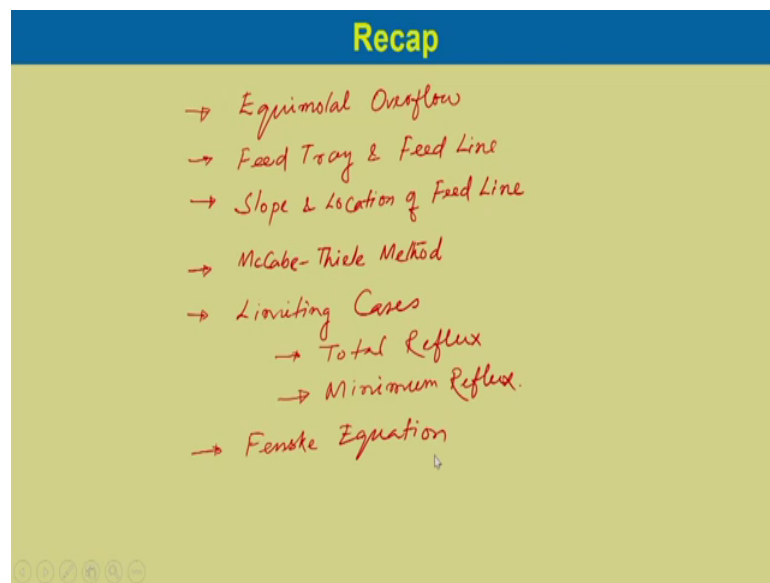


Mass Transfer Operations -I
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Lecture – 32
Pinch Points and minimum reflux

Welcome to 7th lecture of module 5 for Mass Transfer Operation; in this module we are discussing Distillation operation. So, before going to this lecture let us have small recap on our previous lecture. In our previous lecture we mostly discussed the few important topics like equimolal overflow.

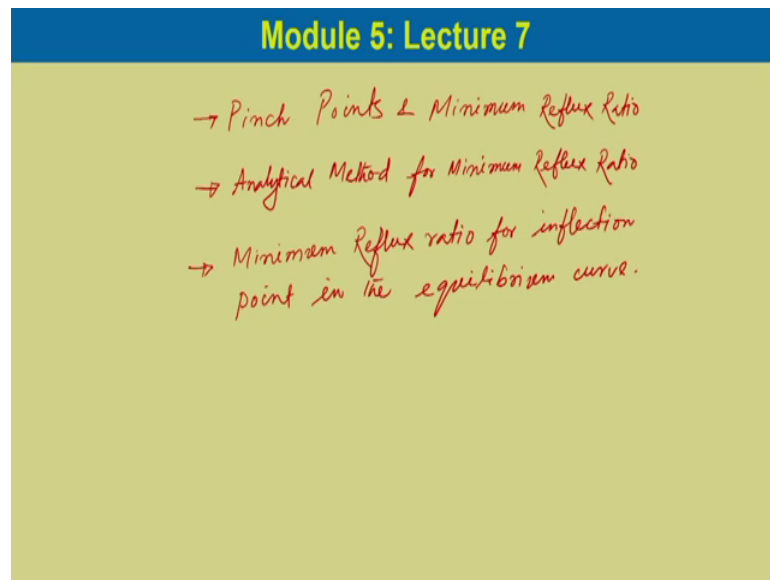
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Then we have discussed feed tray and feed line, slope and location of the feed line and then we discussed McCabe Thiele method; McCabe Thiele method for determination of the number of stages in a distillation column which is graphical method.

And we have discussed different limiting cases and one of them is total reflux and the other case which we have not discussed here that is the minimum reflux and we have considered Fenske equation to calculate the no number of stages Fenske equation to calculate the minimum number of stages required for a particular separation. Now, in this lecture we will mostly concentrate on the pinch point and minimum reflux ratio.

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So, first thing we will consider pinch point and minimum reflux ratio. The second thing we will consider is also very important is analytical method for minimum reflux ratio and the third thing we will consider in this lecture is the minimum reflux ratio for inflection point in the equilibrium curve.

So, basically how to locate the pinch point and how to calculate the minimum reflux ratio from the equilibrium and the operating line diagrams that is the first topic of discussion. Second discussion is whether we can calculate minimum reflux ratio by any analytical method and third think how to calculate the minimum reflux ratio for the equilibrium curve where there is a there is an inflection point in the equilibrium curve. So, how to calculate the minimum reflux ratio for that type of curve? So, let us start with our first point is pinch points and minimum reflux ratio.

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Pinch Points and Minimum reflux ratio

- The intersection of an operating line and the equilibrium curve is called a *pinch point*.
- A simple column will have two pinch points (because there are two operating lines).
- The points change when the operating lines do.
- An existing column can "pinch" if its operating line is too close to its equilibrium curve.
- This means that there are several stages doing very little separation and wasting resources.

So, the intersection of an operating line and the equilibrium curves is called a pinch point. So, by definition it is the intersection point between the operating line curve and the equilibrium curve is called the pinch point. So, a simple column will have two pinch points because there are two operating lines; one is the rectifying section operating line and the other is the stripping section operating line. So, if we plot the both the sections operating line it will essentially cuts 2 different points on the equilibrium curve.

So, we know simply gate for a simple column 2 different pinch points this points can change when the operating lines change. So, if we change the operating lines this pinch point would change. Now an existing column can pinch if its operating line is too close to the equilibrium curve. So, if the operating line is very close to the equilibrium curve now existing column can pinch or the pinch point can be obtained in the existing column.

This means that there are several stages doing very little separation and wasting resources. That means, as the gap between the operating line and equilibrium line are very less. In that case what will happen? The number of stages no required will increase gradually as the distance decreases. And at the pinch point you will see that the you know change or the driving force is very small to make a effect on the separation performance. So, we will need large number of trays or stages for a given separation. So, that will waste our resource. So, if we run the column with very close to the pinch point condition.

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Pinch Points and Minimum reflux ratio

- To cure a pinch, the most direct solution is to move the feed entry point.
- **This is often an expensive proposition.**
- Alternatively, the reflux and boilup ratios can be increased to change the operating lines.
- This will increase operating costs and energy consumption, but may be the only realistic option.
- A pinch at the intersection of the feed line and the equilibrium curve indicates that the column is operating at minimum reflux.

To cure a pinch the most direct solution is to move the feed entry point. So, if we change the feed entry point to some other point or there is a pinch point in the column we can rectify. So, that is the direct solution for the curing of pinch point, but this is of an expensive proposition because you have to change you know all the connections for the feed tray and the design of the feed tray also has to be changed.

So, changing all these will be an expensive proposition. So, alternatively what we can do we can change the reflux or bollup ratio which can be increased to change the operating line. So, this two alternatively we can change this two things; one is the reflux ratio and another is the bollup ratio. So, if this we can increase it will change the or cure the pinch point.

But in this case what will happen? The operating cost and the energy consumption will increase. So, both operating cost and the energy consumption will increase if we increase the reflux ratio and bollup ratios, but should be the only realistic proposition or realistic option to cure the pinch point a pinch at the intersection of the feed line and the equilibrium curve indicates that the column is operating at minimum reflux. So, we know how to draw the feed line and the feed line no touches the or process the equilibrium curve and if the operating line also intersects no at that point. So, in that case that know pins point is known as the reflux condition where you have a very less reflux. So, we call it the column is operating at minimum reflux condition.

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Pinch Points and Minimum reflux ratio

- The minimum reflux condition represents the theoretical **opposite of total reflux -- an infinite number of ideal separation stages.**
- In this case, the intersection of the operating lines lies on the equilibrium curve itself.
- Thus, the distance between the equilibrium curve and the operating lines is at its minimum, the stepping triangles become very small.
- There is **no gap** between the equilibrium curve and the intersection point, so you cannot step past the feed point.

The minimum reflux condition represents the theoretical opposite of total reflux; that means, in case of total reflux we need minimum number of stages because at total reflux both the operating line in the stripping section as well as in the rectifying section lie on the 45 degree diagonal of the equilibrium diagram. So, then the distance between the equilibrium curves and the 45 degree diagonal that becomes all maximum. If we draw the number of stages required for the separation under total reflux we obtained the minimum number of trays required for the separation for a given job.

But in the case of minimum reflux condition which is the opposite of the total reflux; that means an infinite number of ideal stages are required separation stages are required. So, in case of minimum reflux we need infinite number of stages. So, in this case the intersection of the operating lines lies on the equilibrium curve itself. So, intersection point of the operating line lies on the equilibrium curve itself. So, that is the pinch point thus the distance between the equilibrium curve and the operating line is at its minimum. So, you know at the pinch point the distance is 0 because it is lying on the equilibrium curve.

So, since it is known the equilibrium curve if we do the stepping of triangles become very small as it goes from the starting point to the pinch point and know there is no gap between the equilibrium curve and the intersection point. So, you cannot step pass the

feed point. So, if you start from the rectifying section and go to the pinch point so, it will these up to the pinch point you cannot step over because at the pinch no gap is 0.

Similarly in the rectifying section if you start with the starting point and if you go to the towards the pinch point it will know it will lie before that. So, you cannot cross that pinch point because there is no gap. So, it will lie at the intersection point of that is at feed point.

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Pinch Points and Minimum reflux ratio

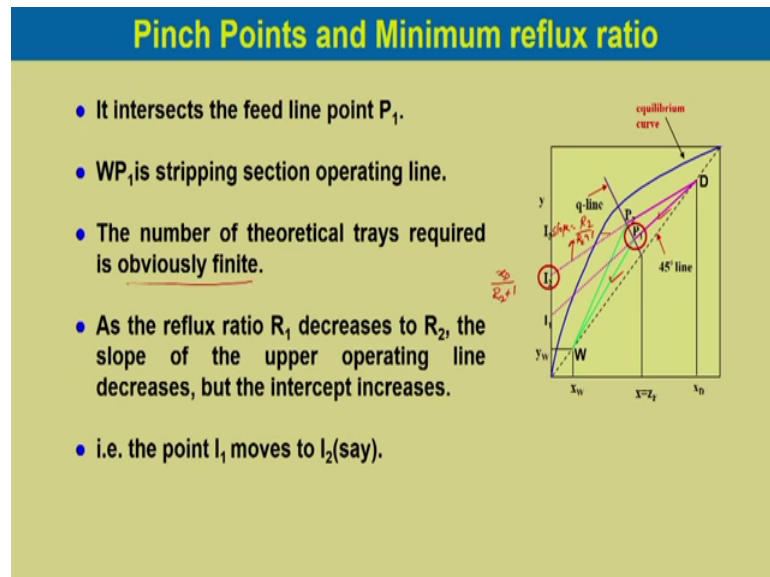
- The determination of the minimum reflux ratio for distillation is based on identifying the pinch point.
- Let us refer to Figure in which the equilibrium curve and the feed line are shown.
- Here: $D(x_D, x_D)$ and $W(x_W, x_W)$ are the terminal points of the operating lines.
- For a particular reflux ratio (R_1 , say), DI_1 is the enriching section operating line having a slope $R_1/(R_1+1)$.

The determination of the minimum reflux ratio for distillation is based on identifying the pinch point to do. So, you know what we need to know let us referred to this figure which is shown over here in which the equilibrium curve and the feed line are shown. So, you can see the equilibrium curve which is the blue line this is the equilibrium curve and you have the feed line which is shown over here that is the q line feed line which is intersection point on the equilibrium curve is over here.

Now, here you we have 2 points; one is point D you can see here point D is the no rectifying section starting point having the coordinates x_D, x_D at this point and you have another point in the stripping section and the starting point is x_W, x_W are the 2 terminal points of the operating lines. Now for a particular reflux ratio say R_1 and DI_1 DI_1 . So, from this D to I 1 this is the enriching section operating line for a particular reflux ratio are and having slope of that operating line of R_1 by R plus 1.

So, that is the slope and intercept is at no I_1 or you we you know calculate by x_D divided by $R_1 + 1$ that is the intercept on the y line. So, I_1 is essentially you know x_D divided by $R_1 + 1$ that is the intersection point and slope is essentially R_1 by $R_1 + 1$.

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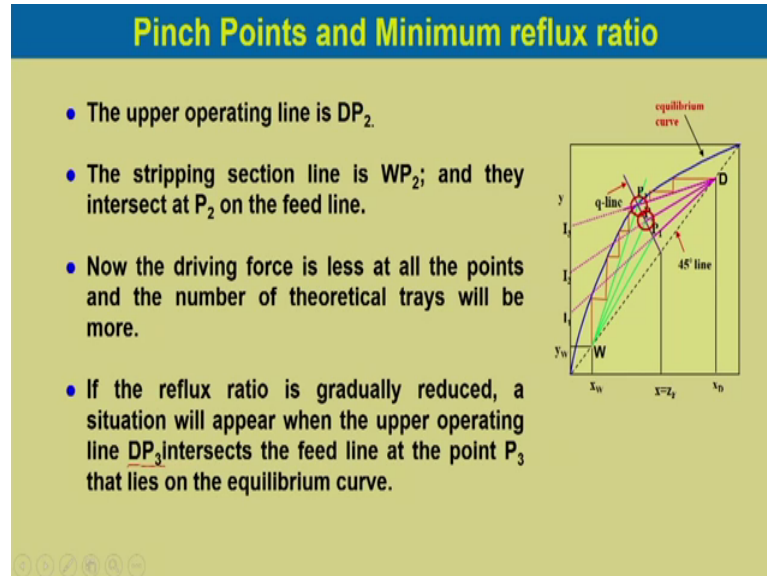
Now, it intercept the feed line point at P_1 . So, this you know $D I_1$ that is the operating line in the rectifying section intercept at a you know at a point between the feed line and operating line at P_1 point and $W P_1$ is the stripping section operating line $W P_1$ would be the stripping section operating line.

Now the number of theoretical trays required is; obviously, finite because there is a gap between the equilibrium curve and these two operating line both in the stripping section operating line and the rectifying section operating line. So, that is $W P_1$ and $D P_1$ which intercept at point P_1 . So, we can just stage off from these two end point and get the number of stages required because there is a gap between these two operating line and the equilibrium line we will get the number of theoretical plate which is finite.

As the reflux ratio R_1 is decreases that is R_1 decreases to R_1 the slope of the upper operating line decreases because the slope is basically will be R_2 by slope of this line would be R_2 by $R_2 + 1$. So, since R_1 decrease to R_2 the slope will also decrease and the intersection point you will reach over the point, I_1 will move to I_2 say I_2 over here

and that is the point it would move I1 to I2 and having the no intercept that is x_D by R_2 plus 1.

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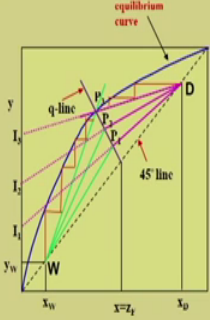
Now, the upper operating line is DP_2 that is intersecting with the feed line at P_2 point over here. So, DP_2 is the upper operating line or the enriching section operating line the stripping section line is WP_2 and they intersect at point P_2 on the feed line. So, both the section operating line will intersect on the feed line. Now the driving force is less at all the points and the number of theoretical trays will be more because in the earlier case you had a large distance compare to the current position which is at P_2 since the driving force is reduced the number of trays required will increase.

Now, if the reflux ratio is gradually reduced and a situation will appear when the upper operating line that is DP_3 intersect the feed line at the point P_3 . So, which is at the point P_3 over here, so, which is over here. So, the upper operating line intercept a point at P_3 and which is on the equilibrium curve. So, the feed line at point P_3 that lies on the equilibrium curve. So, both feed line and the upper section operating line or enriching section operating line meets at the intersection point of the equilibrium curve.

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Pinch Points and Minimum reflux ratio

- The driving force is zero at the point P_3 it is **pinch point**.
- The number of theoretical trays required to achieve the given separation becomes infinite.
- This operating line DP_3 corresponds to the minimum reflux ratio, because if the reflux ratio is further reduced, the operating line will intersect the line at a point R_m .



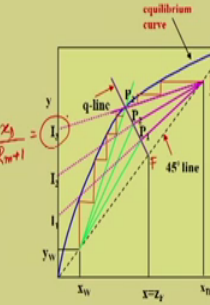
So, in this case the driving force is 0 at point P_3 and it is called pinch point. So, the number of theoretical trays required to achieve the given separation become infinite because as you reach the you know this point to stage off the driving force is very small and you will not be able to pass the feed tray. So, the number of theoretical trays required to give a for a given separations will be infinite at this condition. This operating line DP_3 corresponds to the minimum reflux ratio because if the reflux ratio is further reduced the operating line will intersect the line at point R_m .

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Pinch Points and Minimum reflux ratio

Following steps should be followed:

- Locate the points $D(x_D, x_D)$ & $F(z_f, z_f)$ on the diagonal on the $x-y$ plane
- Draw the feed line through F from the known feed composition and the feed quality (given by q)
- Locate the point P_3 .
- Join DP_3 and extend to intersect the y -axis at I_3 . Find out the ordinate of I_3 and equate it to $x_D/(R_m+1)$. Calculate the minimum reflux ratio, R_m .



Now, what would be the procedure by which we can find the pinch points and the minimum reflux ratio the steps from our discussion we can just summarize is locate the points that is D x D x D and F that is ZFZF and also on the diagonal that is on xy plane.

Then second thing is draw the feed line through point F. So, this is point F over here and this is point D having the coordinates x D x D and point F having the coordinates ZFZF on the xy plane. So, we can just draw the feed line F knowing the feed composition and the feed quality that is given by q. So, as we have discussed the feed quality is defined by the condition fraction of the feed is liquid.

So, based on the feed condition we can draw the q line. Then we can locate once we have the equilibrium diagram and the feed line we have drawn then we can locate the intersection point that is at P 3 between the equilibrium curve and the q line.

Joint DP 3 and extend to intersect into y axis at I 3. So, which is over here from the point of x D x D then we can get the intersect extend this operating line from this pinch point the rectifying section operating line to the y axis to find out the ordinates at I 3 that is equal to x D by R m plus 1. So, at this condition is x D by R m plus 1 which is I 3 because this will give know this values y values corresponds to the pinch point values that is the minimum reflux ratio condition. So, once we get these values the x D is known to us and in this intercept is known to us, so, we can calculate R m.

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Example 1

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 distillation 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.

Solution:

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

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

$$\frac{x_D}{R_m + 1} = 0.26$$

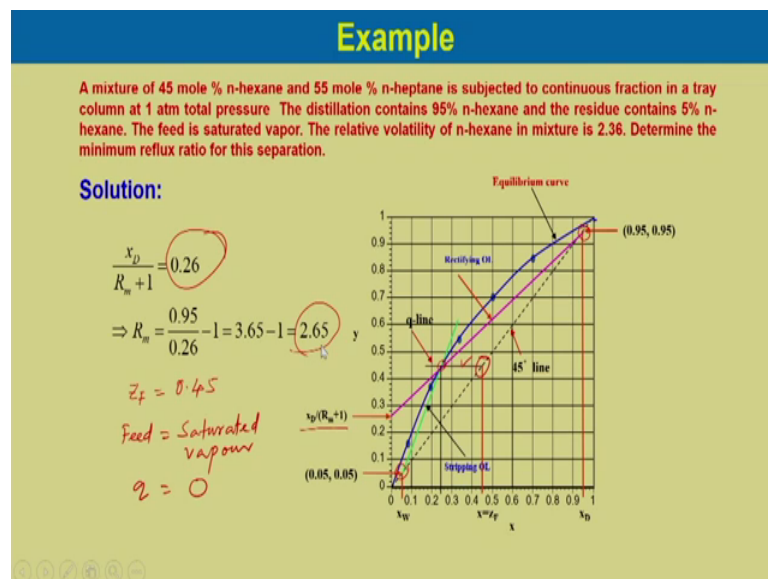
$$\Rightarrow R_m = \frac{0.95}{0.26} - 1 = 3.65 - 1 = 2.65$$

Now, let us take an example to solve the and or to calculate the minimum reflux ratio for a particular separation. A mixture of 45 mole percent n-hexane and 55 mole percent n-heptane is subjected to continuous fractionation in a tray column at 1 atmosphere total pressure the distillate contains ninety 5 percent n-hexane and the residue contains 5 percent n-hexane the feed is saturated vapour the relative volatility of n-hexane in mixture is 2.36.

Now we have to determine the minimum reflux ratio for this separation. So, the relative volatility is given and we know the equations in a relating to relative volatility between x and y to y is equal to alpha x divided by 1 plus alpha minus 1 into x. So, substituting the values of relative volatility we would obtained 2.36 x divided by 1 plus 1.36 x.

Now with these we can get the values of x and y and we can also calculate x D by R m from the intersection point. So, which is 0.26 then R m we can calculate 0.95 by 0.26 minus 1 would be 2.65.

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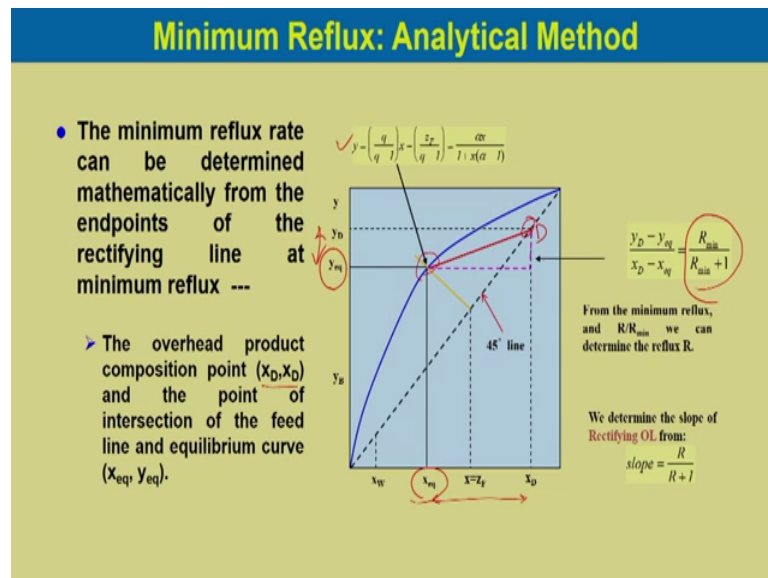
So, which is shown over here. So, this x D by it is shown over here this x D by R m plus 1 is 0.26 from this values. So, we have the you know feed line which is saturated vapour. So, it will be horizontal curve from this points because your feed condition is at point four five that is Z F is 0.45. So, that is at this location. So, Z F you can locate it over here and since the feed is saturated vapour, so, q is equal to 0. So, it is basically you know

horizontal line. So, this is the q line shown over here and feed line it intercept at a particular point over here on the equilibrium curve.

So, we know 95 percent n-hexane in the distillate. So, which is 0.95 95 over here and with this and this point we can just draw the operating line in the rectifying section. So, which is shown over here or we can also draw the operating line in the stripping section knowing 5 percent of the n-hexane in the bottoms or in the on the residue.

So, we can join over here, so, that is the intersection point. And from this we can just locate x_D by $R_m + 1$ which is 0.26. So, once we get these values x_D by R_m we can substitute over here and then if we substitute over here R_m would be equal to 0.95 by 0.26 minus 1 . So, which would be equal to 3.65 minus 1 which is 2.65 , so, the minimum reflux ratio is 2.65 .

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Now, we will discuss the minimum reflux ratio how to calculate the minimum reflux ratio by analytical method. So, if we you know the minimum reflux rate can be determined mathematically from the end points of the rectifying line at minimum reflux. So, at minimum reflux we know the equation of the q line. So, this is the equation of the q line y and this is the equation of the equilibrium line or relative based on the relative volatility equation. So, that should satisfy at this location.

So, the overhead product composition over here that is at point D having the composition of x_D and at this equilibrium position it is x_{eq} and y_{eq} . So, now, if we wanted to calculate the slope of this line at this intersection point. So, it would be $y_D - y_{eq}$ divided by $x_D - x_{eq}$; so, this one and this one. So, this is basically R_{min} divided by $R_{min} + 1$.

So, you know with this if we just substitute this equilibrium values y_{eq} and x_{eq} and x_D is known to us y_D is also known to us. So, at this location x_D , so, we can calculate R_{min} from this expression. So, from this yeah you know equation we can you know calculate the minimum reflux ratio by analytical method.

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Minimum Reflux: Analytical Method

- Minimum reflux corresponds to a pinch at the intersection of the feed line and the equilibrium curve.
- From its formula, the rectifying line has slope = $R_{min}/R_{min} + 1$ and will connect the intersection point (x_{eq}, y_{eq}) and (x_D, x_D) .
- Consequently, we can express the slope in terms of "rise over run" or $(x_D - y_{eq})/(x_D - x_{eq})$

Minimum reflux correspond to a pinch point at the intersection of the feed line and the equilibrium curve. From its formula the rectifying line has the slope R_{min} by $R_{min} + 1$ and we will connect the intersection point of x_{eq} y_{eq} and x_D x_D as we already discussed. So, consequently we can express the slope in terms of the rise over run that is $x_D - y_{eq}$ divided by $x_D - x_{eq}$.

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Minimum Reflux: Analytical Method

Algebraic rearrangement gives the desired formula:

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

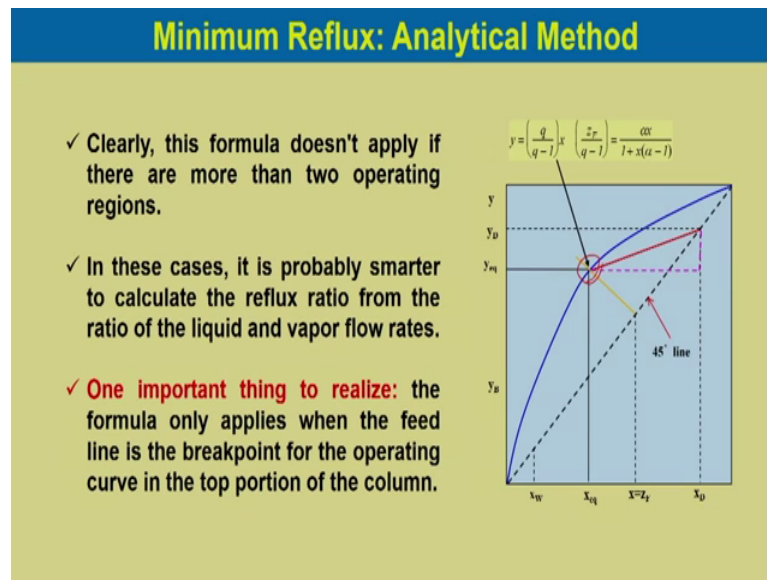
$$\Rightarrow \frac{R_{\min} + 1}{R_{\min}} = \frac{x_D - x_{eq}}{x_D - y_{eq}} \quad \Rightarrow \quad \frac{1}{R_{\min}} = \frac{x_D - x_{eq} - x_D + y_{eq}}{x_D - y_{eq}} = \frac{y_{eq} - x_{eq}}{x_D - y_{eq}}$$

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

So, algebraic rearrangement of the formula as we have already discussed R minimum by R minimum plus 1 would be this and then we can just rearrange this will be x D because that is the point at in a 45 degree diagonal. So, you can replace with x D. So, it would be x D minus y equilibria divided by x D minus x equilibria and after rearrangement we can get 1 by R m would be equal to x D minus x equilibria by x D minus y equilibria minus 1.

And then if you simplify we can get you know R m would be x D minus y equilibria divided by y equilibria minus x equilibria. So, all this you know values are known to us if we substitute we can calculate the minimum reflux ratio.

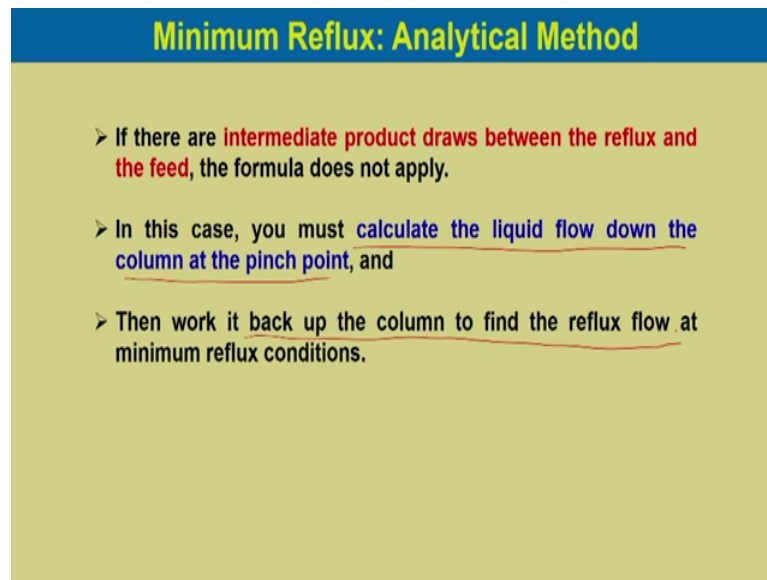
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Clearly this formula does not apply if there are more than two operating region. So, if we have 2 different operating region it will not this formula which we calculated by this analytical formula for which we have used to calculate the minimum reflux ratio does not apply. In these cases it is probably smarter to calculate the reflux ratio from the ratio of the liquid and vapour flow rates. So, if we can know the ratio of the liquid and vapour flow rates of each region, so, then we can calculate the minimum reflux ratio for that case.

One important thing to realize the formula only applies when the feed line is the breakpoint for the operating curve in the top portion of the column. So, this is the formula will apply only when the feed line and the operating line intersects on the equilibrium curve; that means, that is the breakpoint. So, if the breakpoint happened its some other know location this formula does not apply.

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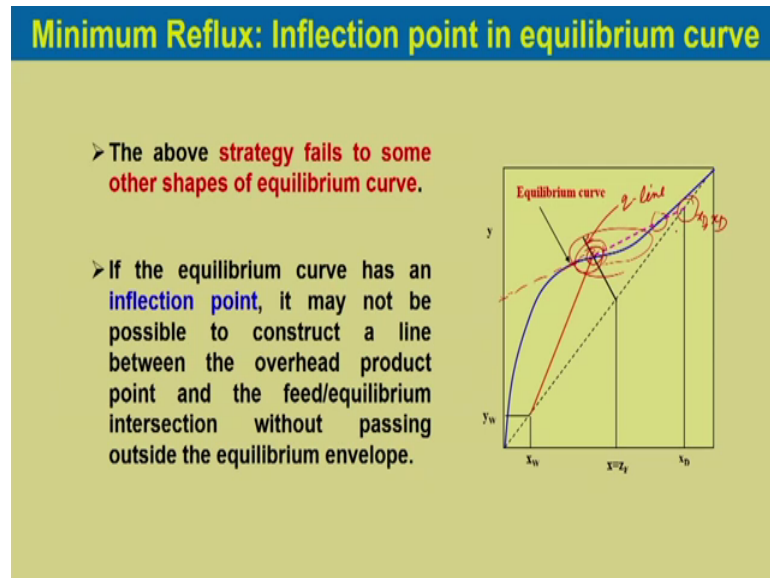
Minimum Reflux: Analytical Method

- If there are **intermediate product draws between the reflux and the feed**, the formula does not apply.
- In this case, you must calculate the liquid flow down the column at the pinch point, and
- Then work it back up the column to find the reflux flow at minimum reflux conditions.

If there are intermediate product draws between the reflux and the feed the formula does not apply. In this case you must calculate the liquid flow down the column at the pinch point and then work it back the up the column to find the reflux flow at the minimum reflux condition.

So, we can if there is you know intermediate product draws between the reflux and the feed point then the formula which we have used cannot be applicable. So, in that case we need to calculate the liquid flow down the column at the pinch point. So, that is below that point of intersection and then we have to work up back to the column to find the reflux flow at the minimum reflux condition.

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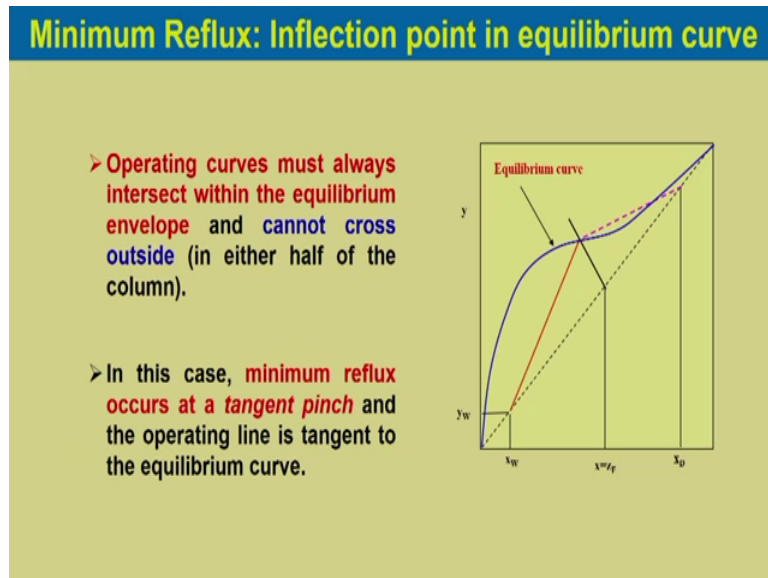


Now, we will discuss the how to calculate the minimum reflux ratio if there is inflection point in the equilibrium curve. So, you can see over here the equilibrium curve there is an inflection point, so, over here in this region.

So, the above strategy fails to some other shapes of the equilibrium curve. So, one of them is the inflection point in the equilibrium curve. So, we cannot apply also to find out the pinch point the way we have done. So, in this case what happens if there is an inflection point on the equilibrium curve it may not be possible to construct a line between the overhead product point and the feed or equilibrium intersection without passing the outside the equilibrium curve?

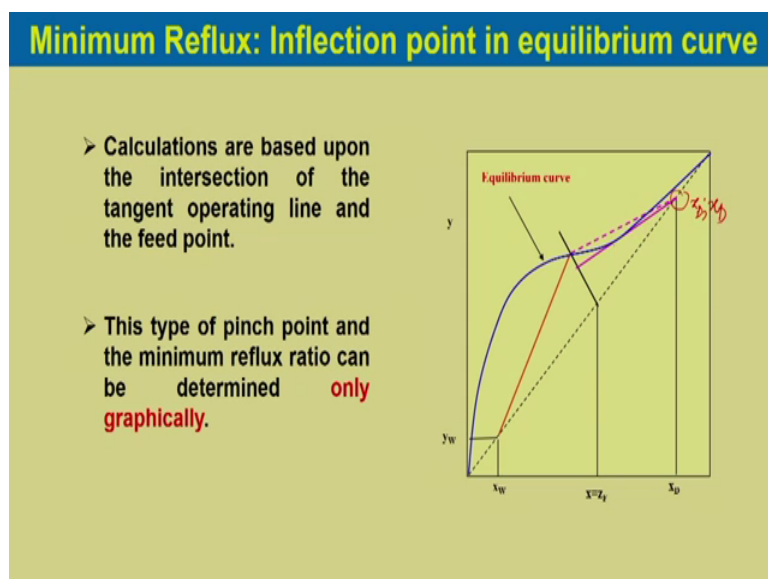
So, you can see this is the point D which is no at x_D x_D and which is the feed line or q line shown over here and q line intercept at this location with the equilibrium curve. Now if we just join this x_D x_D with this you know pinch point. So, in that case you know you would see that it is you know crossing the equilibrium curve at this location and then again intercepting at this point; that means, the operating line for the overhead product or the enriching section has to cross the equilibrium curve.

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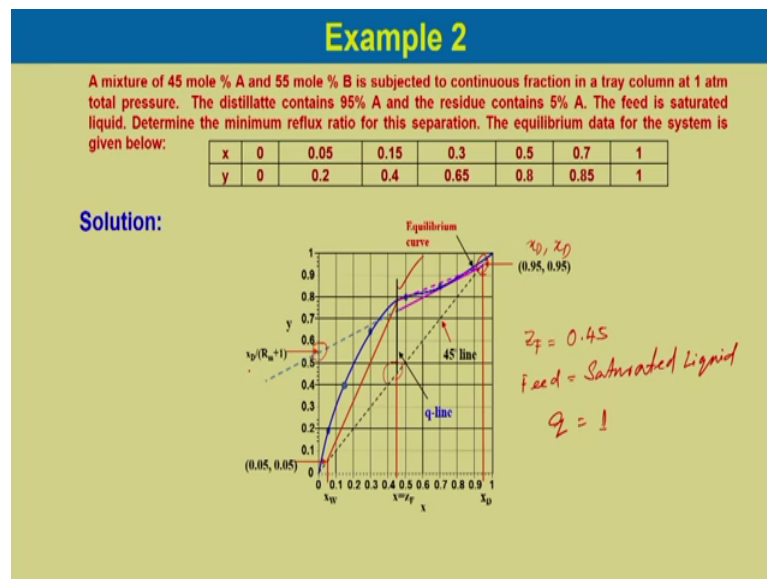
As we know operating curves must always intersect within the equilibrium envelope and cannot cross outside in either half of the column. So, both the stripping section operating line and the enriching section operating line they can never cross the equilibrium curve. So, they must be within the you know below the equilibrium curve in this case the minimum reflux occurs at a tangent pinch and the operating line is tangent to the equilibrium curve.

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So, you can see to calculate the pinch point over here we have to draw the tangent from this point of D which is x_D, x_D and we will draw the tangent on that point and this will give the minimum reflux ratio for the equilibrium curve having inflection point. So, calculation are based upon the intersection of the tangent operating line and the feed point. This type of pinch point and the minimum reflux ratio can only be determined graphically. So, no other formula can be used in this case because it is the tangent, so, we can only do graphically.

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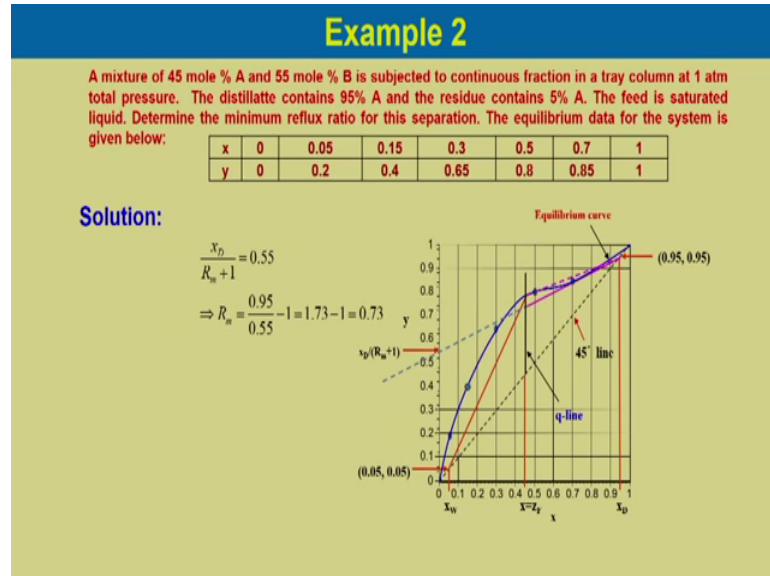


Now let us take an example the similar same examples as we have discussed before, a mixture of 45 mole percent A and 55 mole percent B is subjected to continuous fractionation in a tray column at 1 atmosphere total pressure. The distillate contains 95 percent A and the residue contains 5 percent A the feed is saturated liquid determine the minimum reflux ratio for the separation. The equilibrium data for the system is given below. So, this is the equilibrium data which is given and or for the same case we which we have calculated earlier.

So, to do this what we have to do we know the point x_D, x_D which is over here this is the x_D, x_D point and this is the equilibrium curve which we have drawn here and since the feed is you know having 40 you know 45 mole percent A. So, Z_F is 0.45 and the feed is saturated liquid. So, in this case q is equal to 1, so, it is a vertical line. So, which would be you know vertical it will go vertically. So, this is the q line and then you can

draw the tangent through the equilibrium curve graphically which intersects at this point on the y axis which is x_D by R_m minimum.

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And from this you know you can calculate x_D by $R_m + 1$ is 0.55. So, once you calculate x_D by R_m 0.55 R_m can be calculated which is you know 1.73 minus 1 which is 0.73; show the minimum reflux ratio in this case is 0.73. So, thank you for attending this lecture and we will continue our discussion on the distillation operation in the next class.