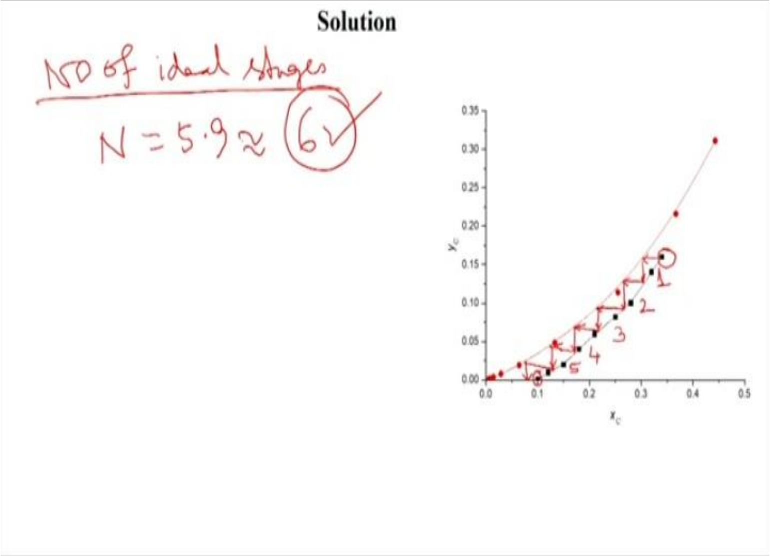

Solution

NO of ideal stages

N = 5.9 ≈ 6

So that will be like this we can say, it will be like this type. Say, what will happen? So this is the operating line. So this is, we can say, 0.34 and this is we can say this one 0.1. So in between this is operating so for this 0.34, this Yc value is 0.16. So this one

0.16, and like this and for 0.1, this Yc value is 0, ok. So, that is that is Rnp value. It is the E1 value, ok. And the equilibrium line actually is drawn from this given equilibrium data points, ok.

Now, we will be doing this one for getting this number of theoretical stages or we can say this one, number of ideal stages. So, what will be the number of ideal stages? Whatever we do for any, equilibrium govern separation processes. We will start from here, this we can say is on top, we can say this one, operating point, to this, we will be moving parallel to this and we will be coming vertically. So, this way. So, this is we can say this one will be stage 1.

Then, again we will be coming parallel to, we can say, is x-axis, then we will be coming vertically to this y-axis, it will be stage 2. Then again we will be coming parallel to x-axis and coming vertically to y-axis. This is the 3. Then again coming parallel to x-axis and coming vertically to y-axis, this is 4. Then we can say this one, we can follow this one. So, that is 5. And again, we can say this one we are coming. This is not the complete one, but you see this in in actual, it is coming at as 5.9. So, number of stages, we can say, this one. Number of stages is equal to, it is coming at 5.9, that is, we can say is equal to 6 number of stages.

So, the number of theoretical stages or ideal stages requirement for this type of, we can say this one, separation using this extraction process is we can say this one, 6 number of stages or 6 extraction units are required to get this one separation. So, means the final raffinate will contain 10 percent solute, ok.

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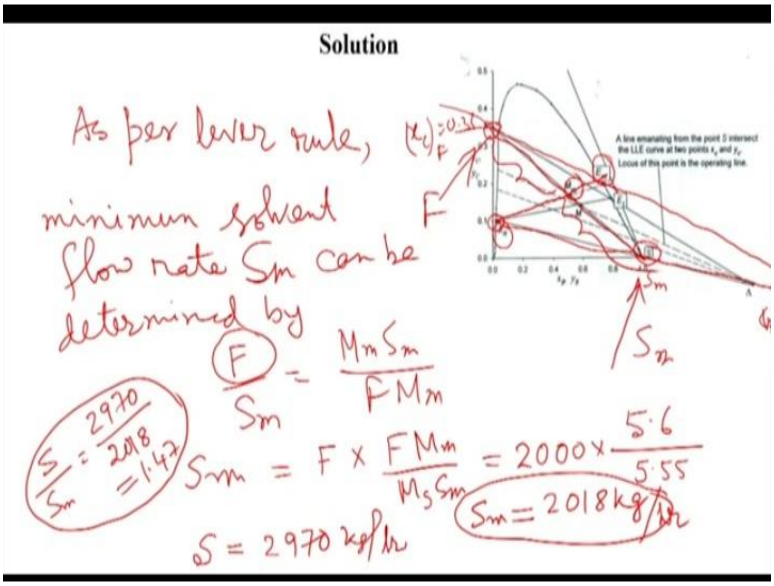

Solution

As per lever rule, minimum solvent flow rate S_m can be determined by

$$\frac{F}{S_m} = \frac{M_m S_m}{F M_m}$$

$$S_m = F \times \frac{F M_m}{M_s S_m} = 2000 \times \frac{5.6}{5.55}$$

$$\boxed{S_m = 2018\ kg/hr}$$

$$S = 2970\ kg/hr$$

$$\frac{S}{S_m} = \frac{2970}{2018} = 1.47$$

So, now we need to get the minimum solvent flow rate so that we have the already this one for this getting this minimum solvent flow rate, what we have done? Say, we will be doing this. We will be following this one to get this minimum solvent flow rate. What we discussed just now that say, we have this Rn. We have this feed point. We have this S. We have this Sm here also, ok. Now, we will be, what we will be doing say, nearby this 0.34 means this is feed point is 0.35, so we can say this one, Xc is equal to is equal to 0.35, ok.

So, this, whatever the nearby this operating line or we can say this one we will be getting this one, it will be like this. This one will be the nearby this operating line which will pass through F. So, that gives Em. That we already discussed this one then we will be extending this one to this and whatever Rn and S or Sm, both are same, is there, that we will extend, then it will be touching one particular point where we will be getting this delta m, ok.

So, that actually we have got. Once we have got this Em and we have this Rn then we need to draw a, say that F and Sm, these are joined, then we need to join this Rn and m. Ok, then we will be getting this Mm. So, this will be now, we can say this one, so solute concentration in the mixture where minimum amount of solvent is required, ok. So, in that case, as per this lever rule, we can say this one. As per lever rule this minimum solvent flow rate, as per lever rule, minimum solvent flow rate that is, Sm, can be determined by F by Sm is equal to, F by Sm is equal to MmSm. This is F. This is Sm. So, whenever F is there, then we will be taking this MmSm divided by FMm.

So, if we measure these values, so we have this feed flow rate we have. Sm we need to get. So, we can say this one Sm is equal to, Sm is equal to F into FMm by MmSm. So, F is say 2000 and say, FMm, that is actually obtained as, say, unit wise, that is 5.6. And MmSm is obtained as 5.55. So, we can easily measure this one just by using this scale we will be getting that what is the length of this one. Whatever unit is there, it is immaterial because we will be getting this one as the ratio. So, that is why it will be coming out as 2018 Kg.

So, you can see this Sm, or you can say, minimum solvent rate actually is coming as 2018 Kg per hour, ok. So, we can say this one the actual flow rate will be like this so solvent flow rate is given as 2970 Kg per hour. So, now we are getting this Sm, so we can say this one, that ratio between S by Sm, so will be like this. So, S by Sm will be like this. S by Sm will be 2970 divided by 2018 that is coming out as 1.47. So this, we can say this one, the actual flow rate is 1.47 times that of the minimum solvent flow rate, ok.

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References:

1. R. E. Treybal “Mass Transfer Operations”, McGraw –Hill International Edition, 3rd Ed., 1981.

2. W. L. McCabe, J. Smith and P. Harriot, "Unit Operations of Chemical Engineering", 6th Ed., McGraw – Hill International Edition, 2001.

3. B.K. Dutta, Principles of Mass transfer and separation processes, 1st Ed., PHI, 2012.

Thank you.

In the next class we will be discussing about the solid-liquid extraction or leaching process.