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

Module - 5
Distillation
Lecture - 5
Fractional Distillation

Welcome to the fifth lecture of module 5, and in module 5, we are discussing distillation. In our previous lecture we have discussed two topics.

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Module 5: Lecture 5

(1) Batch distillation

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(2) Steam distillation

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One is batch distillation, and which has important application in fine chemicals and specialty chemicals production, and this is used in small capacity plants. Batch distillation as, we said had two different operations; one is with reflux and without reflux, we have considered only without reflux. The second thing we have covered is the steam distillation, and this is particularly useful for heat sensitive materials high boiling materials.

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## Fractional Distillation

- Fractional distillation normally used to separate liquid mixtures into two or more vapor or liquid products with different compositions.
- Equilibrium stage operation--- in each stage vapor phase is contacted with a liquid phase.
- Less volatile components --- concentrate in the liquid phase
- More volatile components---concentrate in the vapor.
- Using multiple stages in series with recycle, separation can be accomplished.



So today, we will discuss fractional distillation. So, it is normally used to separate liquid mixture into two or more vapor components or liquid products with different compositions. This operation is an equilibrium stage operation; that means, in each stage the vapor phase is contacted with a liquid phase and the vapor and liquid they come in equilibrium, what happens this less volatile components is concentrate in the liquid phase, and more volatile component it concentrate in the vapor phase. So, if we use multiple stages, where the vapor and liquid come in contact, then the materials transfer from one phase to the other; either from vapor to the liquid, or from liquid to the vapor. So, using the multiple stages in series and with recycle or reflux the separation can be achieved at any degree required.

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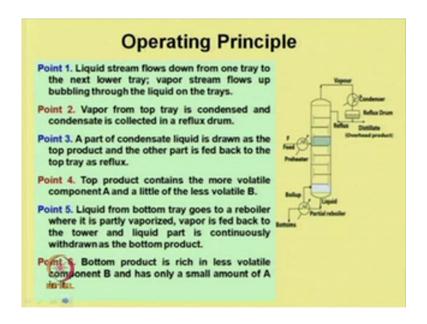
### Fractional Distillation

- Feed Condition: may be liquid, vapor, or a liquid-vapor mixture.
- · Feed Entry: may enter at any point in the column
- Multiple Feed and Product cuts: more than one stream may be fed to the system, and more than one product may be drawn.
- Trays: columns built from a set of distinct "trays" or "plates".
- Trays numbering: may be numbered from top down or bottom up. Flows and compositions take the number of the tray they leave. The top tray of the column "Tray 1" and numbers downward - - this is the convention.

So, the feed which we use has certain conditions; it may be liquid feed, it may be vapor feed, or it may be a mixture of vapor and liquid, and then feed which we fed into the tower or in the distillation column may enter at any point in the column. So, we can use multiple feed and we can take out multiple products. So, more than one stream may be fed into the system, and more than one product card may be withdrawn from the system or may be taken from the system.

So, for multistage system there are stages or tray are numbered and each column is made off with a set of distinct trays, or we call plates. This tray numbering we have to follow either from the top of the tower and then go down to the tower, or we can number from the bottom of the tower to the top of the tower. The top tray of the column is called tray one if we numbered from top to bottom and then we number downward and this convention we usually follow.

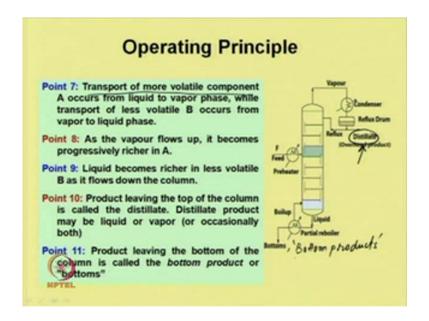
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Now, we will discuss how we can operate fractional distillations. So, this is the diagram of a typical distillation fractionators; where liquid stream flows from top of the tower to the downwards from one tray to the other, and then vapor flows from bottom to top which bubble through the liquid. Vapor from top tray, which is coming at the top is condense in this condenser and then it is collected in a drum which is known as reflux drum. It part of the condensate is taken and fed at the top of the tower is known as the reflux .The top product which is coming out contains the more volatile components which is A, and we can say A, and little amount of less volatile component.

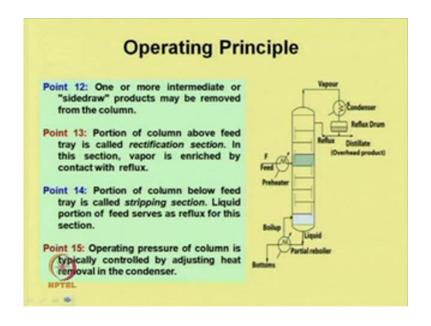
The liquid from the bottom goes to a re boiler where it is partially vaporized, generally it is used as partial re boiler, it is partially vaporized and vapor is paid to the tower, and then some bottom product is continuously withdrawn. Bottom product is reach in less volatile component and has very small fractions of more volatile component.

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Now, the transport of more volatile component A occurs from liquid to the vapor phase, while the transport of the less volatile B occurs from vapor to the liquid phase. As vapor goes of it becomes richer in A, and as the liquid flows down the column it reaches in component B. So the product which is leaving at the top tray is known as the distillate. The product at the top tray is known as the distillate or overhead product and this product may be liquid or vapor, or in some times it may be both. The product which is leaving at the bottom is known as the bottom products or bottoms.

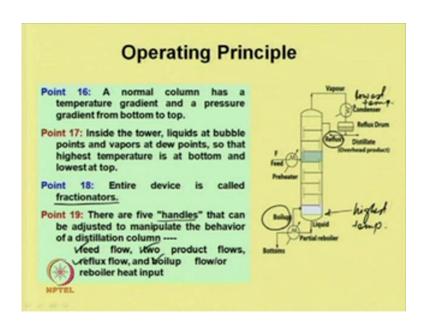
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Then one or more intermediates side draw we can take depending on our requirements. So, we can have different product cards, side card from the tower. Portion of the column above the feed tray, suppose this is the feed tray, so the portions above these is known as the rectification section, and in this section the vapor is enriched by contact with the reflux. So, reflux is coming and then vapor is going out in the rectifying section, the vapor is enriched with the contact with the reflux liquid.

The portions which is below of the feed is known as the stripping section, and in this case the liquid serves as the reflux for this section. The operating pressure of the column is typically we can control by adjusting heat removal in the condenser. So, the operating pressure in the system, we can obtain by removal of the heat using the condenser.

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So, in the column in normal operations it has a temperature and pressure gradient, the temperature and pressure gradient throughout the column from bottom to top; inside the tower, liquids at its bubble points, and vapor at its dew points. So, the highest temperature is at the bottom, and the lowest temperature is at the top. As we said the distillate is richer in the less volatile component, and all the liquids inside the column is at their bubble points, and the vapor which is there is at their dew points. So, the temperature at top of the tower is low compared to the bottom of the tower.

So, this whole device is known as the fractionators, the entire device is known as fractionators. So, there are five handles that can be adjusted to manipulate the behavior

of a distillation column. What are those; feed flow, we can change the feed flow; feed flow rate, we change the feed conditions as well, two product flows that means bottoms; and the distillate reflux flow, the reflux ratio we can change; and boiler flow or the reboiler heat input. So, this boil of which is coming out from the reboiler is known as the boiler, so this flow we can control. So, these are the five things which we can control to manipulate the behavior of a distillation column.

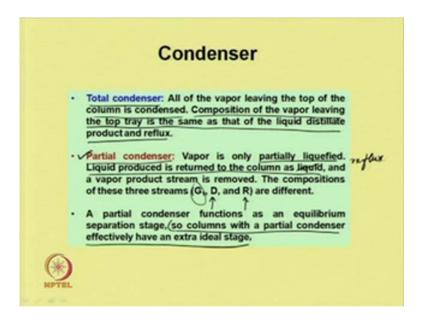
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Now ideal stage; what is ideal stage, if the vapor and liquid leave a particular stage are in equilibrium then we call it equilibrium stage or ideal stage. A key parameters or important results for ideal stage assumption is that, the liquid streams leaving in a ideal stage are assumed to be at their bubble point and vapor streams leave at their dew point. This is the important assumptions for if we assume a stage is in ideal.

The top and bottom products may be obtained in desired purity, if enough stages are provided and enough reflux is available, but there are two limits, that is, the number of stages and the amount of reflux, there is a limit. We cannot vary this indefinitely the number of or infinite number of stages and the total reflux. So, these are the two limits and one is called theoretical limits on performance, these are the total reflux and minimum reflux. The number of stages is fixed by these two parameters; one is called total reflux, and minimum reflux. We will consider this later when we will discuss this two limits.

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Now, the condenser which we use for the distillation column, they are of two kinds; one is total condenser, and another is partial condenser. So, total condenser where all of the vapor leaving the top of the column is condensed is known as the total condenser. The composition of the vapor which is leaving the top tray is the same as that of the liquid distillate product and the reflux.

So, if we look back into our column, so if we use the condenser as a total condenser, and then all the vapors this condense over here, so the composition of the distillate which is coming out, or the product card which is taken, and the reflux which is going back to the tower, or fed back to the tower will have the same composition. Another type of condenser which we use is the partial condenser; here vapor is partially liquefied, the liquid product is returned to the column as liquid. Liquid is returned to the product as reflux, and a vapor this is written as reflux and a vapor product steam is taken as a product, or over heat cards.

The compositions of these three streams, that is, which is coming at the top the vapor phase and then the distillate which is R is the reflux or recycle, the three cases the composition is different. So, the vapor which is coming out say G1, and the reflux which is given is R, and the distillate which is taken as D. So, the compositions of these vapor and after partial condensation the vapor which is taken out, and reflux which is liquid is

feed back to the column, distillation column all these three compositions are different when we use the partial condenser.

A partial condenser functions as an equilibrium separation stage, because in the partial condensers we will have the vapor and the condensed liquid which are in, come in contact in the reflux drum they reach in equilibrium. So, columns with partial condenser effectively have an extra ideal stage. So, this is very important when we use the total condenser, then all the vapors is condensed product card is also liquid will have same compositions and it is returned that is, also have the same compositions as reflux, so in case of total condenser. Number of stages required will not be considered as an stage, when we use the partial condenser, then since the reflux drum will act as an equilibrium stage so one extra ideal stage should be considered.

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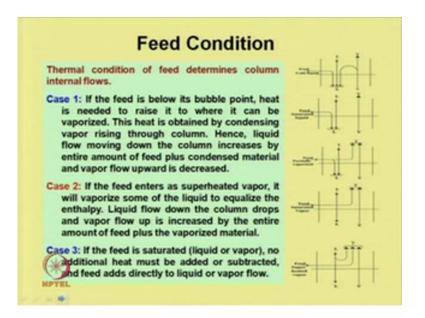


Now reboiler, most of the cases we use the partial reboiler; that means, only partially vaporized part of the liquid in the column base. So, partial reboiler also considered as an ideal stage or one extra stage. The reboilers are of different types; one is thermosiphon types, which rely on the thermal effects on density to draw liquid through the heat exchanger.

So, one is thermosiphon type, and another is forced circulation type. Force circulation type and it use a pump to force a liquid through, in large complex columns sides stream

reboilers can be used these draw liquids of a tray heat it and then return the vapor liquid mixture to the same or similar trays, so which is known as the side stream reboiler.

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Now, as we said the feed which may be multiple feed or single feed, but the feed has different conditions. So, the thermal conditions of feed determine the column internal flow. Suppose, considered this is the feed tray, so we will consider different cases; case one, where if the feed is below its bubble point. So, if it is below its bubble point then we need to supply heat to raise it where it can be vaporized, to raise the temperature of the feed because the feed is below its bubble point or cold feed, and so the heat should be supplied to increase the temperature to vaporize. So, this heat the obtained by condensing vapor rising through the column, this heat is taken from the condensing vapor which is rising through the column.

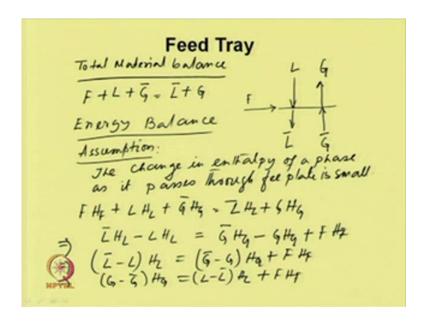
Hence, we can say liquid flow moving down the column increases by entire amount of feed plus condense material and the vapor flow upward is decreased. So, what is happening if we give cold feed, all the feed will flow down along with some part of the vapor which will be condensed that will also flow down. So, from the feed tray the internal flow downwards will be increased, where as the vapor flow towards the top of the tower above the feed tray will be decreased.

Now, considered a case where feed enters as super heated vapor. If it is super heated vapor, so this is the case as super heated vapor, here all the feed will go off along with

some portions of the liquid is coming from the top of the tower will be evaporated, so the column internal flow above the feed tray will increased, where as the liquid flow below the feed tray will decreased. So, this will decrease and this part will increase. So, this is case two.

Another case if the feed is saturated liquid or saturated vapor, if it is saturated liquid, so all the liquid will go down along with the liquid which is coming from the top of the tower and the vapor flow from both the sections remains same. Similarly, if we use the feed saturated vapor, then the vapor flow will be added of in the top of the feed tray, and then the liquid flow both of the sections remains same; and if it is vapor and liquid mixture, if feed contains partially vaporized then it will divide in two sections, so the liquid flow and the vapor flow for both the sections will change.

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Now, let us considered a feed tray where the vapor which is coming to this is G bar and the vapor which is coming is G, and the liquid is L bar, and liquid which is coming from the top is L and the feed which is given is F. so, if we do the total material balance on the feed tray, total material balance if we do we can write, F plus L plus G bar is equal to L bar plus G. Now, if we do the energy balance; we can take assumptions the change in enthalpy, enthalpy of a phase as it passes through feed plate is small.

So, the change in enthalpy of a phase as it passes through plate is small. If we assume that then we can write the energy balance equation; F H F plus L H L plus G bar H G is

equal to L bar H L plus G H G. So, from this we can write L bar H L minus L H L would be equal to G bar H G minus G H G plus F H F. So, we can write L bar minus L H L is equal to G bar minus G H G plus F H F or G minus G bar H G would be equal to L minus L bar H L plus F H F.

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Feed Tray

using Total material balance:

$$\bar{q} - \bar{q} = \bar{L} - L - F$$
 $(\bar{L} - L) + H_2 = (\bar{L} - L - F) + H_3 + F + H_4$ 
 $= (\bar{L} - L) + H_3 + F (H_4 - H_3)$ 
 $= (\bar{L} - L) (H_2 - H_3) = F (H_4 - H_4)$ 
 $= (\bar{L} - L) (H_2 - H_3) = F (H_4 - H_4)$ 
 $= (\bar{L} - L) (H_2 - H_3) = F (H_4 - H_4)$ 
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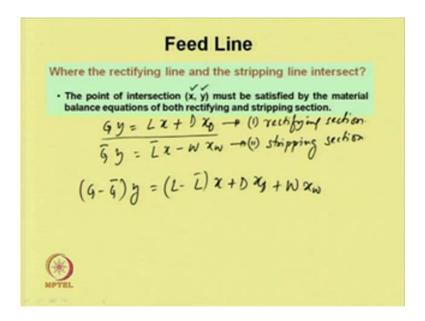
Using total material balance, we can write G bar minus G is equal to L bar minus L minus F. So, we use L bar minus L H L is L bar minus L minus F H G plus F H F. So, if we simplify L bar minus L F G plus F H F minus H G, and from this we can write L bar minus L into H L minus H G is equal to F into H F minus H G, and we can write L bar minus L by F is equal to H F minus H G by H L minus H G, or we can write H G minus H F divided by H G minus H F the quantity we can call it q.

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So, the left hand side, which is L bar minus L, L bar minus L this is nothing but, increase in the liquid flow rate across the feed tray due to introduction of feed; which we can write the rate of input of feed, and hence we can say q is equal to the fraction of liquid feed.

So, another significance of this q is defined as; q is equal to the heat required, if you see q is equal to H G minus H, H G minus H F and H G minus H F. H G minus H F divided by H G minus H L, which we can write the heat required to convert one mole of feed to saturated vapor divided by molar heat of vaporization of saturated liquid, molar heat of vaporization of saturated liquid.

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Now, we will discuss feed line. So this important because it will be very useful to know where the rectifying line and the stripping section line intersect. So, the point of intersections between the stripping line and the rectifying line should meet at a intersection point x, y and since the stripping section line, which we will discuss now how to obtain the stripping section line and the rectifying section line, that is, governed by the material balance equations of both the sections, then both the sections equations must be satisfied with the intersection points.

If we do the material balance for both the sections G into y is equal to L x plus D x D and G dash y is equal to L bar x minus W x w. Let us see the figure where we designate this is D and the composition is x D and the vapor which is coming out is G and the bottom which is coming out is G and G and G and G and below the feed tray is G and G are composition, the liquid above the feed tray is G and below the feed tray is G and the rectifying section we can do the material balance equations for the feed line. This is the rectifying section and this is the stripping section. So, if we subtract one from the two will get, G minus G bar into G is equal to G minus G bar into G ba

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Feed Line

Overall Material balance:

$$(G-\overline{G}) y = (L-\overline{L})x + F z_F$$

Devide by  $F$  to the total material balance  $g_F$ 
and use definition of  $g$ :

$$\overline{G-G} + 1 = \overline{L-L} = 2$$

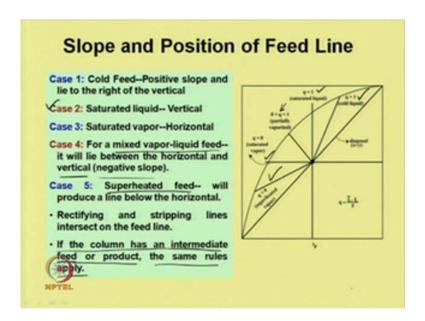
$$\overline{G-G} y = \overline{L-L} x + z_F \Rightarrow \overline{(J-1)} y = -9x + \overline{y}_F$$

Freed line equal in  $\overline{G} = \overline{G} =$ 

Now, if we use the overall material balance; overall material balance if we do that we can write G minus G dash into y is equal to L minus L dash into x plus F z F. Now, if we divide by F to the total material balance equation and use definition of q. So, if we do that we can write G bar minus G by F plus 1 would be equal to L bar minus L divided by F, this would be equal to q, from this relation we can write G bar minus G by F into y is equal to L minus L bar divided by F into x plus z F.

So, then if we apply this q, so this will be minus q, minus 1 into y which is equal to minus q x plus z F. So, from this we can write y is equal to q by q minus 1 into x minus z F divided by q minus 1. So, this is the operating line equations for the feed line or it is known as the feed line equation.

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So, this can be plotted so we will see how to plot the feed line and we will see the positions for different cases. So, one case is cold feed if it is cold feed, then the slope is positive and lie to the right to the vertical line. If it is cold feed the q is greater than 1, so it will lie with a positive slope right to the vertical line.

Now, if it is saturated liquid case two; if it is saturated liquid then the q is 1. So, it will lie in vertically, which is shown over here. Now, if it is saturated vapor then the q is 0; so it will lie horizontally, which is shown over here, this is the for saturated vapor, and if it is mixture of vapor and liquid feed, so it will lie between the horizontal and the vertical since the slope is negative. So, depends on the value of q and with that slope it will lie between the vertical line and the horizontal line.

Now, if feed is superheated feed then it will produce a line below the horizontal line because q is less than 1, which is shown over here which is q is less than 1 it will lie below the horizontal line. The stripping line and the rectifying section lines, they intersect the feed line or the q line. If the column has an intermediate feed or product the same rules apply depending on the conditions.

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# Number of Trays by McCabe Thiele Method

#### Assumption 1:

- Molar flow rate of liquid from one tray to another is constant over any section of the column.
- Molar flow rate of vapor in respective sections remain constant.
- Assumption is true when molar heat of vaporization of mixture does not depend upon composition or temperature in the column.
- If constant molar overflow occurs, mass exchange between phases occurs in equimolar counter-diffusion mode.

#### **Assumption 2:**

· Heat loss from the column is negligible

Major steps of the graphical construction in the McCabe-Thiele method:

Draw equilibrium curve using the available data,

praw operating lines for the rectifying and the stripping sections

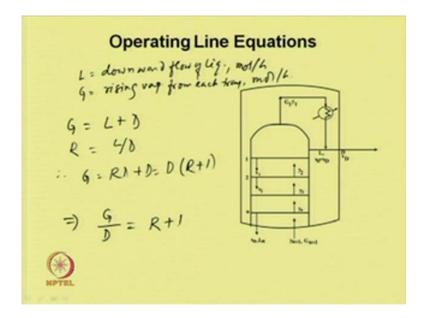
raw steps between the equilibrium and operating lines to find

Now, we will discuss the number of trays required for a given separation by McCabe-Thiele method. So, the important assumptions which are taken in case of McCabe-Thiele method is the two important assumptions; one is molar flow rate of the liquid from one tray to another is constant over any sections of the liquid similarly, the molar flow rate of the vapor in any sections of the column remains constant. So, both molar flow rate of the liquid and molar flow of the vapor of the respective sections rectifying sections or stripping sections any sections of the column remains same.

This assumption is true when the molar heat of vaporization of mixture does not depend upon the composition or temperature in the column, these assumptions would be valid. So, we call it constant molar over flow if constant molar over flow occurs then mass exchange between phases occurs in equimolar counter diffusions unit. Another assumption which is taken is the heat loss from the column is negligible.

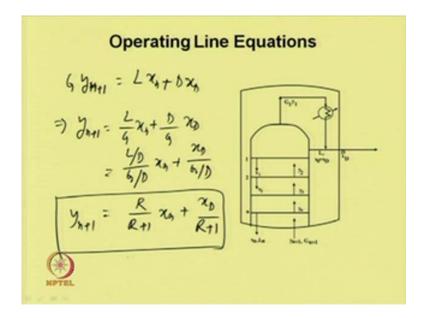
Based on this two assumptions; we follow the graphical method to obtain the number of stages required for a given separations. The procedure normally which follows draw the equilibrium curve using the available data; draw operating lines for the rectifying and stripping sections; draw steps between the equilibrium and operating line to find out the number of ideal trays.

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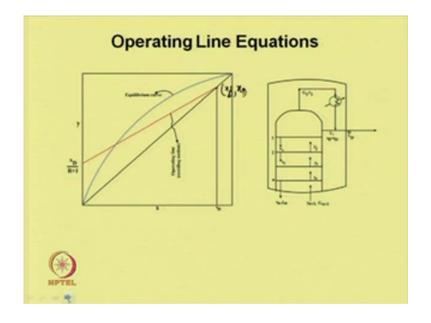
Now, we will try to find out the operating line for different sections. As we said above the feed tray is called the rectifying sections and below the feed tray is known as the stripping section. Now, we will see how to obtain the operating line for both the sections. Let L be the downward flow of liquid, mole per hour and G is the rising vapor from each tray mole per hour. So, if we do the total material balance since we have assume equimolar over flow, so all the subscript for the liquid and vapor flow rates we can amid, and if we do the total material balance for the envelope, this envelope we can write G is equal to L plus D, and another term which is defined reflux ratio R which is L by D. So therefore, we can write G is equal to R D plus D is equal to D into R plus 1. So from this we can write G by D is equal to R plus 1.

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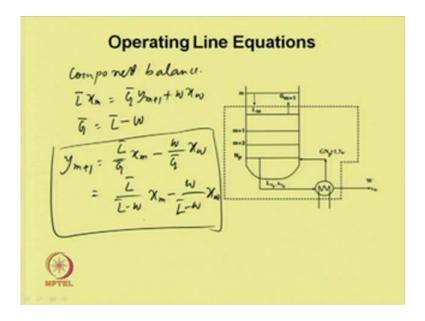
So, if we do the material balance, the material balance equations G y n plus 1 is equal to L x n plus D x D. So, from this we can write Y n plus 1 would be L by G x n plus D by G x D. So, we can write L by D divided by G by D into x n plus x D by G by D. So, we can write this is equal to R by R plus 1 x n plus x D by D plus 1. So, this is the equation of the operating line in the rectifying section. If we know the intercept D by D plus 1, and if we know the point x D then we can plot the operating line.

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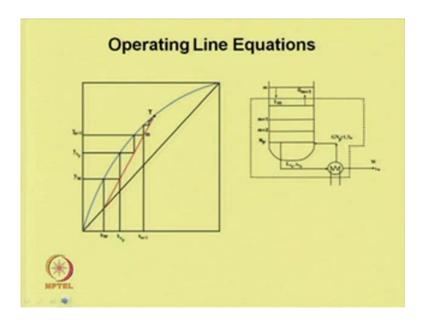
So, this is the point x D, x D and we know the slope then we can plot the operating line, and another simpler way instead of taking slope, we know x D by R plus 1. So, reflux ratio is known x D is known to us, so we know the intercept. So, using this intercept and this x d point we can plot the operating line for the rectifying section.

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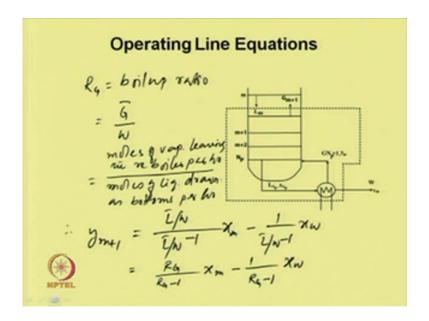
Similarly, if we do the balance over the stripping section, we can write the component balance; we can write L bar x m is equal to G bar y m plus 1 plus W x w. now, if we put G bar the total material balance is L bar minus W. So, we can write Y m plus 1 is equal to L bar by G bar into x m minus W by G bar into x w. so, from which we can write L bar by L bar minus W x m minus W divided by L bar minus W into x w. So, this is the equations of the operating line in the stripping section.

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If we know this slope and we know x w, then we can plot the operating line with x w and the slope we can plot the operating line.

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Rectifying sections, we define the recycle ratio or the reflux ratio; we can define here R G which is known as the boil up ratio. So, which we can write G bar by W is the moles of vapor leaving the reboiler per hour divided by moles of liquid drawn as bottom product bottoms per hour. So, we can write Y m plus 1 would be equal to L bar by W divided by L bar by W minus 1 x m minus 1 by L bar by W minus one x w. So, we can

write R G by R G minus 1 x m minus 1 by R G minus 1 x w. So, this is the operating line equations in terms of the boil up ratio.

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# Example

A mixture of 45 mol% n-hexane and 55 mol% n-heptane is subjected to continuous fractionation in a tray column. The feed rate is 100 kmol/hr. The distillate contains 90% n-hexane and the residue contains 5% n-hexane. The reflux is saturated liquid and the reflux ratio is 2.5. The feed is saturated liquid. The relative volatility of n-hexane in the mixture is 2.36. Plot the operating line for rectifying and stripping section.



Now, let us take simple examples. A mixture of 45 mol percentage n-heptane and 55 mol percentage n-heptane is subjected to continuous fractionation in a tray column. The feed rate is 100 kilo mol per hour. The distillate contains 90 percentage n-hexane and the residue contains 5 percentage n-hexane. The reflux is saturated liquid and the reflux ratio is 2.5. The feed is saturated liquid. The relative volatility of n-hexane in the mixture is 2.36. Plot the operating line for rectifying and stripping section.

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Given, F is equal to 100 kilo mol per hour, z f is 0.45, x D is 0.9, x w is 0.05, and R is 2.5. So, we can calculate from the total material balance F is equal to D plus W. So, from this we can write 100 is equal to D plus W. Now, if we do the material balance, F z F is equal to D x D plus W x w, it will be 100 into 0.045 is equal to D into 0.9 plus W into 0.05. So, from this we can write 45 would be equal to 0.9 D plus 0.05 W. Now, putting the value of D, D is equal to 100 minus W. So, we can write 0.9 100 minus W plus 0.05 W. From this we can write W is equal to 45 divided by 0.85, which would be 53 kilo mole per hour. So, we can get D is equal to 47 kilo mol per hour.

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Solution
$$R : \frac{L_0}{D} = 3 \cdot 2.5 = \frac{L_0}{47}$$

$$= 3 \cdot L_0 = 117.5$$

$$G_1 = 0 \cdot (R+1) = 47x(2.5+1) = 144.5$$

$$G_2 = G_3 = 164.5$$

$$G_3 = 164.5$$

$$G_4 = G_3 = 164.5$$

$$G_4 = G_3 = 144.5$$

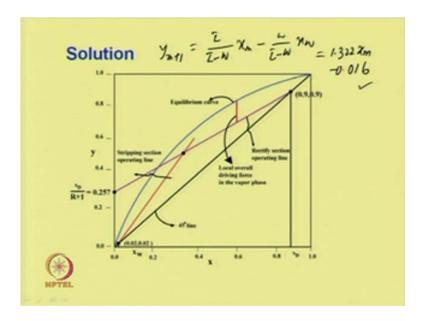
$$G_5 = G_4 = 144.5$$

$$Y_{n+1} = \frac{R}{R_{11}} \chi_0 + \frac{\chi_0}{R_{11}} = \frac{2.5}{2.51} \chi_0 + \frac{0.9}{2.5+1} \chi_0$$

$$= 0.714 \chi_0 + 0.257$$

Now, we know reflux ratio R is L 0 by D, so from this 2.5 reflux ratio is given and F 0 by D is 47. So, from this we can obtain L 0 is equal to 117.5. So, the value of vapor flow G1 is D into R plus 1 which is equal to 47 into 2.5 plus 1, and this will give 164.5. So, G would be G 1 which is 164.5. Now, for stripping section L bar would be L plus 100 is equal to 117.5 plus 100. So, which is equal to 217.5 and G bar would be G which is equal to 164.5. Now, the operating line for the rectifying section Y n plus 1 is equal to R by R plus 1 x n plus x D divided by R plus 1. If we substitute 2.5 by 2.5 plus 1 x n plus 0.9 divided by 2.5 plus 1 x D. So, this will led to 0.714 x n plus 0.257.

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Similarly, for stripping section we can obtain Y m plus 1 would be L bar by L bar minus W x m minus W by L bar minus W x w. So, if we substitute we would obtain 1.322 x m minus 0.016. So, this is the equations of the stripping operating line and this is the equations of the rectifying section operating line. So, we know the value of intercept. So the intercept is 0.257 and with this intercept and we know the value of x D x D, with these two points we draw the operating line and then we know the x w which is at 0.02, 0.02. So, from this with the slope of 1.322, we draw the operating line. This way we can draw the operating line for both the sections.

Thank you.