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

Distillation-V Lecture - 40 Minimum stages and minimum reflux in multicomponent distillation

Welcome to the 15th lecture of module 5 on Mass Transfer Operation. In this module we are discussing distillation operation, before going to this lecture let us have brief recap on our previous lecture. In our previous lecture we have discussed introduction to the multicomponent distillation.

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And we have discussed the number of tower required for multicomponent distillation. Then we have considered design calculation methods for multicomponent distillation. Another thing we have considered which is important in case of multicomponent distillation that is equilibrium in the multicomponent systems.

We have discussed the bubble point and dew point calculation, multicomponent flash distillation, then we have considered concept of key components; and finally, we have discussed the minimum stages in multicomponent systems.

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Module 5: Lecture 15	
. Minimum Reflux Concept Defermination	
. stages and Reflex -fix a given separation -+ Gilliland plots	
. Feed tray ho cation	

Now in this lecture we will consider minimum reflux, under which we will consider two important topic; its concept, and then determination. Then we will discuss stages and reflux for a given separation, particularly we will consider the Gilliland plots, and finally, we will discuss in this lecture feed tray location.

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Now, minimum stages as in binary distillation, the Fenske equation can also be used for multicomponent systems to determine the minimum number of stages for a given separation at total reflux. Equation can be written for any two components in general apply the equation to the key components. So, better to use for the key components, the Fenske equation we can write using the key components and solve for the number of stages. So, the equation Fenske equation for multicomponent systems we can write N minimum is equal to log x LD into D divided by x HD into D into x HW into W divided by x LW into W divided by log alpha L average.

x LD is the mol fractions of light key in distillate, x LW is the mol fractions of the light key in bottoms x HD is the mol fraction of heavy key in distillate, x HW is the mol fraction of heavy key in bottoms.

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Alpha L average that is the mean relative volatility which is used, when alpha is not constant normally a geometric mean value is the best options to choose. Following equations may be used alpha L average would be equal to root over alpha LD into alpha LW which is equation 18. Once n minimum has been established that is minimum number of plates the same equation can be rearranged to determine spilt of the other components in the mixture as given below that is, x i D into D divided by x i W into W would be equal to alpha L average to the power N mean minimum number of plates into x HD into D divided by x HW into W this is equation 19 by which we can determine the spilt of the other component in the mixture.

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Example 3

A mixture of hydrocarbons containing 32 mole% n-hexane, 38 mole% n-heptane and 30 mole% n-octane is to be distilled to obtain a distillate product with 0.05 mole fraction of n-heptane and a bottom product with 0.05 mole fraction of n-hexane. The column operating pressure is 1.2 atm. 40% of the feed is to be vaporized. Calculate the product composition and the minimum number of ideal plates.

Now, let us consider an example to calculate the minimum number of ideal plates. A mixture of hydrocarbons containing 32 mole percent n-hexane, 38 mole percent n heptanes, and 30 mole percent n-octane is to be distilled to obtain a distillate product with 0.05 mole fractions of n-heptane and a bottom products with 0.05 mole fraction of the hexane. The column is operating at 1.2 atmosphere pressure; 40 percent of the feed is to be vaporized. Now we need to calculate the product composition and the minimum number of ideal plates.

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Example 3: Solution

A mixture of hydrocarbons containing 32 mole% n-hexane, 38 mole% n-heptane and 30 mole% n-octane is to be distilled to obtain a distillate product with 0.05 mole fraction of n-heptane and a bottom product with 0.05 mole fraction of n-hexane. The column operating pressure is 1.2 atm. 40% of the feed is to be vaporized. Calculate the product composition and the minimum number of ideal plates.

Solution: Light leg (LK) Heavy keg(HK) Heavy non teg (HNK) n=hxane n- Reptane n- octane Assumption : (1) No n- octane in INE distillate (2) 0.95 mole fraction of n-bexane is in the distillate Solution: Basis: 100 miles/h of the feed rate

Now, here for the first distillation we have to define the key components and the non key components. So, let us define the key components, so one is light key and we can call it LK, another is heavy key we can call it as HK and the third one is heavy non key and we can call it as HNK.

So, this we can consider as n-heptanes, n-hexane this is n-heptane and this is n-octane. Now assumption is that, no the first assumption is no n-octane in the distillate. And second assumption is that 0.95 mole fraction of n hexane is in the distillate. Now to solve this problem let us assume basis or let us consider basis that is 100 moles per hour of the feed rate.

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Now let us do the overall mass balance, that is F is equal to D plus W which is as we have considered the basis is 100 moles, so this would be equal to 100. Now if we do hexane mole balance we can write F x F would be equal to D x D plus W x W and if we substitute this would be 100 into 0.32 which is equal to 0.95 into D plus W from here we can write W is equal to 100 minus D. So, we can write 100 minus D into 0.05. So, this would be equal to 32 is equal to 0.95 D plus 5 minus 0.05 D. And from here we can write 0.9 D is equal to 32 minus 5 which is 27.

So, from here we can write D would be equal to 27 divided by 0.9 which is equal to 30 mole per hour. Now from the overall mass balance we can calculate W would be equal to 100 minus D which is equal to 100 minus 30 which is equal to 70 mole per hour.

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A mixture 30 mole% fraction o column o product c	of hydroca n-octane f n-heptane perating pre omposition	arbons co is to be and a b essure is and the r	ontaining a distilled to ottom pro 1.2 atm. 40 minimum r	32 mole to obtai duct wi 0% of th number	9% n-hexa in a disti ith 0.05 m he feed is t of ideal pl	ine, 38 mo llate prod ole fractio to be vapo lates.	ole% n-heptane luct with 0.05 n on of n-hexane. orized. Calculate	and nole The the
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Now the composition of the bottom product can be calculated using the conditions given all normal octane, all but 0.05 D of the n-heptanes, 0.05 mole fraction of n-hexane. So, now we can calculate the compositions let us develop the table; so, keys components F x F, D x D, x D, W x W, x W and K i at 102.2 degree centigrade and 1.2 atmosphere pressure.

So, what are the key components light key, heavy key and heavy non key components is n C 6, n C 7, n C 8. As per the feed conditions given feed compositions this is 32 this is 38 and this is 30. So, $D \times D$ we have calculated from the material balance equation, which is 28.5 for the light key and then for the heavy key it is 1.5.

Since the heavy non key is only present in the bottom. So, its compositions in the distillate would be 0 and we know D is equal to 30. Now x D is 0.95 that is for n C 6 for the heavy key it is that is n C 7 n-heptane is 0.05 and this would be 0. this n C 8 in the bottom will be completely present which was in the feed which is 30 and W x W from the material balance you can calculate which is 3.5 and this is 36.5 so total W would be 70.

Now, from here we can calculate if we know W x W and W then we can calculate x w. So, x W would be 0.05 for n hexane; for n-heptane it would be 0.521 and for n-octane it would be 0.429. Now as we have seen the K i values at this temperature and pressure for this components is 2.08 and for this is 0.92 and for this component is 0.42.

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Example 3: Solution A mixture of hydrocarbons containing 32 mole% n-hexane, 38 mole% n-heptane and 30 mole% n-octane is to be distilled to obtain a distillate product with 0.05 mole fraction of n-heptane and a bottom product with 0.05 mole fraction of n-hexane. The column operating pressure is 1.2 atm. 40% of the feed is to be vaporized. Calculate the product composition and the minimum number of ideal plates. Solution: (1) Use Fonske egge to obtain minimum number of plates (2) The relative volatilities of the Ught key to the heavy key is the the ratio of Their (2) It relative (3) I/e k-value at the flash temp were obtained in the previous examp. Vir.Ax = 2:08/0.92 = 2:26 1.9 [(XLOD/XLOD) × (XNNW/XLNW)] 19[0.95/0.05)(0.05/0.5U)]

Now, use the Fenske equation to obtain the minimum number of plates, the relative volatility of the light key to the heavy key is the ratio of their K values. So, the relative volatility of the light key to the heavy key is the ratio of their K values; the K values at the flash temperature were obtained, in the previous example which was shown in the last table. The alpha values that is alpha LK HK we can write 2.08 divided by 0.92 that is the heavy key so it would be about 2.26.

Now, if we write N minimum which is equal to log x LD into D divided by x HD into D into x HW into W divided by x LW into W divided by log alpha L average. So, which is equal to log x LD by x LW divided by x HD by x HW divided by log alpha L average.

Now, if we substitute the values, it would be 0.95 divided by 0.05 divided log this divided by 0.05 by 0.521 divided by log 2.26 this would be equal to log 19 by 0.096 divided by log 2.26 which is equal to 5.288 divided by 0.815 which will give you 6.5.

So, this n minimum it includes the re boiler which is 6.5 so minimum number of ideal plates would be 6.5 or if we just subtract 1 that is the re boiler it would be 2 point it would be 5.5 number of ideal plates. This can be refined or more accurate estimates we can obtain if we take the geometric mean in place of alpha LK, HK. So, in place of this if we consider geometric mean between the relative volatility at the top and the bottom then we can obtain much accurate data for the minimum number of plates.

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Now, we will consider minimum reflux, minimum reflux calculation are based on invariant zones around the feed where the compositions stop changing. These are similar to the pinch point idea which is used for the binary columns or binary distillation. One way to determine an approximate minimum reflux ratio is to represent a multicomponent mixture as a pseudo binary system. This is done by creating a hypothetical feed made up of the two key components only then a McCabe-Thiele type construction can be used to determine the pinch and the minimum reflux.

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However the accuracy of the approximation depends heavily on how large a portion of the material is made up of the keys; considering this limitation and the amount of work required to recast the calculation, other methods are usually preferred.

As in the case of binary distillation the minimum reflux ratio that is R m is that reflux ratio which will require an infinite number of trays for a given separation of the key components. For binary distillation only one "pinch point" occurs where the number of steps becomes infinite and that is usually at the feed tray.

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For multicomponent distillations two pinch points or zones of constant composition occurs. One in the section above the feed plate and another below the feed tray; the rigorous plate by plate stepwise procedure for calculating R minimum is trial and error and can be extremely tedious for hand calculations. Often minimum reflux is estimated using Underwood method.

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Underwood method assumes equimolar overflow and defines relative volatility for each component relative to the some reference components, usually the heavy key that is considered as reference component. And then the relative volatility for each components is defined. R m uses constant average alpha values and assumes constant flows in both the section of the tower, both in rectifying as well as striping section. This method provides a reasonably accurate value.

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The two equations to be solved in order to determine the minimum reflux ratios which are given below, 1 minus q is equal to summation over alpha i x i F divided by alpha i minus theta. Another one is R m plus 1 would be equal to summation over alpha i x i D divided by alpha i minus theta. Here q is the fractions and theta is a parameter, the values of x i D for each component in the distillate in equation 21 are supposed to be the values at the minimum reflux. As an approximation the values obtained using the Fenske total reflux equation can be used.

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Since each alpha i may vary with temperature the average value of alpha i to use in the preceding equations is approximated by using alpha i at average temperature considering the top and bottom of the tower.

Now, to solve for R m the value of theta n equation 20 is first obtained by trial and error. So, this is equation 20 it is theta is obtained by trial and error in this case. And then that is put in the equation next equation that is equation 21 to calculate R m, this value of theta lies between alpha value of the light key and the alpha value of the heavy key which is 1.

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Using this value of theta in equation 21 the value of R m is obtained directly. So, R m equation is here and we can obtain R m from this equation, the Underwood method assumes constant molar overflow and relatively constant, relative volatility. These are the two major assumptions of the Underwood method.

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So, let us consider an example to solve the minimum reflux ratio problems, a mixture of hydrocarbon containing 5 mole percent n-pentane 30 mole percent hexane and 55 mole percent n-heptanes, 10 mole percent n octane is to be distilled at one atmosphere pressure

to recover 95 percent hexane and 2 percent n-heptane in the distillate. The liquid feed is at its boiling point; now calculate the minimum reflux ratio for this operation. The vapour pressure and the temperature data can be obtained from this figure.

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Example 4: Solution A mixture of hydrocarbons containing 5 mole% n-pentane, 30 mole% n-hexane, 55 mole% n-heptane and 10 mole% n-octane is to be distilled at 1 atm to recover 95% nhexane and 2% n-heptane in the distillate. The liquid feed is at its boiling point. Calculate the minimum reflux ratio for this operation. The vapour pressure vs. temperature data are given in the Figure. Solution: The light key = n- hexane The Azary Key = n- heptane They components fre sufficiently different in be distrib volatility Assume = 100 moles of Fred Summarize the known information in a Tabular form

So, let us start the solution of these problems. Now we need to define the key components; one is the light key which is n-hexane and the heavy key which is n-heptane. The other components are sufficiently different in volatility to be distributed. Now assume 100 moles of feed, we can summarize the known information in a tabular format.

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So, let us form the table, so this is keys then components x F, F x F, D x D, x D, W x W, and x W. So, keys are light key and heavy key first what are the components you will write? n-pentane C 5 n-hexane C 6, n-heptane C 7 and n-octane C 8. Now we have defined this is the light key and this is the heavy key now, composition of the feed are given that is 0.05. 0.30. 0.55 and 0.10.

So, we can obtain this is in feed we have considered 100 moles of feed. So, then F x F would be 5 this would be 30 this would be 55 and this would be 10. Now D x D would be 5 this would be 0 because this is not distributed and this would be 28.5 and this would be 1.1. Now from this the total D is equal to 34.6 and then we can calculate the values of x D which is 0.145 this is 0.823 and this is 0.032 and these would be 0. In the bottoms this C 5 being a lighter components than the light key it will not be distributed in the bottoms or it will not present in the bottoms.

So, it would be 0 so its mole fractions would be 0 and this would be 1.5 this would be 53.9 and this would be 10. So, total W would be 65.4 and then we can calculate the x W which is 0.023 0.824 and 0.153. So, we need to know the fractional recovery equations that is F R D is equal to fractional recovery of light key, then x D D into x D would be is equal to F R D light key into F x light key. So, this is the equations we will use later.

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Now, let us assume the bubble point which is equal to 80 degree Centigrade now we can calculate different K values, component then K i at 80 degree Centigrade, then K x F. So, we can just note it down the components that is n C 5, n C 6, n C 7, n C 8.

Now K i at 80 degree Centigrade we can get from this we can obtain as 3.62 this is 1.39 0.56 and 0.23. So, once we obtain the K values at 80 degree Centigrade we can calculate K x F values which is 0.181, 0.417 0.308 and 0.023.

So, if we sum them up it would be 0.929 so this is less than 1. So, let us assume a little higher temperature assume higher temperature say 85 degree Centigrade. We can calculate K i at 85 degree C then K i x F and we can see the values. So, K i at 85 degree Centigrade would be 4.1 1.7 0.68 and 0.28 now if we multiply with x F it would be 0.205 this would be 0.510 this would be 0.374 and this would be 0.028.

So, the sum of all these K i x F would be 1.117. So, which is greater than 1 the bubble point temperature is in between 80 degree Centigrade and 85 degree Centigrade say 82 degree Centigrade. Now, let us calculate K i at 82 degree Centigrade which is 3.8 n C 5 K values for n C 5 is 3.8 for n C 6 is 1.5 and for n C 7 is 0.60 and for C 8 this 0.25. Now K i x i would be 0.19 0.45 0.33 and 0.025. So, if we sum them up it would be 0.995 hence. So, this is close to 1 hence the bubble point temperature would be 82 degree Centigrade.

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Now, by using Underwood method K values at 82 degree Centigrade are converted to relative volatility. And the theta values between one and is found by trial since q is equal to 1 as it is given the feed at its bubble point that is feed at its bubble point. So, we can calculate summation over alpha i x i F divided by alpha i minus theta would be equal to 1 minus q, which is equal to 1 minus 1 which is equal to 0.

Now, let us calculate our alpha values and obtain the table components alpha i x i F summation over alpha i x i F divided by alpha i minus theta. So, component is n C 5 n C 6 n C 7 n C 8. So, alpha values are 6.33, 2.5 for n C 6, for n C 7 1.0 for n C 8 is 0.42, x i F is 0.05 0.30 0.55 0.10.

So, by trial and error if we consider theta is equal to 1.62 by several trial you could see that it would be 0.0672, this would be 0.8533, this would be minus 0.8871 and this would be 0.035. So, this would be minus 0.0026. So, theta is 1.62 by trial and error.

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A mixture mole% n-h hexane ar Calculate	of hydrocarbons containing eptane and 10 mole% n-octar d 2% n-heptane in the disti the minimum reflux ratio fo	5 mole% n-pentane, 3 ne is to be distilled at 7 llate. The liquid feed or this operation. The	30 mole% n-hexane, 5 1 atm to recover 95% r is at its boiling poin e vapour pressure vs
temperatu	e data are given in the Figure		
Solution			
Rm	$= \overline{\sum_{i=1}^{n} \frac{\alpha_i x_i}{\alpha_i - \theta_i}} - 1$		a chain
U	6.33 × 0.145 + 2	SO X0.823 1× 0.022 -1	20 20
	= 6.33-1.62 2.5	0-1.64	10 / 10 / 10 / 10 / 10 / 10 / 10 / 10 /
	= 0.195+2.338-0.03	2-1	Or Orun
	= 2.585-1 = 1.303	٧ V	
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Now as we know R m is equal to summation over alpha i x D by alpha i minus theta minus 1. So, now, if we substitute it is 6.33 into 0.145 which we have obtained earlier divided by 6.33 minus 1.62 this is for pentane plus 2.50 into 0.823 divided by 2.50 minus 1.62, this is for hexane plus 1 into 0.032 divided by 1 minus 1.62 minus 1. So, you have not considered here the octane because it will not present in the distillate.

So, this would give 0.195 plus 2.338 minus 0.052 minus 1 and this would be equal to 2.585 minus 1 which is equal to 1.585. So, this way we can calculate the minimum reflux ratio R m using Underwood method.

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Now, stages reflux for a given separation number of stages at operating reflux ratio Gililand plots can be used to find the actual stages required for a given rate or vice versa.

The plot relates two variables one is X is equal to R minus R m divided by R plus 1 R is the actual reflux ratio, minus minimum reflux ratio divided by R plus 1 and Y is equal to N minus N minimum divided by N plus 1. N is the number of trace and N min is the minimum number of trace; X ranges from 0 at R is equal to R min to infinity at N is equal to N min. So, when R is equal to R min then X would be you can see from this equation 22 X would be 0; and when N is equal to N min in that case it would be infinity. And in this case the Y ranges from 1 at R is equal to R min to 0 at N is equal to N min.

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Now feed tray location, Kirkbride has devised an approximate method to estimate the number of theoretical trays above and below the feed, which can be used to estimate the feed tray location. This empirical relation is as follows log N R divided by N S is equal to 0.206 log x HF divided by x LF into W by D into x LF LW divided by x HD square. Here, N R is the number of theoretical stages above the feed tray and N S is the number of theoretical stages below the feed tray.

Thank you very much for your patience hearing and we will finish our lecture for the mass transfer operation course in the next lecture.

Thank you.