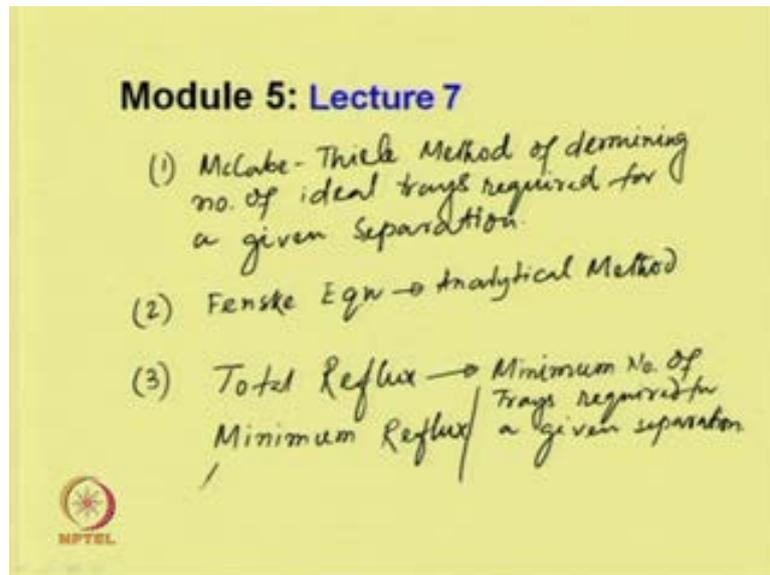


**Mass Transfer Operations I**  
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**Module - 5**  
**Distillation**  
**Lecture - 7**  
**Fractional Distillation: Minimum Reflux and Pinch Point**

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Welcome to the seven lecture of module five, we are discussing in this module distillations. So, let us have a recap of our earlier lecture, in the last lecture, we have considered the assumptions in case of McCabe-Thiele method of determining minimum number of trays in a distillation column required for a given separation. We have done McCabe-Thiele graphical method, method of determining number of ideal trays required for a given separation. Secondly, we have seen how to calculate the number of ideal trays required theoretically. So, Fenske equations we have discussed, how to calculate the number of ideal trays theoretically or analytical method. And we have discussed the limiting cases or the limitations, in case of distillations two important limitations: one is total reflux and second one is minimum reflux.

So, we have discussed the total reflux which is commonly used before the start of the distillation operations, the initially at the feed flow remains closed after the initial injection of the feed, and then the total vapor which is generated at the top of the tower is

condensed and returned back to the tower without taking any product out from the column. In that case, we can calculate or we can obtain the minimum number of trays required for a given separation.

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**Minimum Reflux: Pinch Point**

Pinch Point  $\Rightarrow$  Intersection of an operating line and eqn curve  
 $\Rightarrow$  Two pinch points

To rectify the pinch problem  
 $\rightarrow$  shift the feed entry point  
 $\rightarrow$  increase boilup ratio & reflux which in turn increase the operating cost and energy consumption

If the pinch point is at the intersection of feed line & eqn curve  $\Rightarrow$  minimum reflux condition.

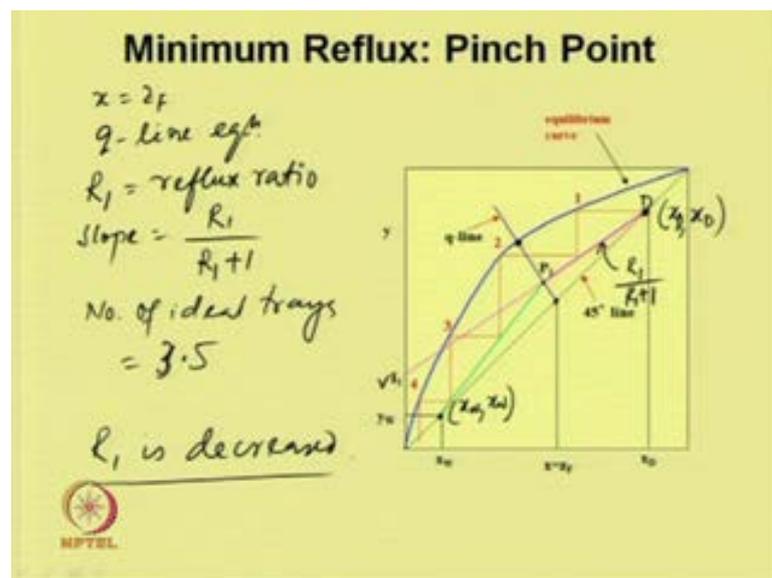
So, today we will discuss another limitations, which is minimum reflux. So, the minimum reflux and pinch point. So, what is pinch point? Pinch point is the intersection of an operating line and the equilibrium line; intersection of an operating line and equilibrium curve. If we considered a simple distillation column or fractionators fractional distillation, where we have two sections as we have discussed before - one is rectifying section and another is stripping section. Since, for the two sections we have two operating line. So, commonly we have two pinch point two pinch point: one for the rectifying sections, another is for the stripping operating lines.

What it indicates? If the distance between the equilibrium curve and the operating line are very close, then the pinch may happen; that means, the distance between the equilibrium curve and the operating line are very close. So, we need a large number of trays to a given separation or it will give a very small separation using very large number of trays. So, how to check or how to rectify this pinch problem; one practice to cure it or to change the pinch point is to shift the feed line, to rectify the pinch problem what we have to do? Shift the feed entry point. So, this will increase the cost of operation, because in this case we have to increase boil up ratio and reflux, which in turn increase the

operating cost and energy consumption, but this is maybe the only realistic solution to rectify this problem.

So, a pinch at the intersections of the feed line, and the equilibrium curve is known as the minimum reflux. If the pinch point is at the intersection of feed line and equilibrium curve, then it is called minimum reflux condition, at the minimum reflux conditions which is opposite to the total reflux; at total reflux, we need minimum number of stages for a given separation whereas, at the pinch point that is at minimum reflux we need the infinite number of stages for the given separations; that means, the distance between the operating line and the equilibrium line is 0. So, that when we try to obtain the number of stages by McCabe-Thiele method by stage of method we cannot go out from the feed point, because the distance between the equilibrium curve and the operating line curve is 0.

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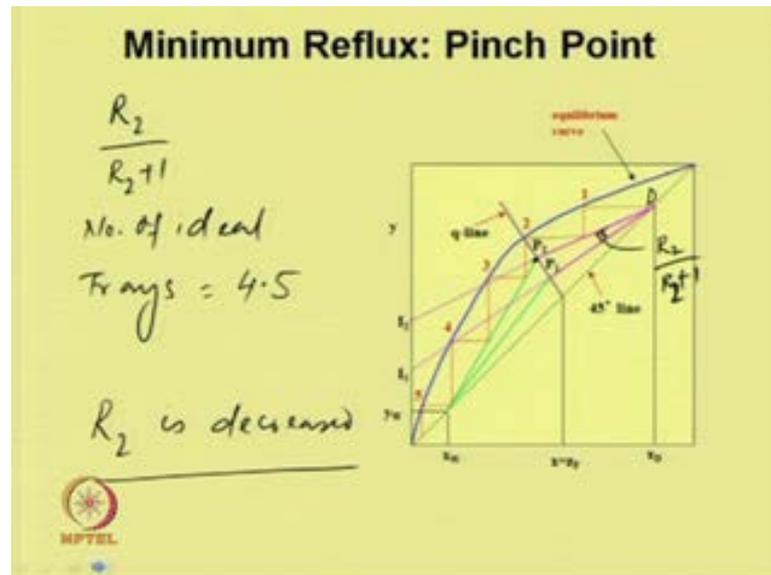
So, to obtain minimum reflux ratio for a particular separation with the method is to find out the pinch point, to find out the pinch point let us see what are the methods should be followed, we should know the feed entry point; that is the feed line equations where the feed compositions is given say  $x$  is equal to  $z_F$ , this is the feed point. So, feed point is known to us, and the feed condition is known to us. So, we know the  $q$  line equation. So,  $q$  line equation is known to us we can plot the  $q$  line, and we have the equilibrium curve, then we can obtain the intersection point between the feed line and the equilibrium line.

So, this will be the pinch point and then we can plot the operating line in the rectifying section, and then that operating line will intersect at what point in the y axis will give the intersect.

So, that we can get the minimum reflux ratio, this is the rectifying section distillate conditions. So, the distillate which is  $x_D$  and  $x_D$  is known to us and another point which is  $x_w$  and  $y_w$  or  $x_w$ , this point is known to us. Let us consider  $R_1$  is the reflux ratio, if we plot the equilibrium curve and then we plot the q line, we know  $x_D$   $x_D$  point and the reflux ratio  $R_1$  is given. So, the slope of the operating line in the rectifying section slope is  $R_1$  divided by  $R_1 + 1$ . So, slope is known to us suppose this is the operating line slope, which is  $R_1$  divided by  $R_1 + 1$ . So, we can plot the operating line, and it intersects the y axis at  $I_1$ , and intersection point between the feed line and q line is at point  $P_1$ , suppose this point is  $D$ . So,  $D P_1$  is the operating line in the rectifying section and  $D I_1$ , where  $I_1$  is the intersection points of operating line of the rectifying section on y x. And we know the point  $x_w$ ,  $x_w$  from that we can plot between the intersection point of operating line of rectifying section and the q line, we can join the stripping section operating line.

So, if we obtain the number of stages required by McCabe-Thiele method we can stretch of from the top of the tower from  $D$  point, then we can come to the equilibrium line and then drop on the operating line rectifying section and it will cross over. So, it will give approximately 4 or 3.5 the number of ideal trays is about 3.5. So, this is the number of trays required.

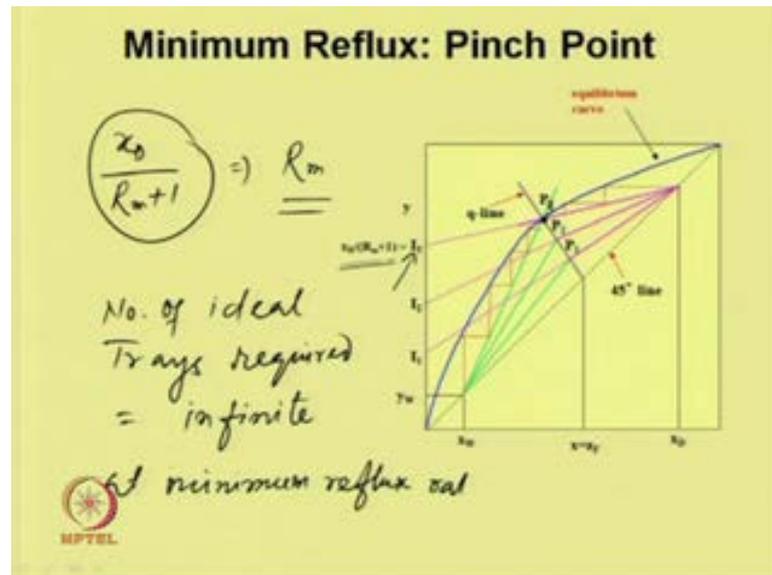
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Suppose, if we decrease the reflux ratio. So,  $R_1$  is decreased. So, what will happen? The slope will be  $R_2$  by  $R_2 + 1$ . So, the slope will decrease, suppose the slope meets at the point  $P_2$  between the operating line and the  $q$  line at point  $P_2$ . So, the slope decreases, but intersection point on the  $y$  axis increases at  $I_2$ . So, this operating line slope is  $R_2$  divided by  $R_2 + 1$ , then we can stretch off and we see that the number of trays required is increases, because the driving force between the operating line and the equilibrium line decreases; the number of ideal trays would be around 4.5, which is more than the earlier case.

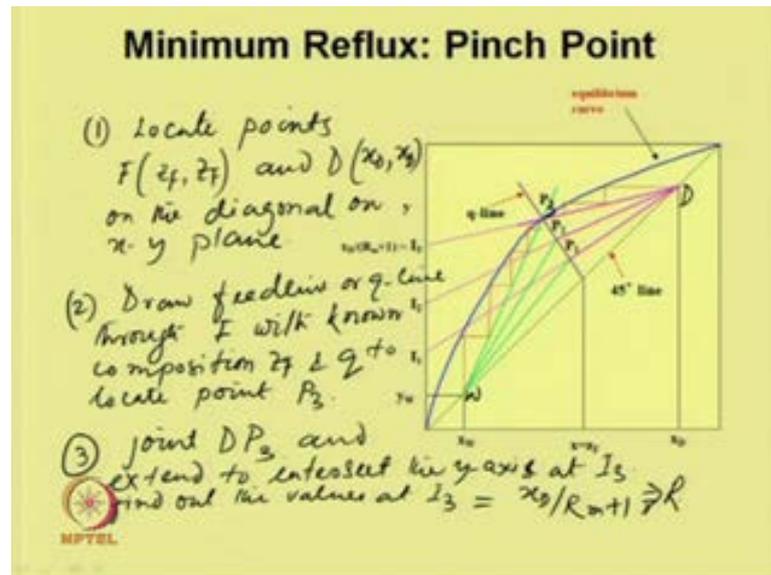
Similarly, if we decreased for the  $R_2$  is decreased, then the operating line of rectifying section and the  $q$  line meets at a point say  $P_3$  which is on the equilibrium curve.

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And the intersection point also increases to I 3. If we try to calculate the number of ideal trays required, we can see that the stage becomes very small as we reach to the pinch point; that is at P 3. And it is not possible to pass this feed tray by the stage of method, because the driving force is 0 at the pinch point, from this point if we intersect at the y axis which is at I 3, this will give the minimum reflux ratio required for this particular separation at minimum reflux ratio, we know  $x_D$  divided by  $R_m$  is the minimum reflux ratio. So, this point we can calculate from the graph, and we can obtain  $R_m$ . So, the number of trays required - number of ideal trays required would be infinite at minimum reflux ratio.

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So, let us write down the procedures to be followed to find out the minimum reflux ratio. So, first we have to locate points  $F$  the feed points which is  $z_f$  and  $D$  that is distillate point  $x_D$  on the diagonal on  $x-y$  plane. Secondly, we have to draw the feed line; feed line or  $q$  line through point  $F$  with non composition  $z_f$  and  $q$  quantity  $q$ , and then locate the point  $P_3$  to locate point  $P_3$ . And then join suppose this is  $D$ . So, this is  $w$ , join  $DP_3$  and extend to intersect the  $y$  axis at  $I_3$ , then we have to find out the value at  $I_3$  find out the value at  $I_3$ , and equate it to  $x_D$  divided by  $R_m + 1$ . So, that we can obtain  $R_{\text{minimum}}$  from which we can calculate  $R_m$ .

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### Example

A mixture of 45 mole % n-hexane and 55 mole % n-heptane is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% n-hexane and the residue contains 5% n-hexane. The feed is saturated vapor. The relative volatility of n-hexane in mixture is 2.36. Determine the minimum reflux ratio for this separation.

Now, let us take an example a mixture of 45 mole percent n-hexane, and 55 mole percent n-heptane is subjected to continuous fractionation in a tray column at one atmosphere total pressure. The distillate contains 95 percent n-hexane, and the residue contains 5 percent n-hexane, the feed is saturated vapor, the relative volatility of n-hexane in mixture is 2.36 determine the minimum reflux ratio for this separation.

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**Solution**

A mixture of 45 mole % n-hexane and 55 mole % n-heptane is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% n-hexane and the residue contains 5% n-hexane. The feed is saturated vapor. The relative volatility of n-hexane in mixture is 2.36. Determine the minimum reflux ratio for this separation.

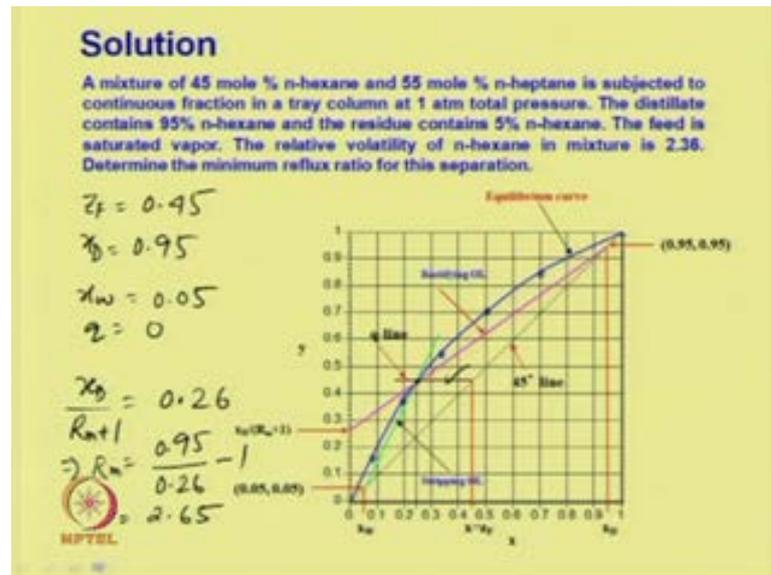
$$y = \frac{\alpha x}{1 + (\alpha - 1)x} = \frac{2.36x}{1 + (2.36 - 1)x} = \frac{2.36x}{1 + 1.36x}$$

x	0	0.076	0.199	0.341	0.505	0.705	1
y	0	0.163	0.37	0.55	0.71	0.85	1



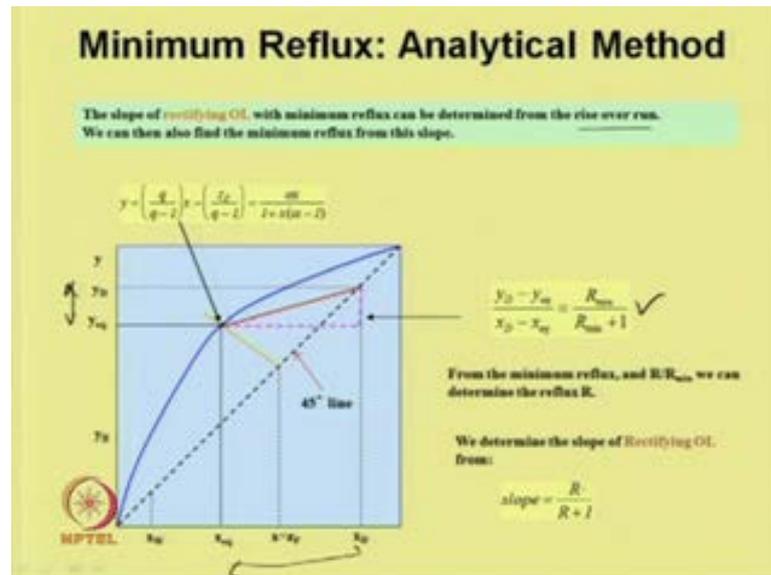
The reflux ratio which is given is 2.36, and we know the equilibrium relation y is equal to alpha x divided by 1 plus alpha minus 1 x, where alpha is the relative volatility this we can substitute and this is the equilibrium relationship between x and y. And if we take a value of different x we can calculate the value of y, then we can plot the equilibrium curve.

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So, this blue line represents the equilibrium curve the conditions which are given  $z_f$  is 0.45,  $x_D$  is 0.95,  $x_W$  is 0.05, the feed is saturated vapor. So,  $q$  is equal to 0 it is the horizontal line, this is the  $q$  line which is plotted and intersects at this point, this is the operating line for the stripping section and this is the operating line from  $x_D$   $x_D$  from the rectifying section operating line, and extend it to this point, and from that point we can calculate  $x_D$  divided by  $R_m + 1$  is around 0.26. So, from this we can calculate  $R_m$  is equal to  $0.95 \times D$  is 0.95 divided by 0.26 minus 1. So, which is equal to 2.65, the minimum reflux ratio is 2.65, this is the graphical method how we can calculate the minimum reflux ratio.

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Now, this minimum reflux ratio we can also obtain analytically, the slope of the rectifying operating line with minimum reflux ratio can be determine from the right over run, then we can find the minimum reflux ratio. As we can see from this, this is the equilibrium curve, and this is the operating line in the rectifying section, this is q line we know the equations of q line y is equal to q by q minus 1 x minus z f by q minus 1 which we can relate with the equilibrium curve, which is alpha x divided by 1 plus x alpha minus one, they should meet at this point and this two equations should be valid. If we see from this figure, the distance y D minus y equilibrium divided by x D minus x equilibrium this distance, and this distance, this will give the slope of the operating line which is nothing but R minimum by R minimum plus 1. Then if we know R by R min then we can calculate R, and then we can calculate the rectifying sections operating line slope R by R plus 1.

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**Minimum Reflux: Analytical Method**

$$\frac{R_{min}}{R_{min} + 1} = \frac{y_D - y_{eq}}{x_D - x_{eq}} = \frac{x_D - y_{eq}}{x_D - x_{eq}}$$

$$\Rightarrow \frac{R_{min} + 1}{R_{min}} = \frac{x_D - x_{eq}}{x_D - y_{eq}}$$

$$\Rightarrow 1 + \frac{1}{R_{min}} = \frac{x_D - x_{eq}}{x_D - y_{eq}}$$

$$\Rightarrow \frac{1}{R_{min}} = \frac{x_D - x_{eq}}{x_D - y_{eq}} - 1 = \frac{x_D - x_{eq} - x_D + y_{eq}}{x_D - y_{eq}} = \frac{y_{eq} - x_{eq}}{x_D - y_{eq}}$$

$$R_{min} = \frac{x_D - y_{eq}}{y_{eq} - x_{eq}}$$



Let us see how to do that R minimum by R minimum plus 1 is equal to y D minus y equilibrium divided by x D minus x equilibrium. So, we can also write x D minus y equilibrium divided by x D minus x equilibrium. So, from this we can write R min plus 1 divided by R min is equal to x D minus x equilibrium divided by x D minus y equilibrium or we can write 1 plus 1 by R minimum would be equal to x D minus x equilibrium divided by x D minus y equilibrium. Then we can write 1 by R min is equal to x D minus x equilibrium divided by x D minus y equilibrium minus 1. So, which is equal to x D minus x equilibrium minus x D plus y equilibrium divided by x D minus y equilibrium, which is equal to y equilibrium minus x equilibrium, because this is cancelled out divided by x D minus y equilibrium.

So, from this we can write R minimum would be equal to x D minus y equilibrium divided by y equilibrium minus x equilibrium. So, from this relations we can calculate the minimum reflux ratio, if we know the equilibrium values.

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**Minimum Reflux: Analytical Method**

$$R_{\min} = \frac{x_D - y_{eq}}{y_{eq} - x_{eq}}$$

- Does not apply if there are more than two operating regions
- Feed line is the breakpoint for the operating curve
- If there are multiple product cut bet<sup>n</sup> feed point & the reflux then this formula does not apply

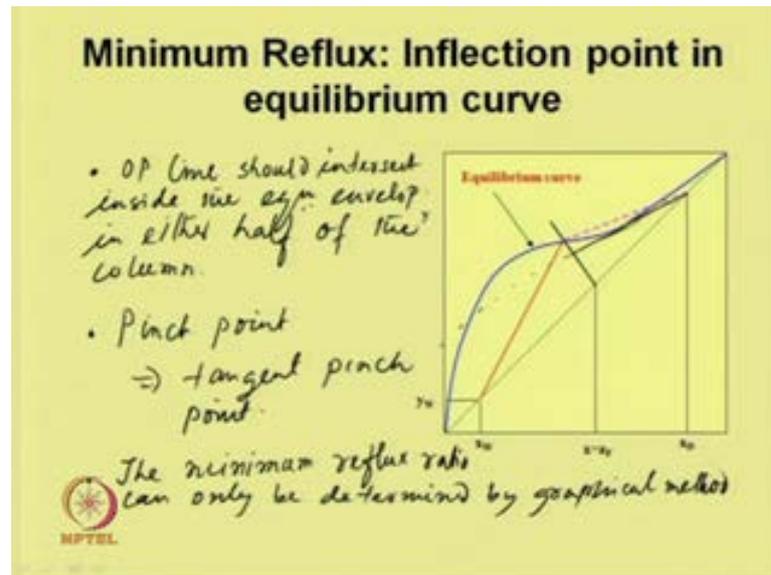


But clearly the expression  $R_{\min}$  is equal to  $x_D$  minus  $y_{\text{equilibrium}}$  divided by  $y_{\text{equilibrium}}$  minus  $x_{\text{equilibrium}}$ , these equations do not apply, if there are two operating regions. If there are more than two operating regions, if there are more than two operating regions in that case the minimum reflux ratio may be calculated from the ratio of the liquid and vapor flows.

Another thing is that this formula only applies when the feed line is the break point for the operating curve in the top portion of the column, feed line is the break point for the operating for the operating curve. So, if there are intermediate product draws that is between the feed point and the reflux entry point, then this formula does not apply, if there are multiple product cut between feed point, and the reflux then this formula does not apply.



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So, at anytime the operating line should not intersect the equilibrium curve at the pinch point outside the equilibrium curve. It should always intersect inside the equilibrium curve. The point is that the operating line should intersect, intersect inside the equilibrium envelop in either half, half of the column. So, in that case the minimum reflux ratio which is obtained is based on the pinch point, which is known as the tangent pinch point, which is shown earlier, this is the point D and this is the operating line, and it should intersect at a point over here. And then we can plot it, this is the tangent points, and if we extend to the y axis then this will give the  $x_D$  by  $R_m + 1$ . So, in this type of figure the minimum reflux can only be determined by the graphical method, the minimum reflux ratio can only be determined by graphical method as shown over here.

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### Example

A mixture of 45 mole % A and 55 mole % B is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% A and the residue contains 5% A. The feed is saturated liquid. Determine the minimum reflux ratio for this separation. The equilibrium data for the system is given below:

x	0	0.05	0.15	0.3	0.5	0.7	1
y	0	0.2	0.4	0.65	0.8	0.85	1



Let us take an example a mixture of 45 mole percent A and 55 mole percent B is subjected to continuous fractionation in a tray column at 1 atmosphere total pressure. The distillate contains 95 percent A, and the residue contains 5 percent A, the feed is saturated liquid determine the minimum reflux ratio for this separation the equilibrium data for this system is given below. So, this is the equilibrium data which are given.

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### Solution

A mixture of 45 mole % A and 55 mole % B is subjected to continuous fraction in a tray column at 1 atm total pressure. The distillate contains 95% A and the residue contains 5% A. The feed is saturated liquid. Determine the minimum reflux ratio for this separation. The equilibrium data for the system is given below:

x	0	0.05	0.15	0.3	0.5	0.7	1
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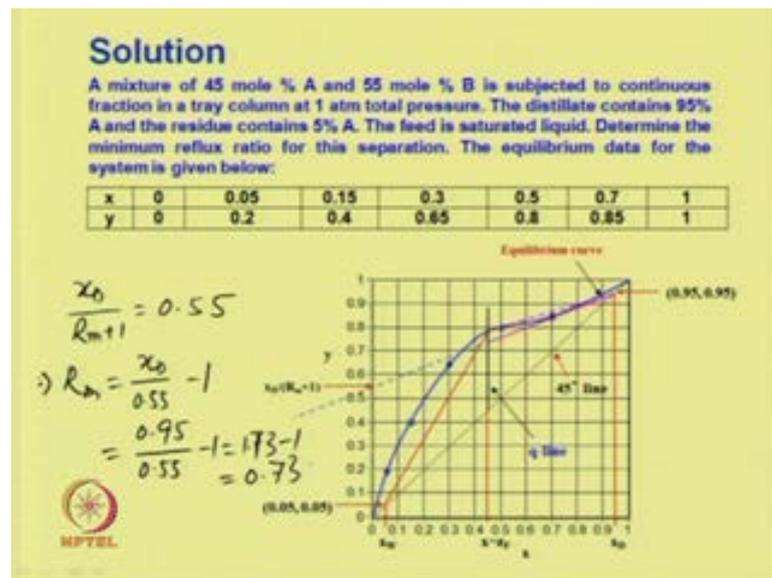
$x_D = 0.95$   
 $x_W = 0.05$   
 $q = 1$   
 $\frac{z}{z-1} = \alpha$   
 $z = 0.45$



If we plot the equilibrium data, so this is the nature of the curve as shown over here. We know the distillate points which is  $x_D$  is 0.95, and  $x_W$  is 0.05, the feed condition

is given  $q$  is equal to 1 saturated liquid. So, slope of the  $q$  line  $q$  by  $q$  minus 1 is infinite which is a vertical line at point  $z$ ,  $z$  is given which is 0.45, so at 0.45. So, this is point F, this is point D, and this is point w, this is the  $q$  line. Now, if we plot the operating line between the intersection point of equilibrium curve, and the  $q$  line and if we connect the operating line for the rectifying section we can see that it intersects outside the envelop of the equilibrium.

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So, what we have to do we need to draw a tangent to this point, and then extend to the  $y$  axis and calculate  $x_D$  by  $R_m$  plus 1, which is about 0.55. Then we can calculate  $R_m$  would be  $x_D$  by 0.55 minus 1, which is equal to 0.95 by 0.55 minus 1 which is 1.173 minus 1 and which is equal to 0.73. So, this is the minimum reflux ratio for this type of separation. So, in general when we design a distillation column, the reflux ratio in general could be varied between 1.1  $R_m$  to 1.5  $R_m$ ; 1.5 times of the minimum reflux ratio reasonable value generally used is 1.2  $R_m$ .

Thank you.