Mass Transfer Operations I

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Module - 5
Distillation
Lecture - 4
Batch and Steam Distillation

Welcome to the fourth lecture of module 5. Here, we are discussing distillation operations.

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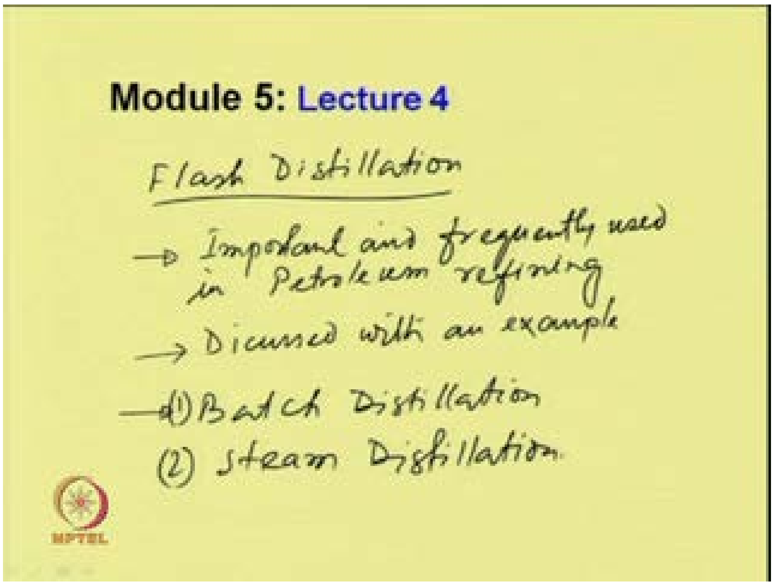

Module 5: Lecture 4

Flash Distillation

→ Important and frequently used in Petroleum refining

→ Dicussed with an example

→ (1) Batch Distillation

(2) Steam Distillation

So, let us have recap of our earlier lectures. In lecture 3, we discussed flash distillation, and this flash distillation is very important particularly in petroleum refining; important and frequently used in petroleum refining, and we have also taken the example of a mixture of n-hexane and n-heptane. Today, we will discuss two more topics; one is batch distillation and second is steam distillation.

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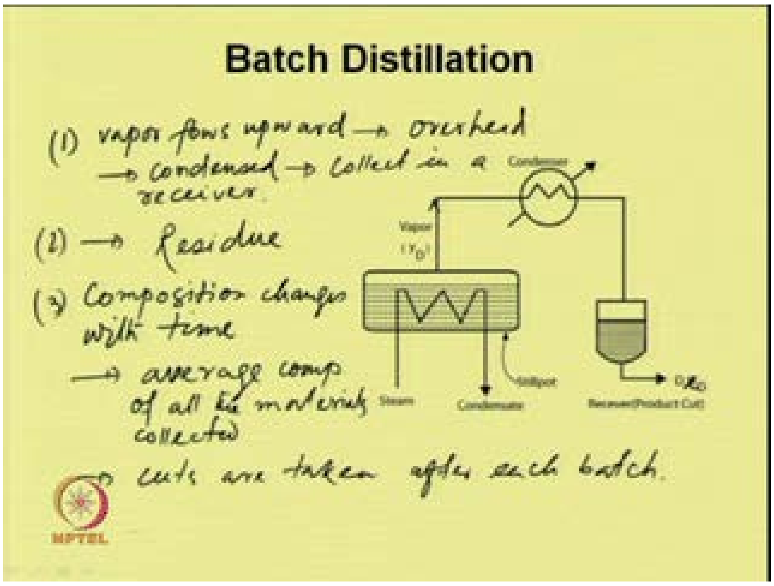

Batch Distillation

(1) vapor flows upward → overhead
→ condensed → collect in a receiver.

(2) → Residue

(3) Composition changes with time
→ average comp of all the materials collected
→ cuts are taken after each batch.

So, let us consider batch distillation. This is a simple distillation, where a tank is charged with the feet material, and which is fitted with the steam jacket, and it heated with a steam coils. What happens? The vapor flows upward; then it goes to overhead, it is then condensed, and then it is collected with a receiver, and then the remaining material which is left in the tank is known as the residue. So, the composition which is collected in the receiver, composition changes with time; this is small x D. The composition which is obtained at the receiver is the average compositions of all the materials collected.

So this is a batch process, and this can be switched off every time as the operation is done, and the cuts are taken out. So, this batch process is inherently; this is an unsteady state process or we can say it is a dynamic process.

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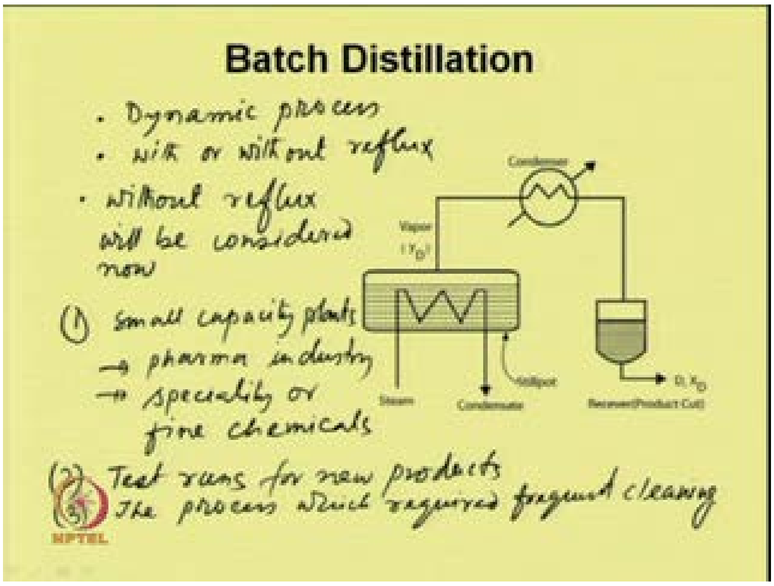

Batch Distillation

- Dynamic process
- with or without reflux
- without reflux will be considered now

(1) small capacity plants
→ pharma industry
→ speciality or fine chemicals

(2) Test runs for new products

(3) The process which required frequent cleaning

So this cannot be modeled at steady state say dynamic process, and this operation, it can be done with or without reflux. Two methods can be followed with or without reflux. When we consider with reflux, then we can use the total reflux or we can vary the reflux as well. Here, we will consider now without reflux. So, this batch distillation operation, it is very common in small capacities plant like pharmaceuticals, pharma industry, specialty chemicals, or fine chemicals, and this can be also used for test runs for new products, or this can be used for the continuous process where the frequent cleaning is required. In that case, this operations can be done; the process which required frequent cleaning.

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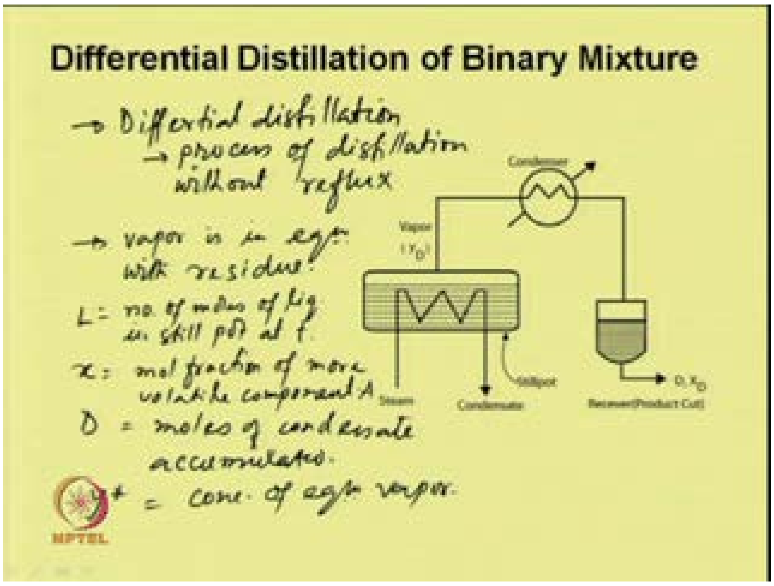

Differential Distillation of Binary Mixture

→ Differential distillation

→ process of distillation without reflux

→ vapor is in eqm. with residue.

L = no. of moles of liq in still pot at t.

x = mol fraction of more volatile component A

D = moles of condensate accumulated.

* = conc. of eqm. vapor.

Let us consider differential distillation of a binary mixture. So, we will consider binary mixture a and b, and we will consider without reflux. When the batch distillation without reflux is operated, the process is called differential distillation. Since there is no reflux, the vapor which is producing from the still is assumed to be in equilibrium with that liquid, which is the residue; vapor is in equilibrium with residue.

Now, let us take L is the number of moles of liquid in the still pot at any time t, and small x is the mole fraction of more volatile component say A, and D is the moles of condensate accumulated, y star is equal to concentration of the equilibrium vapor.

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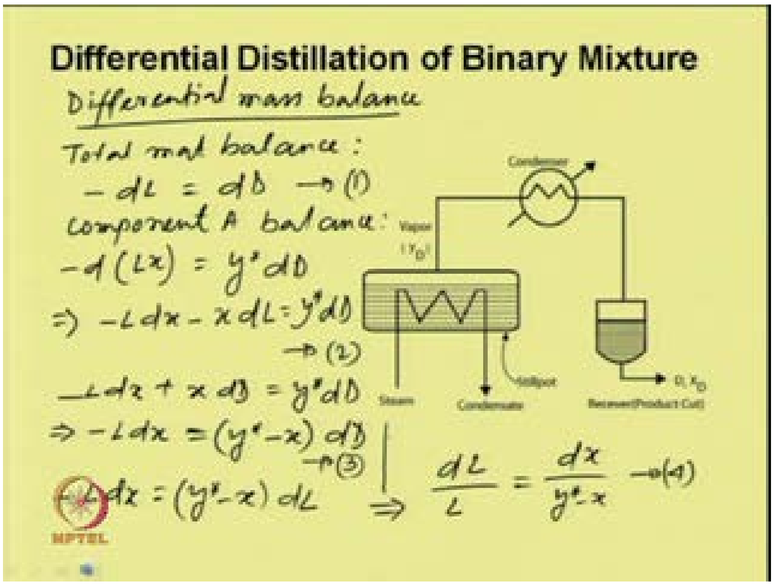

Differential Distillation of Binary Mixture

Differential mass balance

Total mat balance:

$-dL = dD \rightarrow (1)$

component A balance:

$-d(Lx) = y^* dD$

$\Rightarrow -L\,dx - x\,dL = y^* dD \rightarrow (2)$

$-L\,dx + x\,dD = y^* dD$

$\Rightarrow -L\,dx = (y^* - x)\,dD \rightarrow (3)$

$-L\,dx = (y^* - x)\,dL \Rightarrow \frac{dL}{L} = \frac{dx}{y^* - x} \rightarrow (4)$

Now if we do the total material balance, the differential mass balance we will do. First, we will do the total material balance is minus dL is dD; say this is equation 1. Now component A balance we can write minus d of Lx would be equal to y star dD.

So, from this we can write minus L dx minus x dL would be equal to y star dD. So, this is equation 2. Now if you use equation 1 in place of equation 2, we can write minus L dx plus x dD is equal to y star dD. So, from this we can write minus L dx would be equal to y star minus x dD; this is equation 3. Suppose and if we use equation 1 in this case; so, it will be minus L dx would be y star minus x into dL. So, from this we can write dL by L is equal to dx by y star minus x. So, this is equation 4.

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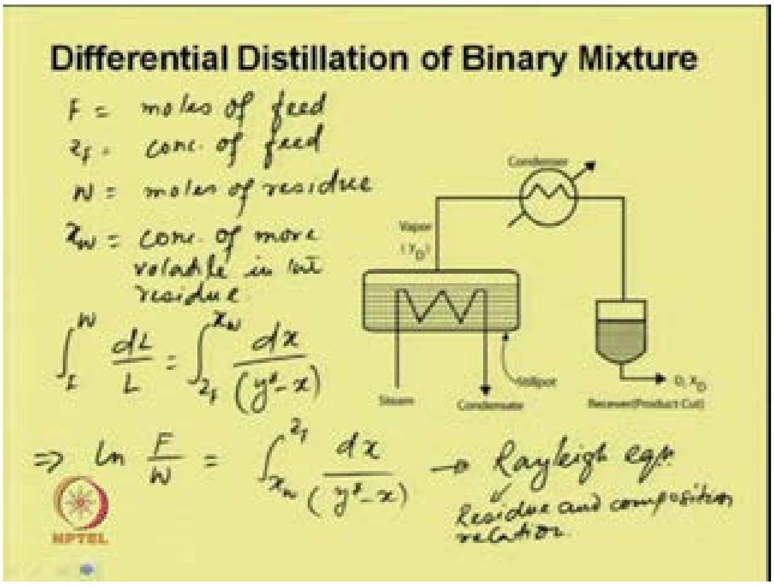

Differential Distillation of Binary Mixture

F = moles of feed

z_f = conc. of feed

W = moles of residue

x_w = conc. of more volatile in the residue

$$\int_F^W \frac{dL}{L} = \int_{z_f}^{x_w} \frac{dx}{(y^* - x)}$$

$$\Rightarrow \ln \frac{F}{W} = \int_{x_w}^{z_f} \frac{dx}{(y^* - x)} \rightarrow \text{Rayleigh eqn.}$$

Residue and composition relation

Now if we consider F is equal to a moles of feed, Z F concentration of feed, and W is the moles of residue, and also x w is the concentration of more volatile in the residue. Then we can write from equation 4; this equation we can write, integral F to W dL by L would be equal to integral Z F to x w dx divided by y star minus x. So, from this we can write ln F by w would be equal to integral x w two Z F dx by y star minus x. So, this equation is known as the Rayleigh equation, and this relates with the residue and the composition relation.

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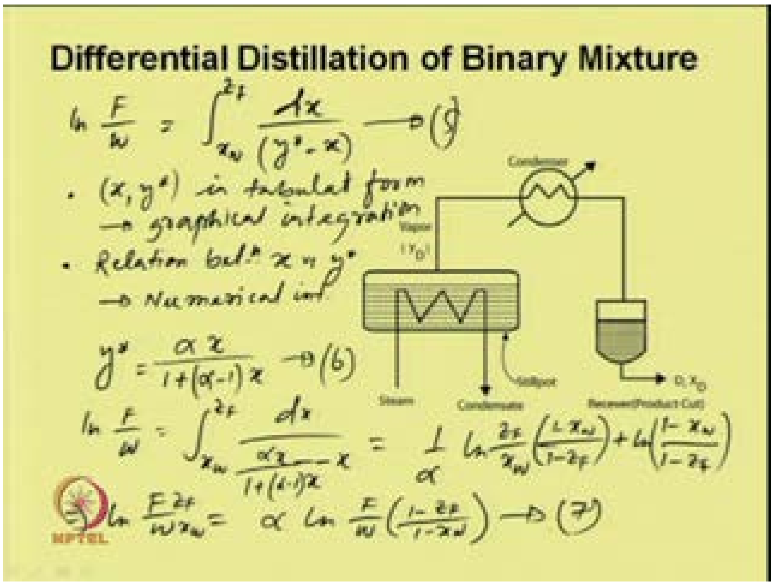

Differential Distillation of Binary Mixture

$$\ln \frac{F}{W} = \int_{x_w}^{z_f} \frac{dx}{(y^* - x)} \rightarrow (5)$$

- (x, y^*) in tabulat form → graphical integration
- Relation betn x & y^* → Numerical int.

$$y^* = \frac{\alpha x}{1 + (\alpha - 1)x} \rightarrow (6)$$

$$\ln \frac{F}{W} = \int_{x_w}^{z_f} \frac{dx}{\frac{\alpha x}{1 + (\alpha - 1)x} - x} = \frac{1}{\alpha} \ln \frac{z_f}{x_w} \left(\frac{1 - x_w}{1 - z_f} \right) + \ln \left(\frac{1 - x_w}{1 - z_f} \right)$$

$$\ln \frac{F z_f}{W x_w} = \alpha \ln \frac{F}{W} \left(\frac{1 - z_f}{1 - x_w} \right) \rightarrow (7)$$

So, ln F by w is equal to integral x w to Z F dx by y star minus x. So, in this equations the right hand side if we have the equilibrium data in tabular form, suppose x and y star; this data are available in tabular form, equilibrium data. Then graphical integration is possible; graphical integration can be done. But if we have the relations between x and y star, then numerical integration is possible; numerical integration we can do. Suppose, if we take the relations y star is equal to alpha x divided by 1 plus alpha minus 1 into x.

So, if we use in this Rayleigh equations. So, it will be ln F by W would be equal to integral x w to Z F dx divided by alpha x by 1 plus alpha minus 1 into x minus x, which we can write 1 by alpha ln Z F by x w into 1 minus x w divided by 1 minus Z F plus ln 1 minus x w by 1 minus Z F. So, this can be written in more convenient form is ln F Z F by W x w would be equal to alpha ln F by W 1 minus Z F by 1 minus x w. Say this is equation 5, and this is equation 6, and this is the equation 7.

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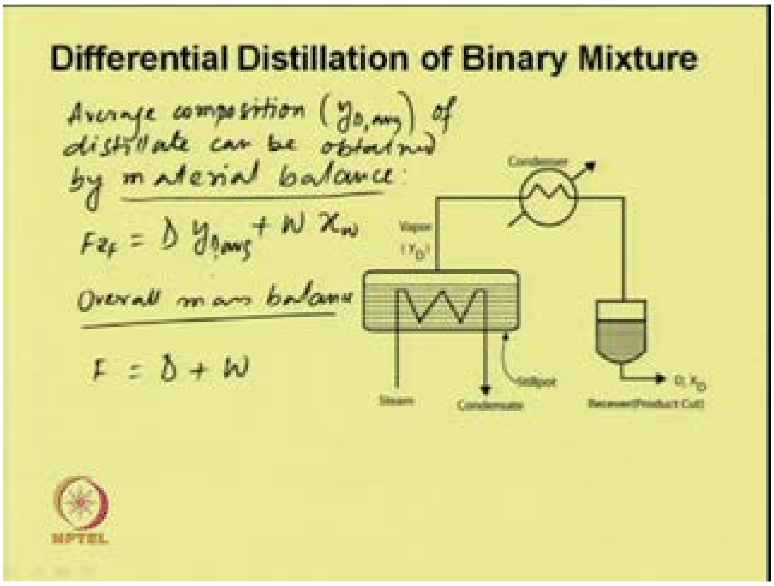

Differential Distillation of Binary Mixture

Average composition $(y_{D,avg})$ of distillate can be obtained by material balance:

$$Fz_F = D\,y_{D,avg} + W\,x_W$$

Overall mass balance

$$F = D + W$$

Now, the average composition y D average; so, this is y D. This can be obtained average composition, that is, y D average of accumulated distillate can be obtained by material balance. So, if we do the little balance F Z F is equal to D y D average plus W x w, and then we can do the overall mass balance, F is equal to D plus W. So, from this, we can calculate y D average.

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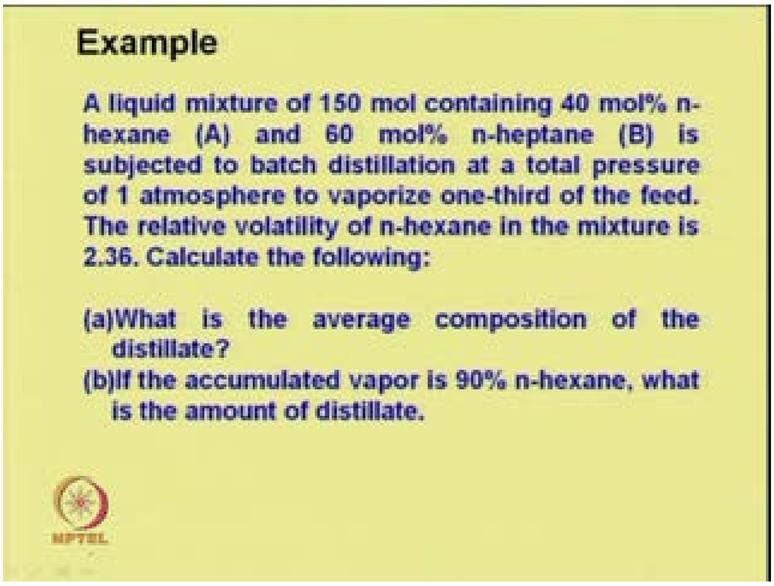

Example

A liquid mixture of 150 mol containing 40 mol% n-hexane (A) and 60 mol% n-heptane (B) is subjected to batch distillation at a total pressure of 1 atmosphere to vaporize one-third of the feed. The relative volatility of n-hexane in the mixture is 2.36. Calculate the following:

(a)What is the average composition of the distillate?

(b)If the accumulated vapor is 90% n-hexane, what is the amount of distillate.

Now, let us take an example to see how we can calculate the average concentration. A liquid mixture of 150 mol containing 40 mol percent n-hexane and 60 mol percent n-heptane, which is, B is subjected to batch distillation at a total pressure of 1 atmosphere to vaporize one-third of the feed. The relative volatility of n-hexane in the mixture is 2.36. Calculate the following: What is the average composition of the distillate? If the accumulated vapor is 90 percent n-hexane, what is the amount of distillate?

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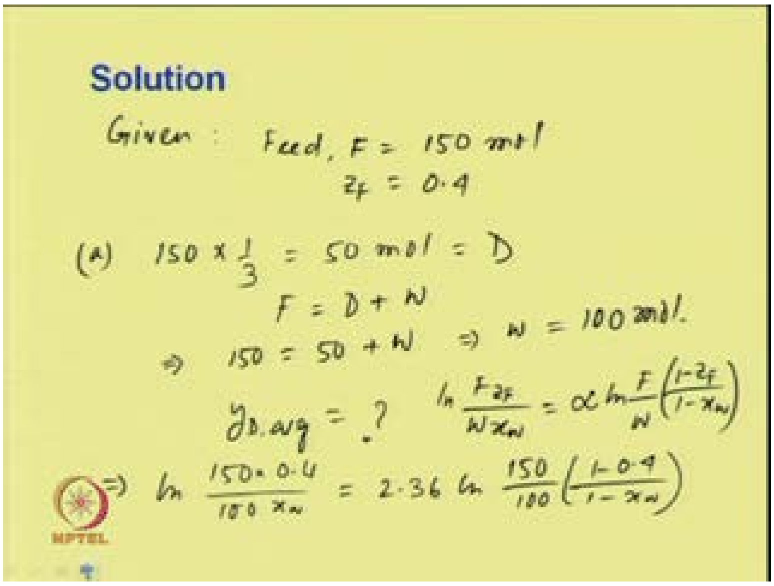

Solution

Given : Feed, $F = 150$ mol

$z_F = 0.4$

(a) $150 \times \frac{1}{3} = 50 \text{ mol} = D$

$F = D + W$

$\Rightarrow 150 = 50 + W \Rightarrow W = 100$ mol.

$y_{D,avg} = ?$ $\quad \ln \frac{F z_F}{W x_W} = \alpha \ln \frac{F}{W}\left(\frac{1-z_F}{1-x_W}\right)$

$\Rightarrow \ln \frac{150 \times 0.4}{100 \, x_W} = 2.36 \ln \frac{150}{100}\left(\frac{1-0.4}{1-x_W}\right)$

So, first we will try to calculate the average compositions of the distillate. It is given the feed, which is F is equal to 150 mol, and the composition Z F which is 0.4; 40 mol percent n-hexane, and for problem 1, it is given that to vaporize one-third of the feed. So, feed is vaporized one-third; that means 150 into one-third. So, 50 mol is vaporized which is D. From the total material balance if we look, F is equal to D plus W. So, 150 is equal to 50 plus W; so, the W is 100 mol.

Now, you have to calculate y D average. So, let us use Rayleigh equations, which is ln F Z F by W x w is equal to alpha ln F by W 1 minus Z F by 1 minus x w. Now putting the values; if we put the values ln 150 into 0.4 divided by 100 x w is equal to 2.36 ln 150 by 100 1 minus 0.4 divide by 1 minus x w.

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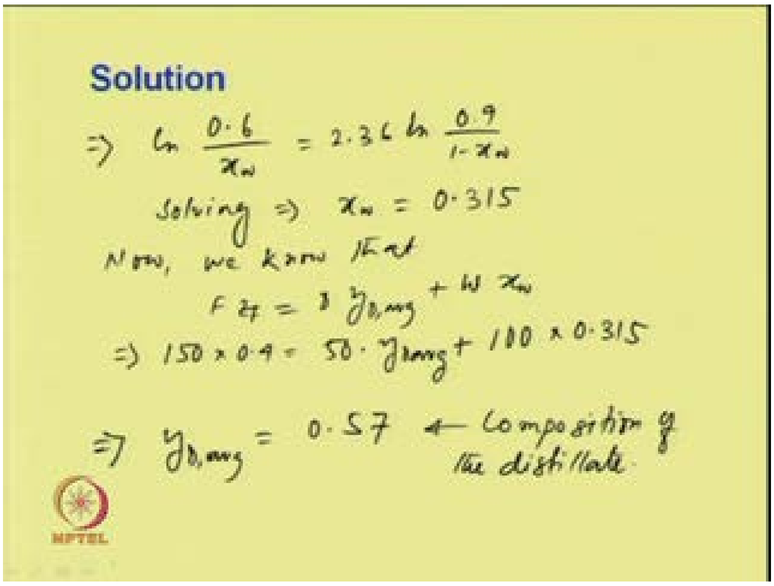

Solution

$$\Rightarrow \ln \frac{0.6}{x_w} = 2.36 \ln \frac{0.9}{1-x_w}$$

Solving $\Rightarrow x_w = 0.315$

Now, we know that

$$F z_F = D\, y_{D,avg} + W x_w$$

$$\Rightarrow 150 \times 0.4 = 50 \cdot y_{D,avg} + 100 \times 0.315$$

$$\Rightarrow y_{D,avg} = 0.57 \leftarrow \text{composition of the distillate}$$

So, this we can write ln 0.6 by x w would be equal to 2.36 ln 0.9 by 1 minus x w. So, after solving we can do trial and error method guessing x w from both sides. So, we can get x w is equal to 0.315. Now we know that, F Z F is equal to D y D average plus W x w. So, then we can use 150 into 0.4 would be equal to 50 into y D average plus 100 into 0.315. So, from this we can obtain y D average is equal to 0.57. So, this is the composition of the distillate.

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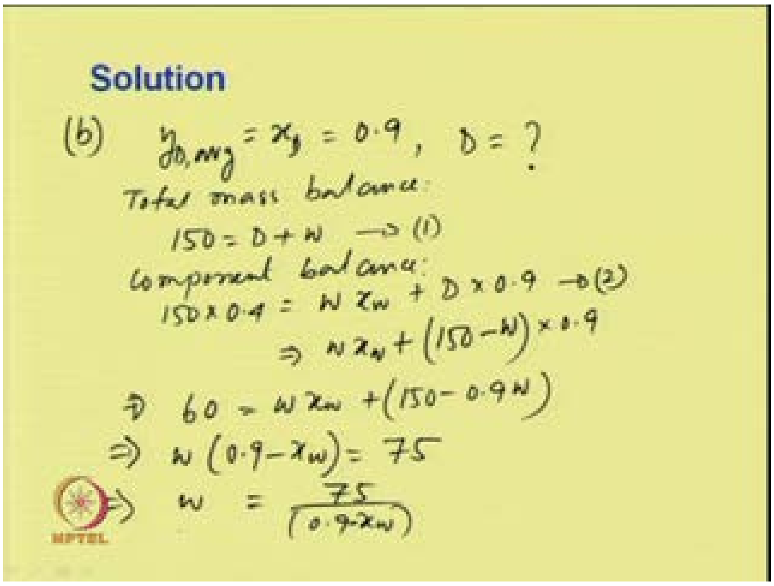

Solution

(b) $y_{D,avg} = x_D = 0.9$, $D = ?$

Total mass balance:

$150 = D + W \longrightarrow (1)$

Component balance:

$150 \times 0.4 = W x_W + D \times 0.9 \longrightarrow (2)$

$\Rightarrow W x_W + (150 - W) \times 0.9$

$\Rightarrow 60 = W x_W + (150 - 0.9W)$

$\Rightarrow W(0.9 - x_W) = 75$

$\Rightarrow W = \frac{75}{(0.9 - x_W)}$

Now, let us do for second part b. So, it is given y D average is equal to x D which is 0.9, and we have to calculate what is the value of distillate; amount of distillate? We know from the total material balance, mass balance 150 is equal to D plus W; this is equation 1, and we can write the material balance; component balance is 150 into 0.4 would be W x w plus D into 0.9. So, from these two relation we can write W x w plus 150 minus W into 0.9, and from this we can get 60 is equal to W x W plus 150 minus 0.9 W, and from this we can write W into 0.9 minus x w is equal to 75. So, W is equal to 75 divided by 0.9 minus x w.

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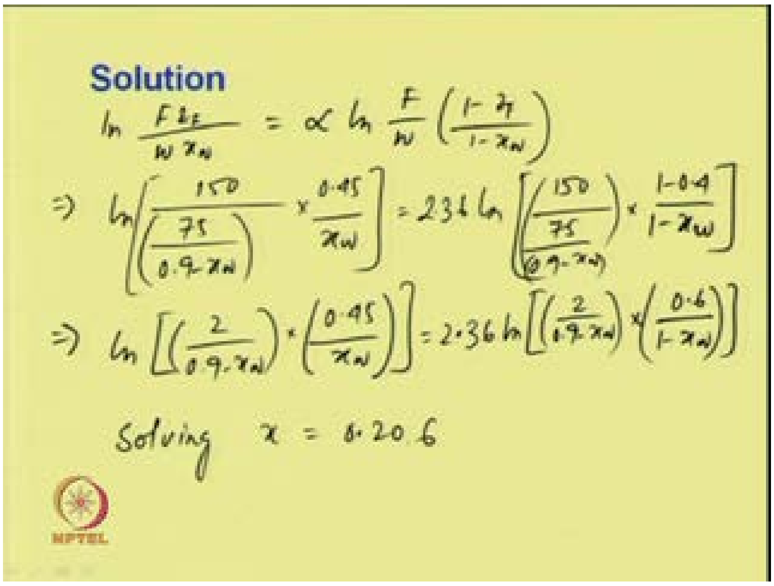

Solution

$\ln \frac{F x_F}{W x_W} = \alpha \ln \frac{F}{W}\left(\frac{1-x_F}{1-x_W}\right)$

$\Rightarrow \ln\left[\frac{150}{\left(\frac{75}{0.9-x_W}\right)} \times \frac{0.45}{x_W}\right] = 2.36 \ln\left[\left(\frac{150}{\frac{75}{0.9-x_W}}\right) \times \frac{1-0.4}{1-x_W}\right]$

$\Rightarrow \ln\left[\left(\frac{2}{0.9-x_W}\right) \cdot \left(\frac{0.45}{x_W}\right)\right] = 2.36 \ln\left[\left(\frac{2}{0.9-x_W}\right) \times \left(\frac{0.6}{1-x_W}\right)\right]$

Solving $x = 0.206$

Now if we use this w in place of Rayleigh equation, we know ln F Z F by W x w is equal to alpha ln F by W 1 minus Z F by 1 minus x w. So, we can write l n 150 by 75 by 0.9 minus x w into 0.45 by x w is equal to 2.36 ln 150 by 75 divided by 0.9 minus x w into 1 minus 0.4 divided by 1 minus x w . So, if we simplify this will be ln 2 by 0.9 minus x w into 0.5 by x w is equal to 2.36 ln 2 by 0.9 minus x w into 0.6 divided by 1 minus x w. So if we solve this, then we can obtain x is equal to 0.206.

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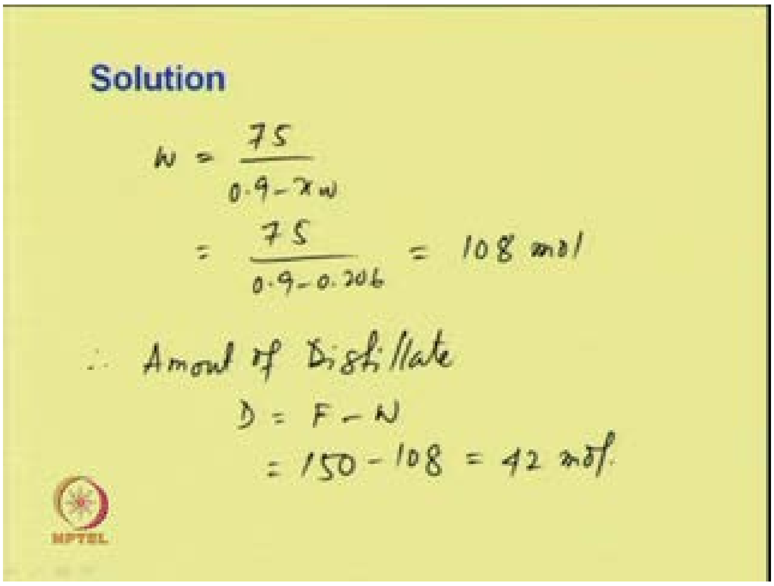

Solution

$$W = \frac{75}{0.9 - x_w}$$

$$= \frac{75}{0.9 - 0.206} = 108 \text{ mol}$$

$\therefore$ Amount of Distillate

$$D = F - W$$

$$= 150 - 108 = 42 \text{ mol}$$

So, we know that W is equal to 75 divided by 0.9 minus x w. So, if we put 75 by 0.9 minus 0.206, which would be 108. So, this is this much mol of W. So, the amount of distillate D is equal to F minus W from the total mol balance. So, it is 150 minus 108; so, this is equal to 42 mol.

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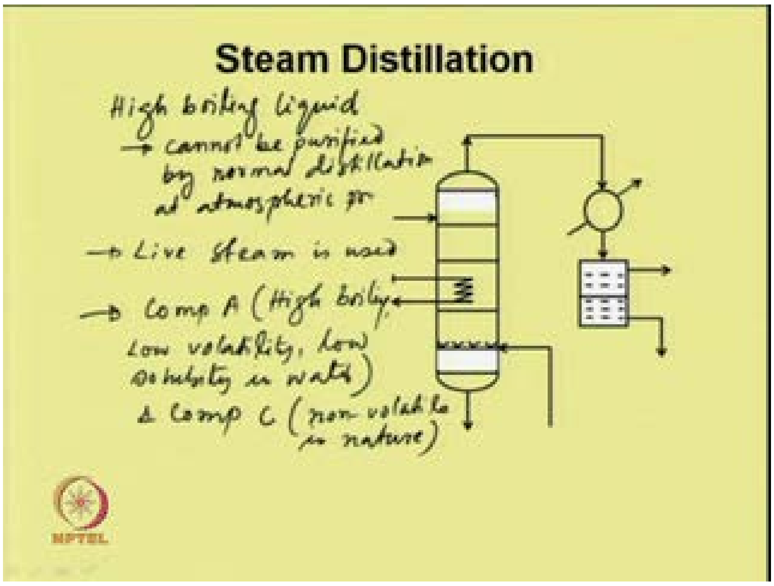

Steam Distillation

High boiling liquid

→ cannot be purified by normal distillation at atmospheric pr

→ Live steam is used

→ Comp A (High boiling, Low volatility, low solubility in water) & Comp C (non volatile in nature)

Let us consider another type of distillation which is steam distillation, particularly when some liquid which are very high boiling and it has some contaminant of non volatile components, which cannot be separated at normal distillation at atmospheric pressure. So, first is high boiling component. High boiling liquid cannot be purified by normal distillation at atmospheric pressure; because if we apply high temperature, there is a high possibility of decomposing the material at high temperature.

So when the components are high boiling, then they are in general immiscible in water. So, a separation at lower temperature can be possible by using steam distillation. So, it is an operation where live steam each used for distillation with the mixture of high boiling liquid with other components, which may be traces of impurities of non volatile impurities or any other component, which are non volatile in nature. So, our target is to recover the high boiling components, which has low volatility and low solubility in water. Suppose component A which is high boiling, low volatility, low solubility in water; and component C say is non volatile in nature.

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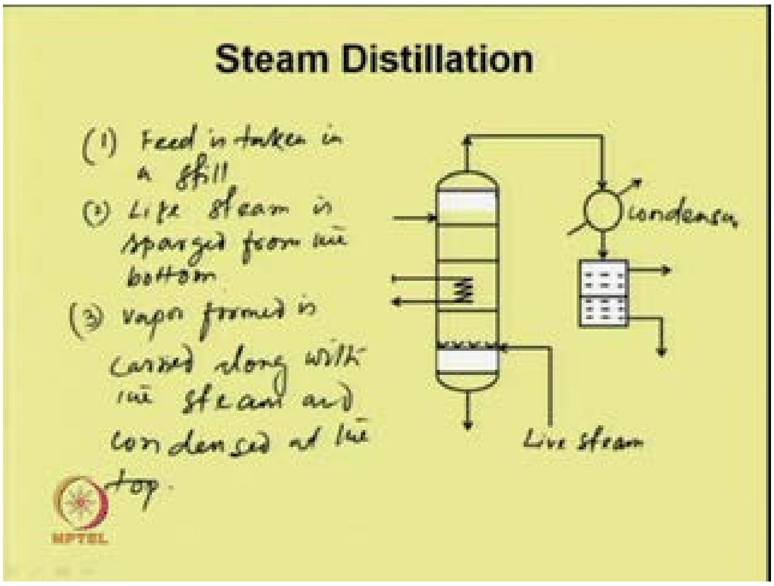

Steam Distillation

(1) Feed is taken in a still

(2) Live steam is sparged from the bottom

(3) vapor formed is carried along with the steam and condensed at the top.

So, this is shown in the figure. As we can see, the feed is taken in a tank and live steam is injected through the sparger. Feed is taken in a still and then live steam is sparged from the bottom. So, the vapor which will produce it will carried by the steam and it is condensed. This is condenser. Vapor formed is carried along with the steam and condensed at the top.

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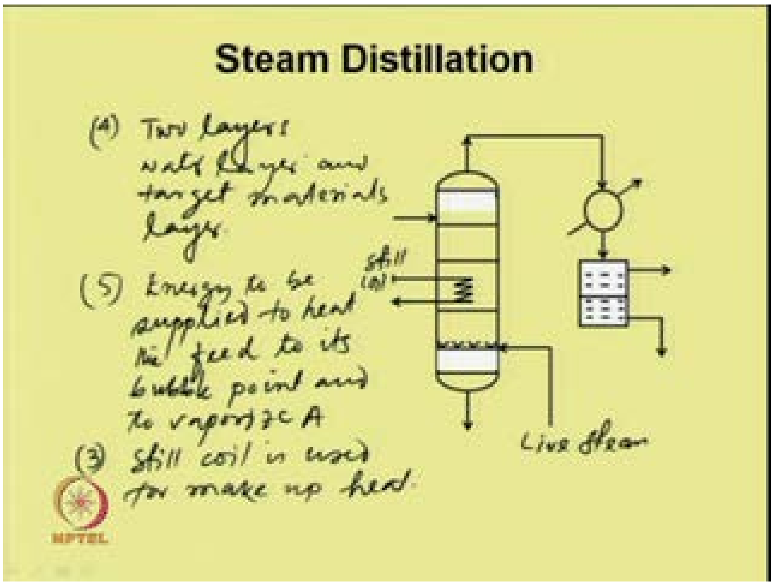

Steam Distillation

(4) Two layers water layer and target materials layer.

(5) Energy to be supplied to heat the feed to its bubble point and to vaporize A

(3) still coil is used for make up heat.

Now since, A has the less miscibility with water it will form two layers, and one layer is water layer and target materials layer. Then it is separated. This two layers can be easily separated, and another thing is that the energy required to heating the feed to its bubble point, so energy we have to be supplied to heat the feed to its bubble point and to vaporize A.

So if we use only live steam, say, there is a high chance of condensing the steam in the steel to heat the material. So to avoid these, a still coil is used to provide the energy for heating. So, a still coil is used for make up heat.

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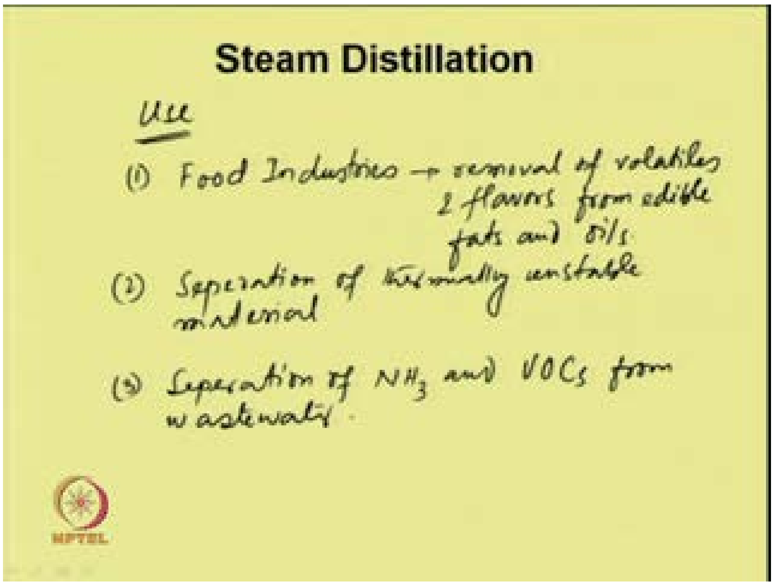

Steam Distillation

Use

(1) Food Industries → removal of volatiles & flavors from edible fats and oils

(2) Seperation of thermally unstable material

(3) Seperation of NH_3 and VOCs from wastewater.

Now, what is the use of this steam distillation? Use importantly. This is used in food industries for the removal of volatile materials, volatiles, and flavors from edible fats and oils, separation of thermally unstable material and third, separation of ammonia and VOCs volatile organic materials from waste water, and so on. There are so many applications.

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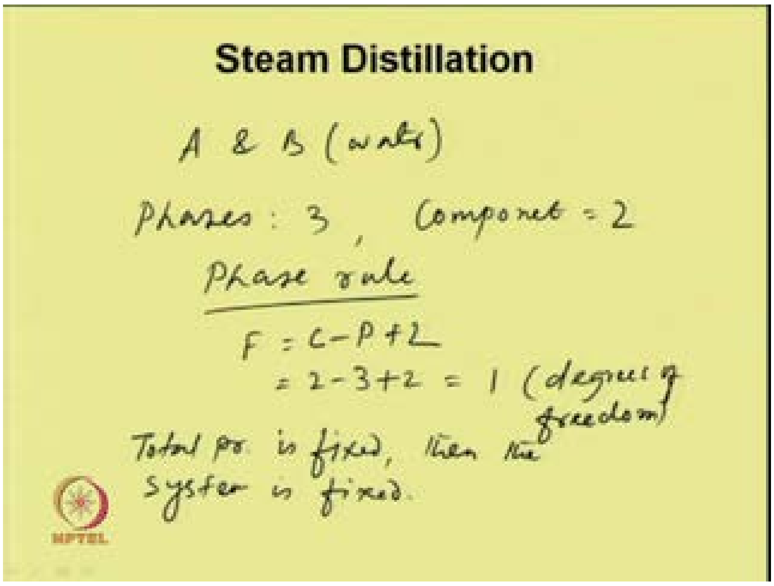

Steam Distillation

A & B (water)

Phases : 3 , Componet = 2

Phase rule

$F = C - P + 2$

$= 2 - 3 + 2 = 1$ (degree of freedom)

Total pr. is fixed, then the system is fixed.

Now let us consider if we have a two immiscible liquids A and B, which is water and this is boiled. So, vapor will generate. So, 3 phase system; so, phases 3, component 2. So, if we apply here phase rule we can see F is equal to C minus P plus 2. So, this will be 2 minus 3 plus 2 is equal to 1. This is the degrees of freedom. So if the total pressure is fixed, then the system is fixed.

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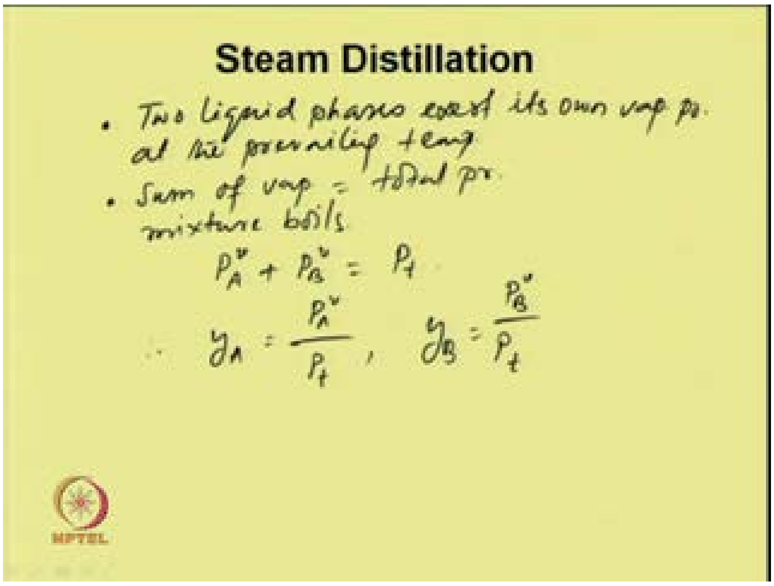

Steam Distillation

- Two liquid phases exert its own vap pr. at the prevailing temp
- Sum of vap = total pr mixture boils

$P_A^v + P_B^v = P_t$

$\therefore y_A = \frac{P_A^v}{P_t}, \quad y_B = \frac{P_B^v}{P_t}$

Now two liquid phases exert its own vapor pressure at the prevailing temperature, and this cannot be influenced by others. When the sum of the vapor pressure equals the total pressure, then the mixture boils. So, we can write P A v plus P B v is equal to P t. Therefore, y A should be P A v by P t; P A v and P B v are the vapor pressures of the components, and P t is the total pressure, y B should be P B v by P t. So, these are the vapor compositions.

Now by steam distillation as long as water is present, so the high boiling component A at a temperature below its normal boiling point will vaporize and without using the vapor pressure.

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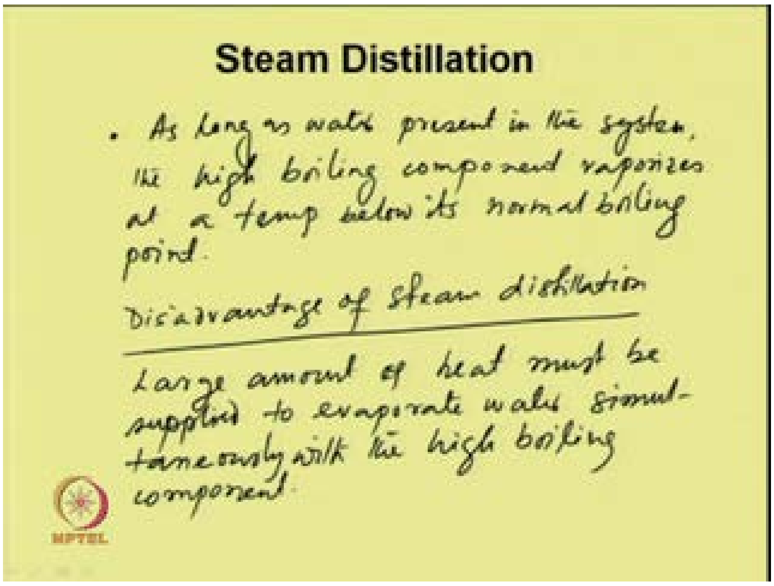

Steam Distillation

- As long as water present in the system, the high boiling component vaporizes at a temp below its normal boiling point.

Disadvantage of steam distillation

Large amount of heat must be supplied to evaporate water simultaneously with the high boiling component.

This means as long as water present in the system, the high boiling component vaporizes at a temperature below its normal boiling point. The disadvantage of the method: Large amount of heat must be supplied to evaporate water simultaneously with the high boiling component.

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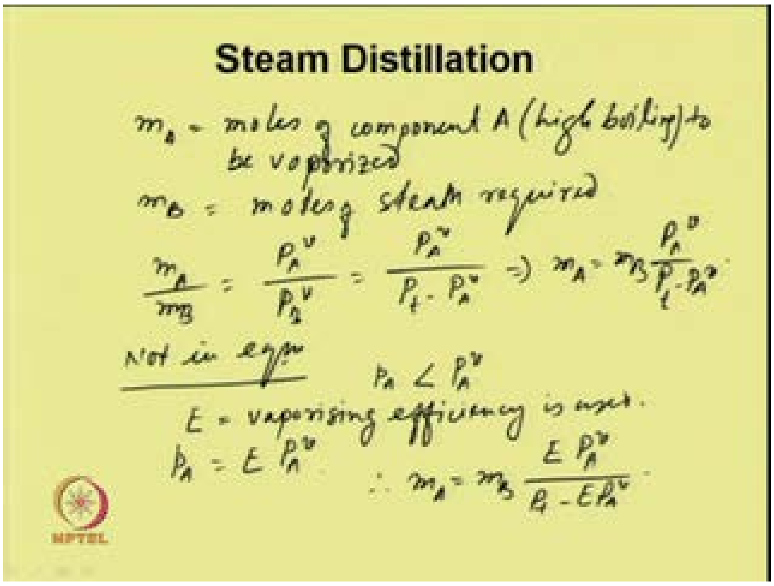

Steam Distillation

m_A = moles of component A (high boiling) to be vaporized

m_B = moles of steam required

$$\frac{m_A}{m_B} = \frac{P_A^v}{P_B^v} = \frac{P_A^v}{P_t - P_A^v} \Rightarrow m_A = m_B \frac{P_A^v}{P_t - P_A^v}$$

Not in eqm $\quad P_A < P_A^v$

E = vaporising efficiency is used.

$$p_A = E P_A^v \qquad \therefore m_A = m_B \frac{E P_A^v}{P_t - E P_A^v}$$

Now, if you take m A moles of component A which is high boiling to be vaporized, and m B moles of steam required. Then, if the system operates in equilibrium we can write m A by m B would be equal to P A v by P B v, which is P A v by P t minus P A v. So, from this we can write m A would be m B P A v by P t minus P A v. But if the system does not operate in equilibrium, then the partial pressure of A will be less than it is vapor pressure; partial pressure of A should be less then P A v. So, in that case another factor which is E is known as the vaporizing efficiency is used. So, P A would be E P A v. Therefore, we can write m A would be m B E P A v divided by P t minus E P A v.

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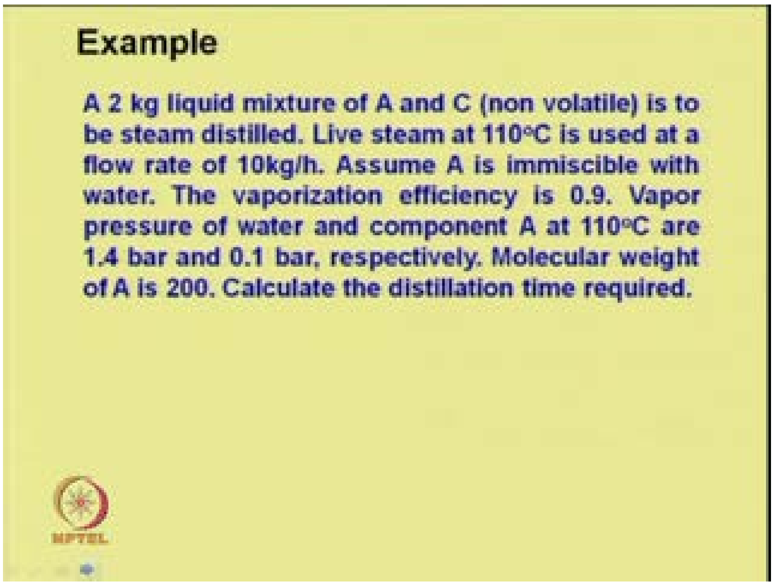

Example

A 2 kg liquid mixture of A and C (non volatile) is to be steam distilled. Live steam at 110°C is used at a flow rate of 10kg/h. Assume A is immiscible with water. The vaporization efficiency is 0.9. Vapor pressure of water and component A at 110°C are 1.4 bar and 0.1 bar, respectively. Molecular weight of A is 200. Calculate the distillation time required.

Now, let us take a very simple example. A 2 kg liquid mixture of A and C, which is non volatile is to be steam distilled. Live steam at 110 degree centigrade is used at a flow rate of 10 kg per hour. Assume A is immiscible with water. The vaporization efficiency is 0.9. Vapor pressure of water and component A at 110 degree centigrade are 1.4 bar and 0.1 bar respectively. Molecular weight of A is 200. Calculate the distillation time required.

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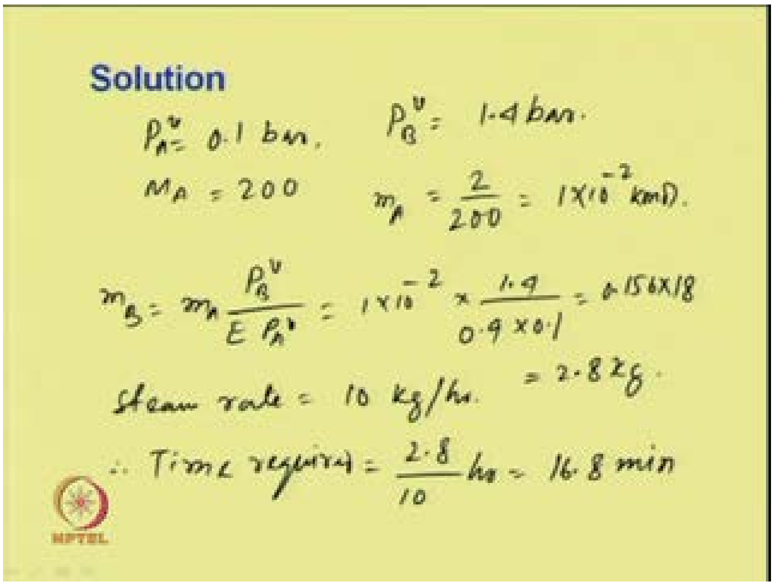

Solution

$P_A^v = 0.1$ bar. $P_B^v = 1.4$ bar.

$M_A = 200$ $m_A = \frac{2}{200} = 1\times10^{-2}$ kmol.

$$m_B = m_A \frac{P_B^v}{E\,P_A^v} = 1\times10^{-2} \times \frac{1.4}{0.9\times0.1} = 0.156\times18$$

$= 2.8$ kg.

Steam rate = 10 kg/hr.

∴ Time required = $\frac{2.8}{10}$ hr = 16.8 min

It is given that P A v is 0.1 bar and P B v is 1.4 bar. Molecular weight of component A is 200 and m A which is 2 by 200 is 2 kg mixture, which is equal to 1 into 10 to the power minus 2 Kmol. So, m B is m A P B v by E P A v, which is equal to 1 into 10 to the power minus 2 into 1.4 divided by 0.9 into 0.1, which is equal to 0.156 into 18, which is equal to 2.8 k g. Now it is given that steam rate is equal to 10 k g per hour. Therefore, time required is equal to 2.8 divided by 10 hour, which is equal to 16.8 minute.