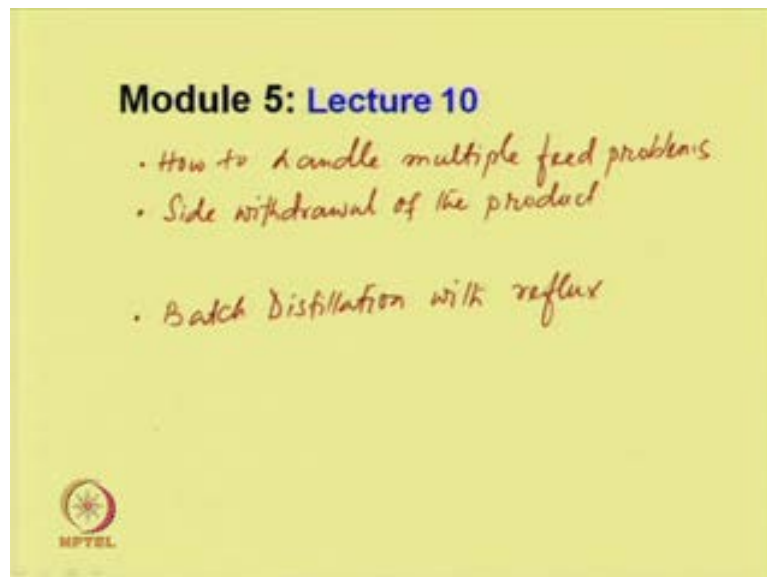


Mass Transfer Operations I
Prof. Bishnupada Mandal
Department of Chemical Engineering
Indian Institute of Technology, Guwahati

Module - 5
Distillation
Lecture - 10
Multistage Batch Distillation with Reflux

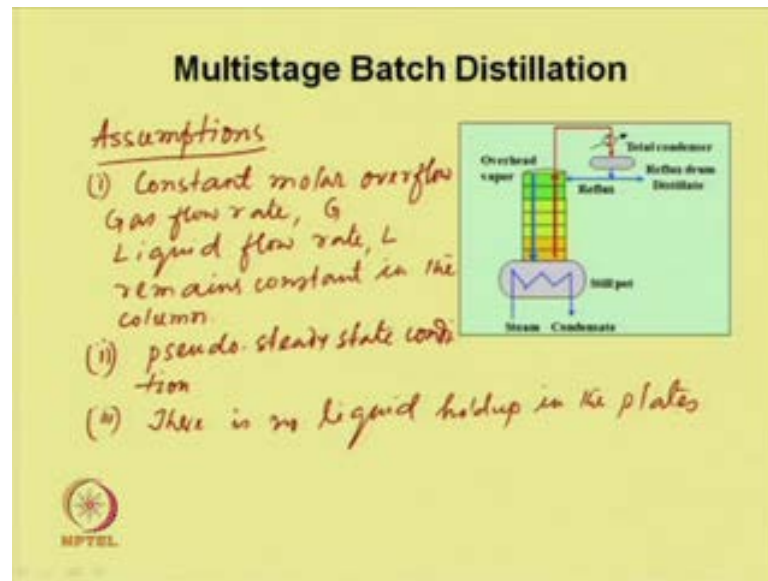
Welcome to tenth lecture of distillation; this is module 5, and we are discussing distillation.

(Refer Slide Time: 00:36)



In our previous lecture, we have discussed how to handle multiple feed problems and also we have discussed side withdrawal of the product, side withdrawal of the product. Particularly, we have discussed if there are multiple feeds then what will be the material balance for a particular problem, and how to calculate the number of ideal stage required for a given separation, also to locate the different patria locations in the column. Similarly, if there is a side withdrawal of the product including the override distillate withdrawal then how the material balance will change, and what would be the operating equations and then how to find out the locations of the plate from where the product is withdrawn. In this lecture, we will mainly focus on another important topic that is batch distillation with reflux.

(Refer Slide Time: 02:40)



So, let us see a schematic of the batch distillation with reflux. This is a still pot, where the feed is taken over here and steam is injected and which is through a coil and taken as condensed state at the outlet, so that the energy is transfer to the feet to boiler. So that, the vapor forms and it goes off from tray to tray, and then there is similar to the fractionators, there is a top total condenser then reflux drum and then we have a distillate out and the reflux back to the column. We can chose how many stages we require for a given separations. As we can see the purity of the product is governed by the concentration of the feet and the fractions distilled out, which is taken out. Whatever we want the product purity can be achieved if we use the multi stage and we can get more pure products.

Now, in this case we will assume the similar to fractionators that is constant molar overflow; that means, the gas flow rate and liquid flow rate remains constant in the column. And second thing, we will assume pseudo steady state conditions maintained, pseudo steady state condition in general the batch distillation is a unsteady state process, but we will assume pseudo steady state condition. And third there is no liquid holdup in the plate, there is no liquid holdup in the plates. So based on these three primary assumptions, we will do the theoretical analysis of the multistage batch distillation problem with reflux.

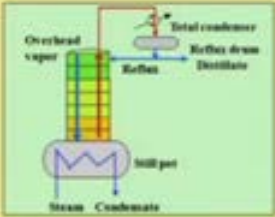
(Refer Slide Time: 06:20)


Multistage Batch Distillation

(i) Reflux ratio constant
→ conc. of the distillate will decrease as the distillation proceeds.

(ii) $x_D = \text{constant}$
Variable reflux ratio.

Rayleigh eqⁿ \Rightarrow iteration is required.





Now, this multistage batch distillation can be operated with two types. One is we can keep the reflux rate constant or reflux ratio constant, reflux ratio constant that is case one. So what will happen in this case, the concentration of the distillate will decrease as the distillation proceeds. And second we can keep the overhead distillate concentration constant that is x_D constant, x_D constant the reflux ratio will change, so variable reflux. So, the first one is known as the constant reflux operations, and the second one is known as the variable reflux operation. If we want to solve using the Rayleigh equations as we discussed before, then some iteration is required, and if you use the McCabe Thiele method of diagram, and we can obtain some of the information, which will be used for this iteration.

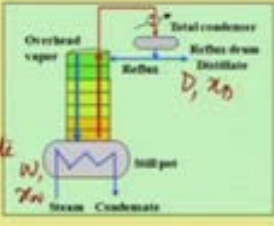
(Refer Slide Time: 08:37)

Constant Reflux

W mole of the feed in the still pot.
 Conc. of the feed = x_w
 Over a very small time dt , dD mole of distillate is withdrawn, dW mole will be removed in the still pot.

$$-d(Wx_w) = x_D dD$$

$$\Rightarrow -W dx_w - x_w dW = x_D dD, dD = -\frac{dW}{x_D - x_w}$$

$$\Rightarrow \frac{dW}{W} = \frac{dx_w}{x_D - x_w}$$


Now let us consider the first case which is constant reflux operation. We will discuss constant reflux operations. We have taken say W mole of the feed in the still pot, and its concentration of the feed is x_w . Assume very small time free out over a very small time dt – dt , dD is the distillate and this is the W and its x_D and its composition is x_w .

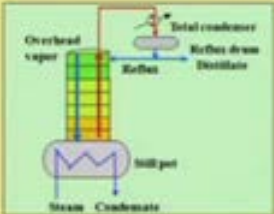
So, if dD mole of distillate is withdrawn, so the amount in the still pot which will also decrease by an amount dW mole will be decreased in the still pot. So, if we do the balance minus $dW x_w$ would be equal to $x_D dD$ from where we can write minus $W d x_w$ minus $x_w dW$ would be equal to $x_D dD$ and we know that dD is equal to minus $dW / (x_D - x_w)$. And we know that dD would be equal to minus dW . So, we can substitute this one over here and which would be dW / W would be $dx_w / (x_D - x_w)$.

(Refer Slide Time: 11:51)

Constant Reflux

$$\ln \frac{W_i}{W_f} = \int_{x_{wf}}^{x_{wi}} \frac{dx_w}{(x_D - x_w)}$$

change
→ graphical integration
 W_i, W_f, x_{wi}, x_{wf}

$$x_{D,avg} = \frac{W_i x_{wi} - W_f x_{wf}}{W_i - W_f}$$


MPTEL

If we integrate over the concentration range from the initial to the final, we would be able to get $\ln W_i$ by W_f is the final concentration would be equal to integral $x W_f x W_i$ $d x_w$ divided by x_D minus x_w . So, as the distillation would occur this x_D and x_w this will change. So, the integral of this can be evaluated graphically. So, graphical integration can be done to evaluate this integral, this we will discuss in detail with an example. In this, we have four terms W_i , W_f , x_{wi} , and x_{wf} ; any three of this if known to us then we could be able to calculate the others. And the concentration of the distillate can be obtained from the following expression $x_{D,avg}$, we could be able to obtain $W_i x_{wi} - W_f x_{wf}$ divided by $W_i - W_f$.

(Refer Slide Time: 13:57)

Example

A 100 kmol mixture of 40% A and 60% B is subjected to batch distillation column at 1 atm total pressure. The column has one tray, a still pot and a total condenser. The final concentration of residue is 5 mol% A. The reflux is a saturated liquid and a reflux ratio of 1.6 is used. Calculate the composition and amount of distillate. The equilibrium data is given below:

x	0	0.05	0.10	0.20	0.40	0.7	1
y	0	0.25	0.40	0.60	0.80	0.82	1



Let us consider an example, how to solve this problem. A 100 kilo mole mixture of 40 percent A and 60 percent B is subjected to batch distillation column at one atmosphere total pressure. The column has only one tray, or still pot and a total condenser. The final concentration of the residue is 5 mole percent A. The reflux is a saturated liquid and a reflux ratio of 1.6 is used. Calculate the composition and amount of distillate. The equilibrium data is given over here. So, the reflux ratio which is fixed at 1.6 and equilibrium data is given. So, we could able to plot the equilibrium curve. Since the concentration of the vapor from top tray will gradually decrease.

(Refer Slide Time: 15:02)

Solution

A 100 kmol mixture of 40% A and 60% B is subjected to batch distillation column at 1 atm total pressure. The column has one tray, a still pot and a total condenser. The final concentration of residue is 5 mol% A. The reflux is a saturated liquid and a reflux ratio of 1.6 is used. Calculate the composition and amount of distillate. The equilibrium data is given below:

x	0	0.05	0.10	0.20	0.40	0.7	1
y	0	0.25	0.40	0.60	0.80	0.82	1

- (i) Select numbers of x_D values on the diagonal and draw operating lines. All operating lines are parallel.
- (ii) For each x_D value, obtain x_W by drawing two stages. Set of (x_D, x_W) in the range $x_{Wi} = 0.4$, $x_{Wf}(\text{final}) = 0.05$.



The procedural of solving this problem will be like this. Select number of x_D values on the diagonal and draw operating lines. Since, the reflux ratio remains constant that is constant reflux operations all the operating line will be parallel because their slope is constant which is r by r plus 1. All operating lines are parallel. Second, for each x_D values obtain x_W by drawing two stages, two stages means we have one tray and one still pot. So, we will consider still pot as one tray, as we considered in case of the fractionators, the re-boiler as one stage, so it will be one equilibrium stage.

In this case, we will draw two stages and we will obtain the values of x_W corresponding to a particular value of x_D . We will have a set of x_D , x_W values. This will be in the range x_{W_i} would be which is initial that is 40 percent – 0.4, and the final x_{W_f} which is final would be 0.05. So, this is the final and this is the initial the mole fractions in the liquid phase. So, within this we would get a set of x_D and x_W .

(Refer Slide Time: 18:00)


Solution

A 100 kmol mixture of 40% A and 60% B is subjected to batch distillation column at 1 atm total pressure. The column has one tray, a still pot and a total condenser. The final concentration of residue is 5 mol% A. The reflux is a saturated liquid and a reflux ratio of 1.6 is used. Calculate the composition and amount of distillate. The equilibrium data is given below:

x	0	0.05	0.10	0.20	0.40	0.7	1
y	0	0.25	0.40	0.60	0.80	0.82	1

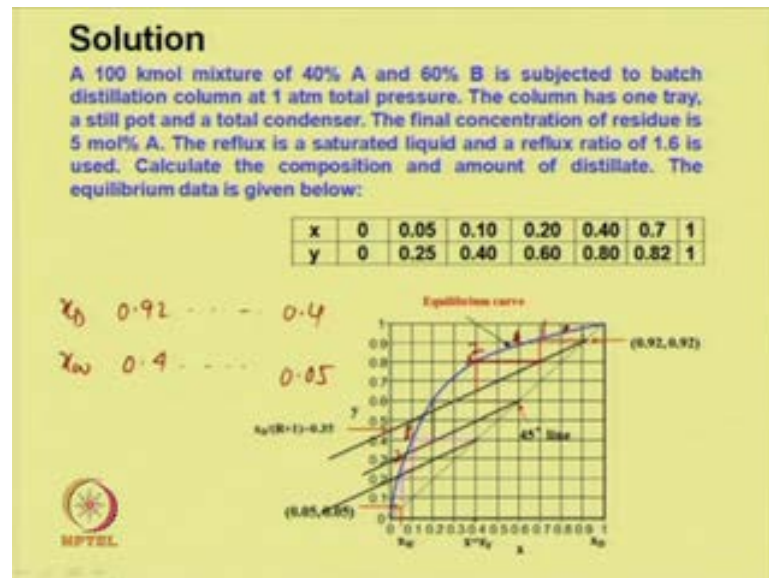
$$\ln \frac{W_i}{W_f} = \int_{x_{W_f}}^{x_{W_i}} \frac{dx_W}{x_D - x_W}$$

Reflux ratio = 1.6

$$\therefore \text{slope} = \frac{R}{R+1} = \frac{1.6}{1.6+1} = 0.615$$


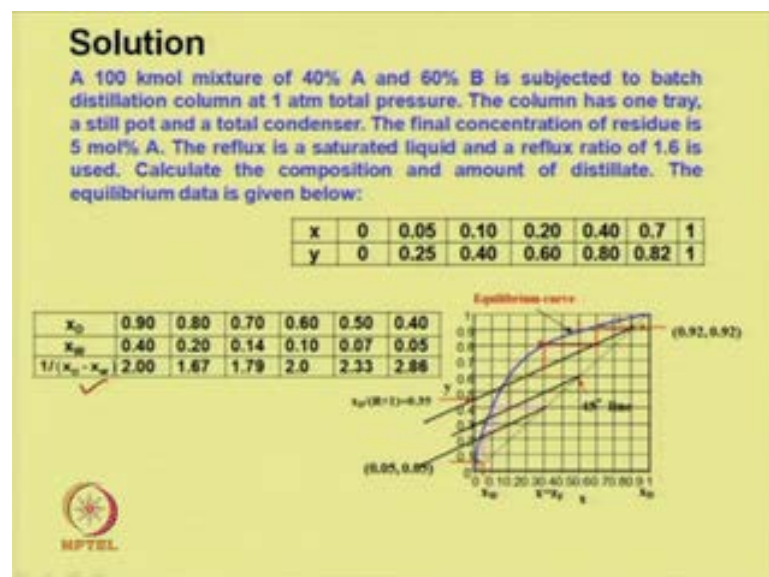
And then the integral equations, we know $\ln W_i$ by W_f would be equal to W_f to W_i x_{W_f} to x_{W_i} dx_W divided by x_D minus x_W . And the reflux ratio is given 1.6, therefore slope would be equal to R by R plus 1 which is equal to 1.6 divided by 1.6 plus 1 which is equal to 0.615.

(Refer Slide Time: 18:59)



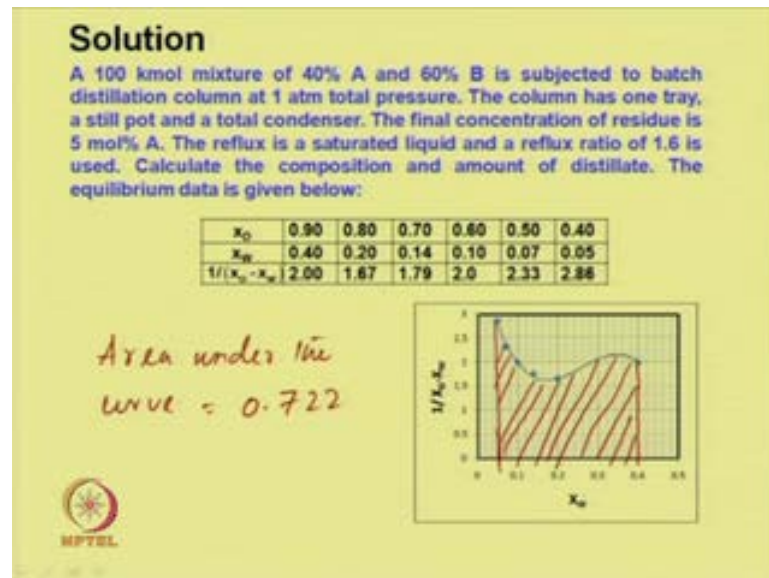
Now, from the equilibrium data we can draw the equilibrium line assume. This is the initial x_D values is 0.92, two trays we can draw and there is composition is giving 0.4. So for this 0.92 and we could obtain that is x_D and x_W we could obtain 0.4. Similarly, at the end, we can see if we start at 0.4, so if we plot two number of stages one and two. So, you could obtain 0.05, so we could obtain 0.4 and 0.05. So, this is x_D and x_W data.

(Refer Slide Time: 19:53)



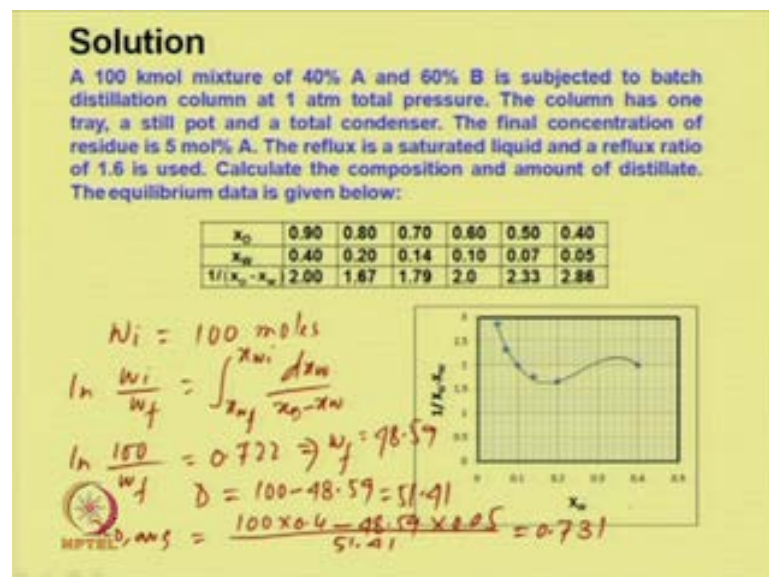
Drawing several similar parallel lines we have obtained x_D and x_W values, and from which we have calculated $1/(x_D - x_W)$ these are the values of $1/(x_D - x_W)$. Now, we can plot $1/(x_D - x_W)$ versus x_W to get area under the curve.

(Refer Slide Time: 20:18)



So, which is plotted over here and the area between this, so area under this curve, this is by any method we can obtain the area 0.722. So, area under the curve we can obtain.

(Refer Slide Time: 21:10)



And then we know the initial amount of moles taken. So, W_i which is 100 mole and we know $\ln W_i$ by W_f is equal to integral x_{Wf} to x_{Wi} of $dx_W / (x_D - x_W)$. So, here we

can substitute $1 - 0.05$ by w_f is equal to 0.722 and from here we can calculate W_f is 48.59. Then we can obtain D using the total material balance equations. So, it would be 100 minus 48.59 which is equal to 51.41. And average concentration x_D average which would be 100 into 0.4 minus 48.59 multiplied by 0.05 divided by 51.41 which would be around 0.731; that means 73.1 percent is the distillate compositions.

(Refer Slide Time: 23:11)

Variable Reflux

x_D constant

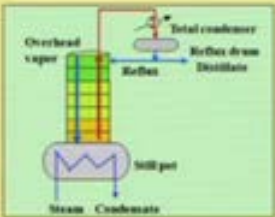
dG = change in vapor flow
 dL = " " liquid
 dD = " " distillate

$dG = dL + dD \rightarrow (1)$

$\Rightarrow dD = \left(1 - \frac{dL}{dG}\right) dG \rightarrow (2)$

$W x_n = W_i x_{wi} - (N_i - N) x_D$

$\Rightarrow W = W_i \left(\frac{x_{wi} - x_D}{x_w - x_D} \right) \rightarrow (3)$



Now, we will consider variable reflux problem, where we are keeping constant overhead concentrations, so x_D constant. In this case, the reflux ratio would be changed continuously whereas keeping the x_D constant, so the concentrations of the distillate will not fall any time; however, the reflux ratio will change continuously. Now say dG is the change in vapor flow, and dL change in liquid flow, and dD change in distillate withdrawal. If we do the total material balance, we can write dG would be dL plus dD . Just rearrange these equations, we can write dD would be equal to 1 minus dL by dG into dG . Now if we do the sepsis mole balance, we can write $W x_w$ would be $W_i x_{wi}$ minus W_i minus W into x_D . So, from here we can write W is equal to $W_i x_{wi}$ minus x_D divided by x_w minus x_D .

(Refer Slide Time: 25:29)

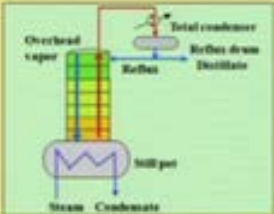
Variable Reflux

Differentiate eqn (2)

$$dW = \frac{W_i (x_{wi} - x_D) dx_w}{(x_D - x_w)^2}$$

$$dD = -dW$$

$$G = \int dG = \int_{x_{wi}}^{x_{wf}} \frac{W_i (x_{wi} - x_D) dx_w}{(x_D - x_w)^2 \left[1 - \frac{dL}{dG}\right]}$$

$$= W_i (x_D - x_{wi}) \int_{x_{wi}}^{x_{wf}} \frac{dx_w}{(x_D - x_w)^2 \left(1 - \frac{dL}{dG}\right)}$$


Now, if we differentiate this equations, equation three we will obtain differentiate, we can obtain dW would be equal to $W_i \times x_{wi} \text{ minus } x_D \times dx_w$ divided by $x_D \text{ minus } x_w$ square. We know that dD would be equal to minus dW . So, if you substitute this equations in equation two, this one we could obtain dG is equal to $W_i \times x_{wi} \text{ minus } x_D \times dx_w$ divided by $x_D \text{ minus } x_w$ square into $1 \text{ minus } dL \text{ by } dG$. If we want to calculate the total gas flow rate or amount generated, we can write is G integral x_{wi} to x_{wf} it will be W_i into $x_D \text{ minus } x_{wi}$ into integral x_{wi} to x_{wf} dx_w divided by $x_D \text{ minus } x_w$ square into $1 \text{ minus } dL \text{ by } dG$.

Now, area under the curve, we can obtain if we know the values of x_D and x_w as well as the slope, and then we can calculate the slope of the lines then we can calculate the values of g . Integral can be obtain graphically like we did before and then we can calculate the vapor which is generated.

(Refer Slide Time: 28:07)

Solution

A 100 kmol mixture of 40% A and 60% B is subjected to batch distillation column at 1 atm total pressure. The column has three trays, a still pot and a total condenser. The top product contains 96% A. The final concentration of residue is 4% A. The reflux is a saturated liquid.

(a) Calculate the amount of top and bottom products.
 (b) What initial and final reflux ratios should be used?
 (c) Determine the total moles of vapor to be generated to achieve the separation.

The equilibrium data is given below:

x	0	0.05	0.10	0.20	0.40	0.7	1
y	0	0.25	0.40	0.60	0.80	0.82	1

$F = D + W \Rightarrow 100 = D + W$
 $Fz_F = Dx_D + Wx_W \Rightarrow 100 \times 0.4 = D \times 0.96 + W \times 0.04$
 $\Rightarrow 40 = 0.96D + 0.04W$
 $\Rightarrow 40 = 0.96D + 0.04(100 - D) = 0.96D + 4 - 0.04D$
 $\Rightarrow 36 = 0.92D$
 $D = 36 / 0.92 = 39.13, W = 100 - 39.13 = 60.87$

Let us take an example a 100 kilo mole mixture of 40 percent A and 60 percent B is subjected to batch distillation column at one atmosphere total pressure. The column has three trays, a still pot and a total condenser. The top product which contains 96 percent A which is fixed that is x_D is fixed at 0.96. The final concentration of the residue is percent A. The reflux is saturated liquid calculate the amount of top and bottom top of to be generated to achieve the separation the equilibrium data are given below.

So, this is the equilibrium data which is given let us do the total material balance that is F is D plus W which is 100 would be D plus W component balance if we do Fz_F would be Dx_D plus Wx_W . So, you can write 100 into 0.4 would be equal to D into 0.96 plus w into 0.04 then we can write 40 would be equal to 0.96 D plus 0.04 W .

Now, we can substitute D from here and we can write 40 would be equal to 0.96 D plus if we substitute W from here. So, it will be 0.04 into 100 minus D . So, this would be equal to 0.96 D plus 4 minus 0.04 D . So, d would be 36 divided by 0.92 and which is equal to 39.13. So, W also we can calculate W would be 100 minus 39.13 which is equal to 60.87.


(Refer Slide Time: 30:51)

Solution

A 100 kmol mixture of 40% A and 60% B is subjected to batch distillation column at 1 atm total pressure. The column has three trays, a still pot and a total condenser. The top product contains 96% A. The final concentration of residue is 4% A. The reflux is a saturated liquid.

(a) Calculate the amount of top and bottom products.
(b) What initial and final reflux ratios should be used?
(c) Determine the total moles of vapor to be generated to achieve the separation.

(i) Draw the eqn curve (data given)
(ii) Locate point $(x_D = 0.96, x_D = 0.96)$
(iii) Draw multiple operating line from the point $(x_D = 0.96, x_D = 0.96)$ with different slopes.



Now, draw the equilibrium line the data is given. Second will locate point x D which is 0.96. From this since the x D is constant the slop of the particular line will change. So, we will draw from starting from this point, we will draw different operating lines, draw multiple operating line from the point x D is equal to 0.96 x D is equal to 0.96.


(Refer Slide Time: 32:22)

Solution

A 100 kmol mixture of 40% A and 60% B is subjected to batch distillation column at 1 atm total pressure. The column has three trays, a still pot and a total condenser. The top product contains 96% A. The final concentration of residue is 4% A. The reflux is a saturated liquid.

(a) Calculate the amount of top and bottom products.
(b) What initial and final reflux ratios should be used?
(c) Determine the total moles of vapor to be generated to achieve the separation.

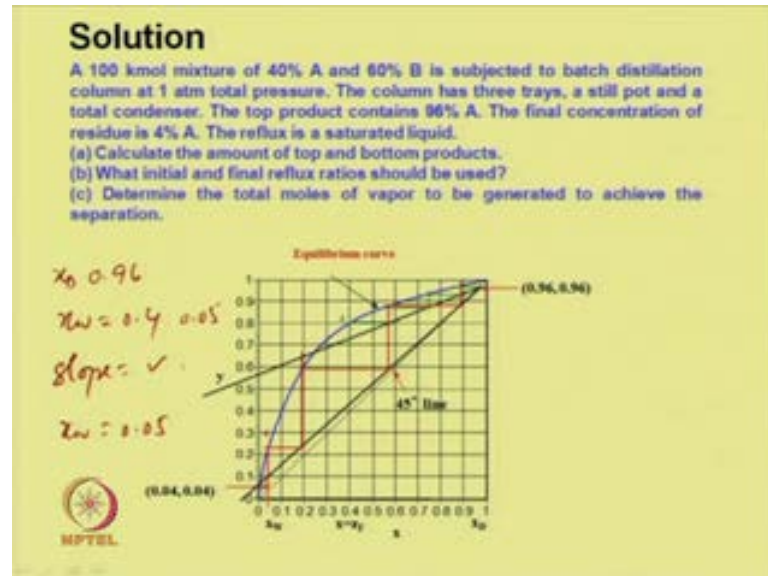
(i) In each case, draw four stages betⁿ eqn line and the operating line. Calculate x_n
(ii) Set of (x_D, x_n) ; slopes of the operating line.



So, from this point draw multiple operating lines with different slopes, and then what we have to do in each case each case draw four stages between equilibrium line and the

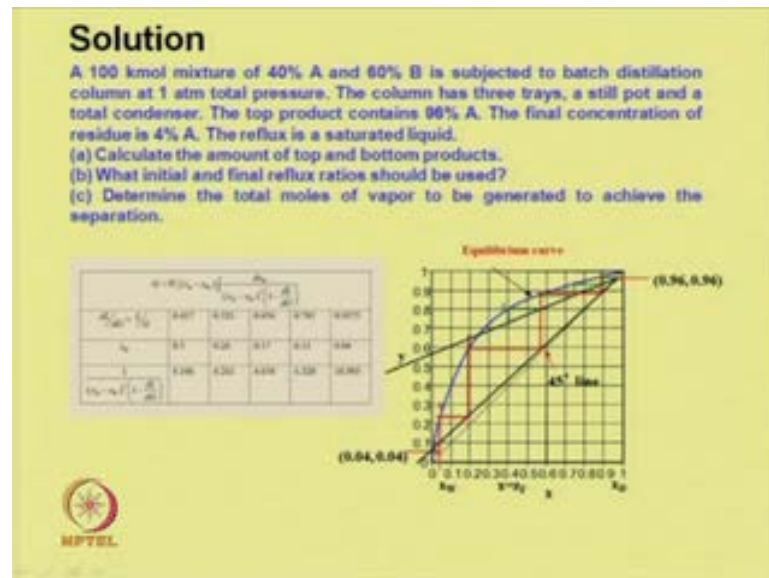
operating line. And then calculate calculate x_w then we will have set of x_D and x_w . Since, we have x_D and x_w and also we have slopes of the operating line.

(Refer Slide Time: 33:29)



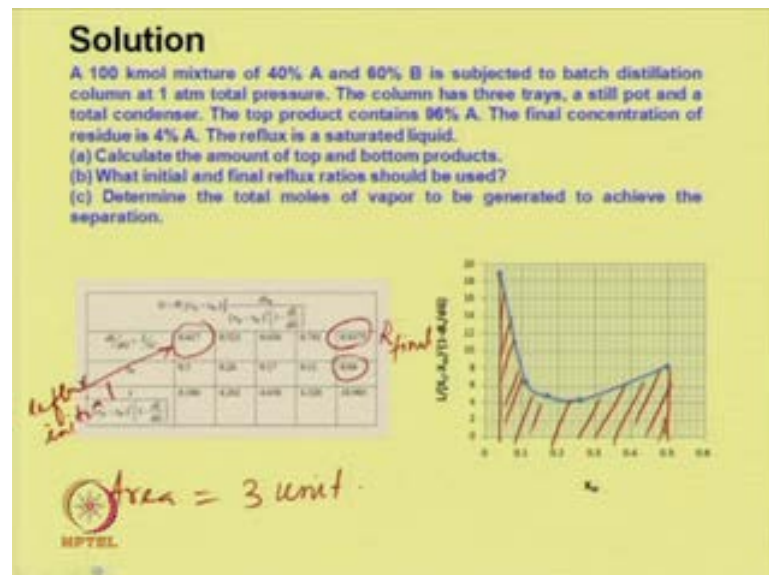
There are two operating line which is shown over here we start at 0.96. And we draw the four stages and the point which is coming 0.96, and we can obtain x_D and we could obtain x_w with four stages each 0.4. And we can calculate the slope of this operating line slope, also we can calculate for this which is over here. So, slope is known. Similarly, another operating line which is over here x_w is 0.05 and slope we can calculate from this operating line. So, that we have a set of x_w and we have a slope for each case.

(Refer Slide Time: 34:20)



Let us see the table. The first one has slope of 0.417 \times w is 0.5, so we could able to calculate this one. Similarly, for the second we have operated 0.521. So, we have a set of data with these we can plot this $1 \text{ by } x \text{ D minus } x \text{ w square into } 1 \text{ minus } d \text{ l by } d \text{ g verses } x \text{ w}$. So, we can calculate the area under the curve for this integral.

(Refer Slide Time: 34:50)



So, the area under the curve for this integral is this, so this is the area is around 3. Now, area is known to us, then the initial reflux which could be used calculate the amount of top and bottom products we have already calculated. What initial and final reflux ratio


should be used, the initial and final reflux ratio which is given over here initial is corresponding to 0.5, it is 0.417; and final corresponding to 0.04, it is 0.39. So, it is reflux ratio final, and this is reflux ratio initial. The area under the curve is 3 units.

(Refer Slide Time: 36:03)

Solution

A 100 kmol mixture of 40% A and 60% B is subjected to batch distillation column at 1 atm total pressure. The column has three trays, a still pot and a total condenser. The top product contains 96% A. The final concentration of residue is 4% A. The reflux is a saturated liquid.

(a) Calculate the amount of top and bottom products.
 (b) What initial and final reflux ratios should be used?
 (c) Determine the total moles of vapor to be generated to achieve the separation.

$$\begin{aligned}
 G &= W_i (x_D - z_f) \times \text{Area} \\
 &= 100 \times (0.96 - 0.04) \times 3 \\
 &= 300 \times 0.56 \\
 &= \underline{168}
 \end{aligned}$$


So, we can calculate G which is equals to $W_i \times D$ minus Z_f into area under the curve, so which is equal to 100 multiplied by 0.96 minus 0.04 into is 3 unit. So, it would be equal to 300 multiplied by 0.56 which is equal to 168. So, this way we can determine the total moles of the vapor which is generated to achieve this separation, and the initial and final reflux ratio and the amount of top and bottom products.

Thank you.