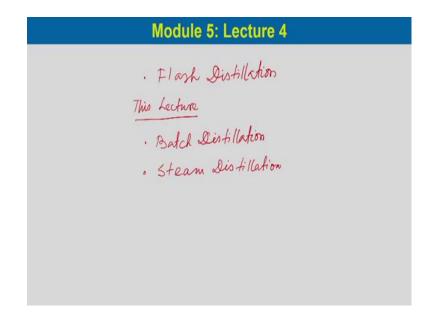
# Mass Transfer Operations-I Prof. Bishnupada Mandal Department of Chemical Engineering Indian Institute of Technology, Guwahati

# Lecture – 29 Batch and steam distillation

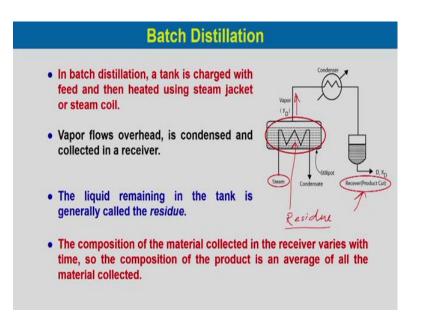
Welcome to the 4th lecture of module 5 of Mass Transfer Operation and in module 5, we are discussing distillation operation. So, before going to this lecture let us have brief recap on our previous lecture. In our previous lecture we mainly considered flash distillation.

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So, under flash distillation we mainly discuss the material balance, component balance and the energy balance equation and we have solved flash distillation problem for binary mixture. And, in this lecture we mainly consider two important things: one is batch distillation and second thing we will consider steam distillation.

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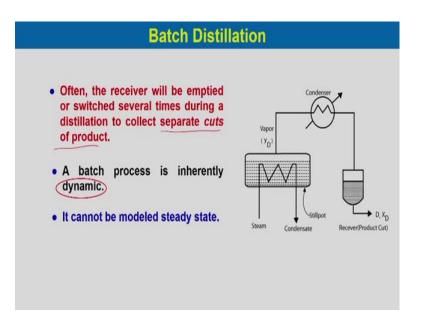


So, batch distillation basically in a batch distillation a tank is charged with feed and then heated using steam jacket or steam coil. So, basically you can see over here this is the tank which is charged with the feed and then it is closed and then using the steam jacket. So, steam over here and there is a coil or jacket or steam coil can be used and to heat the feed in this tank. So, what happens? The vapor flows over here which is goes up upwards through this vapor flow and it goes through a condenser it condensed and then the liquid is taken as a product in the receiver as a bottom product. So, it is the receiver as a product.

So, what happens in this case now, the liquid remaining in the tank which no generally called the residue. So, the liquid which is remaining here as in you know distillation process the liquid level is gradually decrease the total feed, gradually decrease and the which is remaining after the final distillation operation we call it residue. The composition of the material collected in the receiver so, over here varies with time.

So, the composition of the product is an average of all material collected. So, as long as the distillation continues the more and more product comes to this receiver and composition continuously changed. So, the final composition what we get is the average composition of the material collected over a time period.

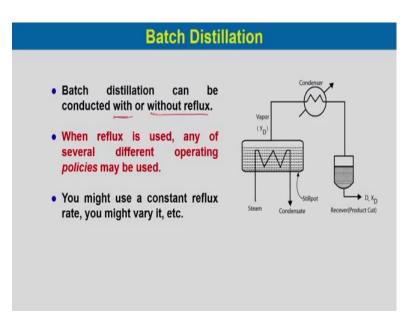
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Often the receiver will be emptied or switched several times during a distillation to collect separate cuts of products. So, basically what happens? So, if you need different product cut different product cut so, you can have at different time interval depending on the requirement you can take out the product.

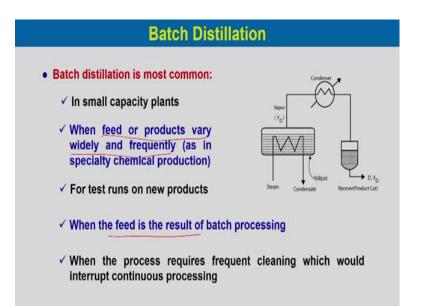
So, you will get different composition; each product taken out is known as the separate cut of the product. So, as you can see the batch process is inherently dynamic process because, the composition continuously change. So, it cannot be modeled at steady state that is why batch distillation cannot be model as steady state process.

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Now, batch distillation can work can be conducted with or without the flux. So, anyone can be possible. So, we can have this is without reflux, we can have also reflux to the feed drum. When reflux is used any of several different operating policies may be used. So, you might use a constant reflux rate or you might vary it. So, reflux rate like general distillation operation fractionator which we can change the reflux ratio. So, you can have fixed or constant reflux ratio or you can also use variable reflux ratio whatever you want. So, you can vary it or you can keep it constant so, depending on the requirement.

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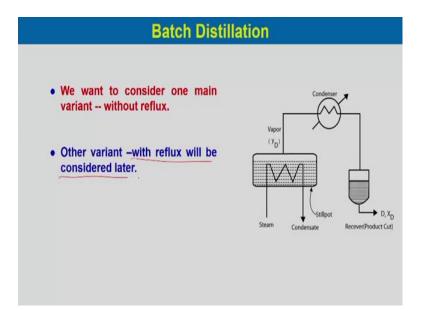


Batch distillation is most common and in small capacity plants, not everywhere if you have a very small capacity plants. So, you may not need to have continuous fractionator you can use batch distillation unit; when feed or product vary widely and frequently as in no specialty chemical production. For specialty chemicals in a basically it is produced depending on the needs or no depending on the availability of the feed. So, when feed or products vary widely and frequently so, in that case no batch distillation is preferred over the continuous distillation. For test run on new products generally we used to have batch distillation.

So, before going to the continuous process big large scale process to test it before going to scale of generally it is done as a test run for the new products in batch distillation column. And, also in the feed is result of batch processing; when the process requires frequent cleaning which would interrupt continuous processing. Because, of the material we used to take for the distillation and it creates lot of scaling which requires the cleaning of the apparatus frequently, in that case if we use the continuous process that may not be feasible option.

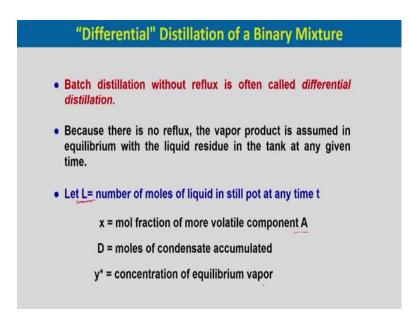
So, we need to interrupt the process of continuous operation and then we can clean it and there will be a shut down time for longer period. And so, it would be very difficult to run the continuous process. So, in that case it is better to use batch distillation process in that respect or in that case.

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Now, we want to consider one main variant of the batch distillation process: one is called without reflux and then the other one we will take up, the other variant that is with reflux will be considered in the later stage of our discussion.

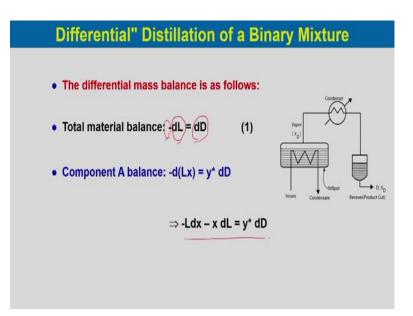
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Batch distillation without reflux is often called differential distillation. Because there is no reflux, the vapor product is assumed in equilibrium with the liquid residue in the tank at any given time. As it is continuously distillation process is going on the composition is gradually changing and in the tank where, the feed is fed there is a vapor space and there is liquid space and no reflux is coming from the product. So, there is an intimate contact between the vapor and liquid in the drum. So, it reaches equilibrium with the residue liquid in the tank, that is why it is called as differential distillation.

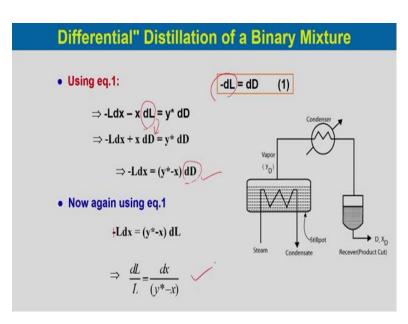
Let L be the number of moles of the liquid in the steel pot at anytime. So, L is the moles which is no present in the steel pot at any time t, x is the mole fractions of the more volatile component A. And, D is the moles of the condensate accumulated which is taken as outlet product and its composition concentration of equilibrium vapor that is y star.

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So, the differential mass balance is as follows minus dL would be dD so; that means, the liquid is vaporized differential amount small amount and it is going to the distillate. So, if it is dL amount is distilled you will get dD amount that should be equal because, there is no reflux and the negative sign is because the liquid is decreasing gradually. So, the component balance component A balance we can write minus d Lx would be equal to y star dD. So, from here we can write minus Ld x minus x dL would be equal to y star dD.

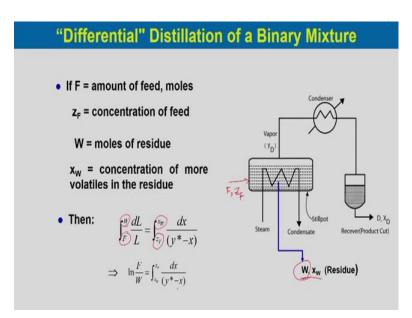
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Now, this is the material balance equation, equation 1 and this is the you know species balance equation component balance equation. And, if we just substitute this dL with dD we will get dL would be minus dD and if we substitute here so, it will be minus L dx plus x dD would be equal to y star dD. And, from here we can write minus L dx would be equal to y star minus x dD.

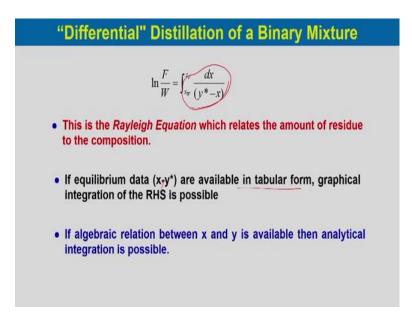
Now, again using equation 1 this equation we can write minus L dx would be equal to y star minus x and instead of dD we can write no dL here. So, you will obtain d L by L would be equal to dx by y star minus x. So, this would be plus because this dD would be substituted by minus dL so, it would be no plus. So, dL by L would be equal to dx by y star minus x; so we will obtain this equation.

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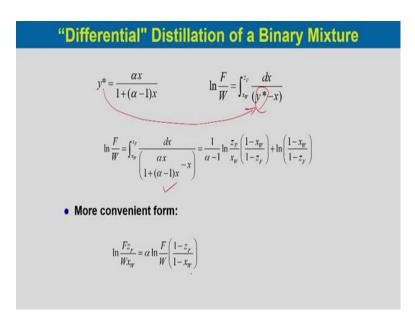
Now, you consider F is the amount of heat in moles and z F is the concentration of the feed. So, which is at the beginning in the steel pot now, W is the moles of residue which is over here its composition is x W. So, composition or concentration of the more volatiles in the residue is you know x W. So, then we can integrate from F to W which is present in the residue. So, integral F to W dL by L would be equal to integral z F to x W d x divided by y star minus x. So, in the feed we had at the beginning F z F so, this changes to W x W so, the limit accordingly change F to W and z F to x W. So now, if we integrate this we will obtain ln F by W would be equal to integral x W to z F dx by y star minus x.

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Now, this equation is a famous equation and is known as the Rayleigh equation and which relates the amount of residue to the composition. Now, if the equilibrium data or xy data xy star data are available in the tabular form graphical integration of the right hand side is possible. So, we need x y data so, if we have equilibrium data x y star data then no graphical or if it is available in the tabular form then we can do the graphical integration of this.

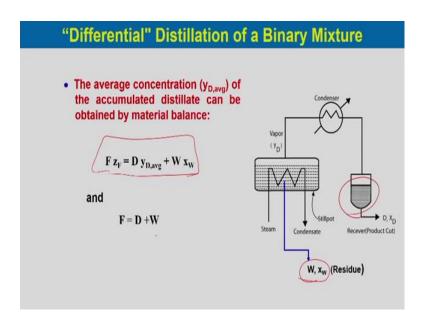
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If algebraic relations between x and y is available then analytical integration is possible. y star this is analytical equation, you can relative volatility equation; y star is equal to alpha x divided by 1 plus alpha minus 1 into x and we have ln F by W would be equal to integral x W to z F dx by y star minus x. So, now if we substitute in place of y star this equation this would be like this.

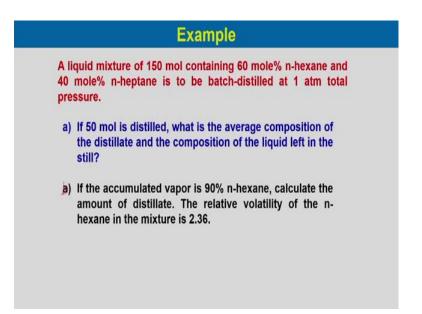
And, if you just do the analytical integration it would be 1 by alpha minus 1 ln z F by x W into 1 minus x W divided by 1 minus z F plus ln 1 minus x W divided by 1 minus z F. The more convenient form from this we can write ln F z F divided by W x W would be equal to alpha ln F by W into 1 minus z F divided by 1 minus x W.

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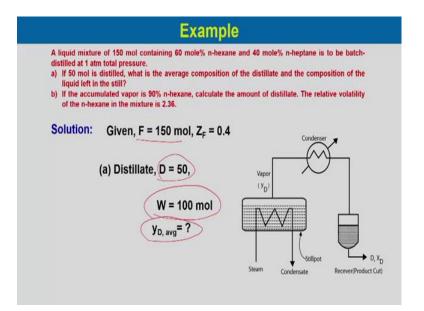
So, the average concentration which is you know coming to this over here with respect to time is y D average of the accumulated distillate and that can be obtained through the material balance which is F z F would be equal to D y D average plus W x W. So, this is the material balance equation or the component balance equation. And, this is the residue as you can see which is W x W and feed is known and this is the total material balance equation F is equal to D plus W.

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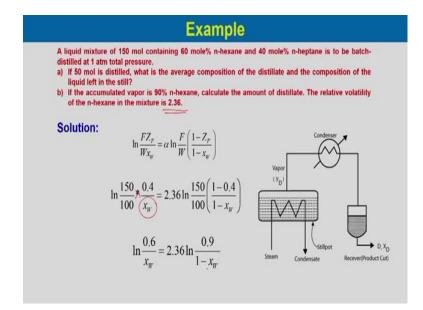
Now, let us take an example to solve this batch distillation problem. A liquid mixture of 150 mole containing 60 mole percent n hexane and 40 mole percent n-heptane is to be batch distilled at 1 atmosphere total pressure. Now, if 50 mol is distilled what is the average composition of the distillate and the composition of the liquid left in the still? Now second part is no this is b, if the accumulated vapor is 90 percent n-hexane calculate the amount of distillate and it is given that the relative volatility of n-hexane in the mixture is 2.36.

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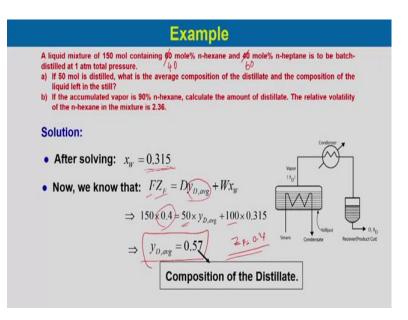
So, it is given the feed is 150 mole and z F is 0.4, distillate is given which is you know D is equal to 50, W is equal 100 mole. So, since D is equal to you know total feed is 150 mole and D is 50 and so, W would be you know 100 moles. So, we need to calculate y D average.

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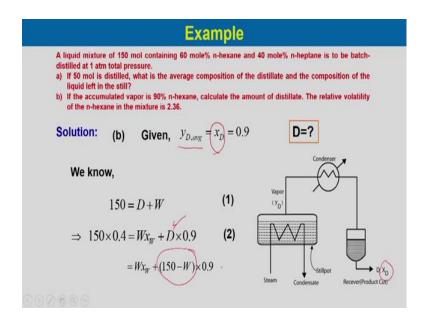
Now, if we you know substitute over here is ln F Z F by W x W would be equal to alpha ln F by W 1 minus z F divided by 1 minus x W. Now, if we substitute here you know F is 150 divided by W is 100 and you know z F is 0.4 and x W we need to you know calculate would be equal to 2.36 which is given, ln 150 by 100 into 1 minus 0.4 divided by 1 minus x W. So, if we just you know write this equation. So, rearrange this equation it is ln 0.6 divided by x W would be equal to 2.36 ln 0.9 divided by 1 minus x W.

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Now, if you solve you know iteratively you can get you know x W would be equal to 0.315. So, now, you know that F z F would be equal to D y y D average plus W x W. Now, this terms are known 150 z F is 0.4 and you know D is 50 y D average we need to calculate; so, this plus 100 into 0.315. So, from this we can calculate y D average is equal to 0.57; please note that we have considered here z F is 0.4. So, which is this should be 40 mole percent and this is 60 mole percent n-heptane. So, make this correction. So, with this you know we can obtain y D average would be equal to 0.57; now this is the composition of the distillate.

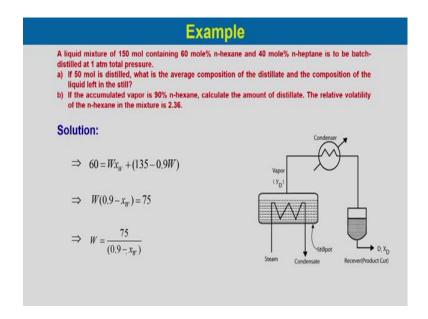
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Now, the second part we have to calculate the accumulated vapor is 90 percent n-hexane. So, y D average would be equal to x D because, that is what is coming over here and completely would be accumulated at this you know receiver. So, the composition the average composition y D average should be equal to x D so, which is 0.9. Now, we need to calculate D.

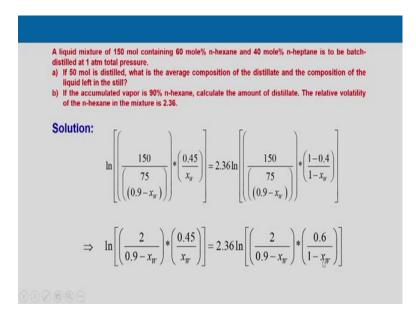
So, from the total material balance it is 150 would be equal to D plus W and if you write you know the species balance equation, it would be 150 in to 0.4 would be equal to W x W plus D into 0.9. Now, in this case if we substitute here this D would be 150 minus W. So, which is substituted here and you know in place of D it is 150 minus W into 0.9. Now, if you solve it 60 would be equal to W x W plus 135 minus 0.9 W.

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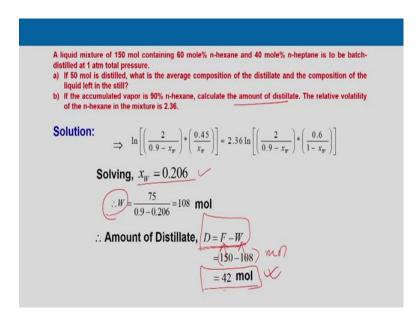
Now, after solving it will give W would be equal to 75 divided by 0.9 minus x W.

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So, in the integral equation the Rayleigh equation, if we substitute the values of W; so the Rayleigh's equations can be written like this and you will obtain ln 2 by 0.9 minus x into 0.45 divided by x W would be equal to 2.36 ln 2 by 0.9 minus x W in to 0.6 divided by 1 minus x W. So, this we can solve iteratively.

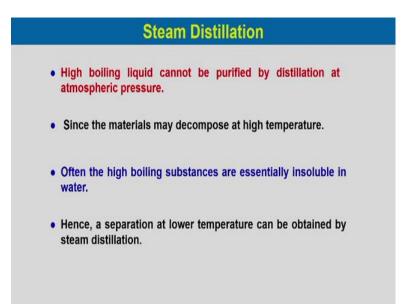
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And we can obtain the values x W is equal to 0.206. So, once we obtained x W we can calculate you know W so, which is equal to 75 divided by 0.9 minus 0.206 which is equal to 108 mole. So, once W is known to us now, the distillate we can calculate from

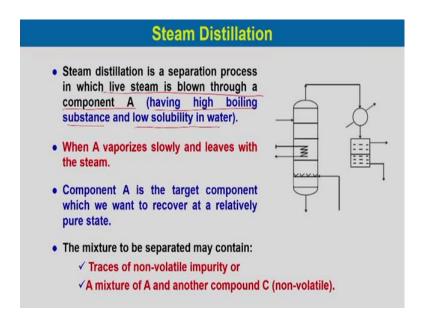
the total material balance equation feed is known to us and no W is known to now we have calculated. So, it is 150 minus 108 so, it will give you know this much mole. So, it will give you know 42 mole so, 42 mole of distillate. So, for the second problem that is the accumulated vapor is 90 percent n-hexane and we need to calculate the distillate amount. So, the distillate this way we can calculate is about 42 mole.

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Now, we will discuss the next topic which is steam distillation. High boiling liquid cannot be purified by distillation at atmospheric pressure. Since the materials may decompose at high temperature. Often the high boiling substances are essentially in soluble in water. In this case a separation at lower temperature can be obtained by steam distillation.

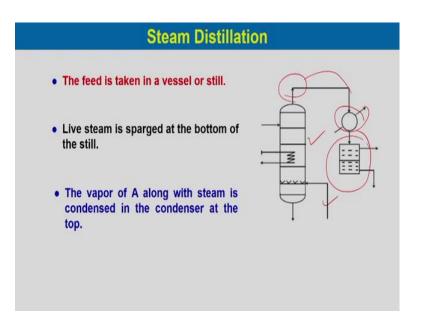
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Steam distillation is a separation process in which live steam is blown through a component A having high boiling substance and low solubility in water. In this case you can see a sketch over here, we need live steam is blown through a component A having high boiling substance and low solubility in water. So, if you have a mixture of components and their boiling points is high.

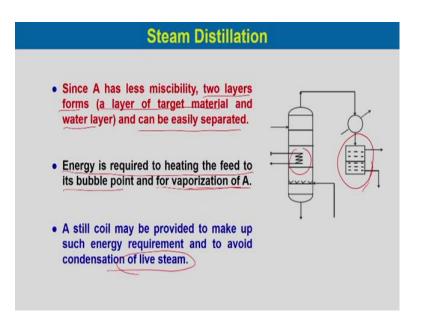
And, it is very low solubility in water we can just inject the live steam through that solution. When A vaporizes slowly and leaves the steams, the component A is the target component which we want to recover at a relatively pure state. The mixture is to be separated may contain traces of non-volatile impurity or a mixture of A and another compound C that is non-volatile.

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So, the feed is taken in a vessel or still you can see over here, live steam is sparged at the bottom of the steel. A vapor A along with steam is condensed in the condenser at top. So, the vapor of component A it comes over here and this is the condenser and it condenses and then it is put to the you know separator where, the water and the component vapor A is separated in this. So, you will have two product cut: one is the water which is as a liquid form you can get and you can the vapor phase we will get component A.

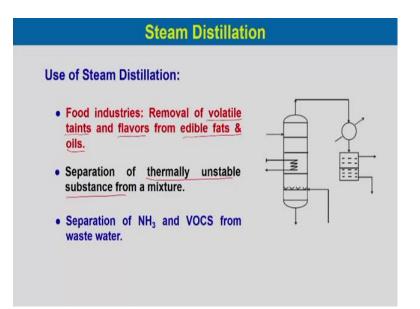
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So, since A has less miscibility two layers forms or it may be in the liquid forms as it says A has less miscibility with water two layers forms; a layer of the target material and a water layer and that can be separated in this separator. So, energy is required to hitting the feed to its bubble point and for vaporization of A. So, good amount of heat need to be supplied for the heating of the feed to its bubble point and then it will be vaporized for a certain period of time, vaporization of component A.

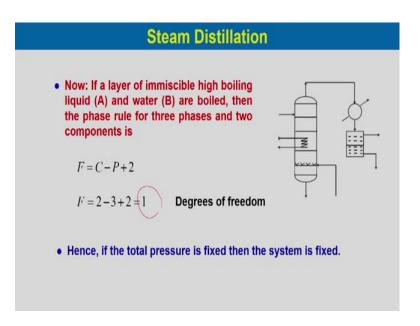
So, a steel coil you can see over here steel coil may be provided to make up such energy requirement and to avoid condensation of the live steam. So, live stream should not be condensed inside this column, inside the steam distillation column. So, in between we can install some coil inside the column so, that always it will be in a live steam. So, it should not condense there.

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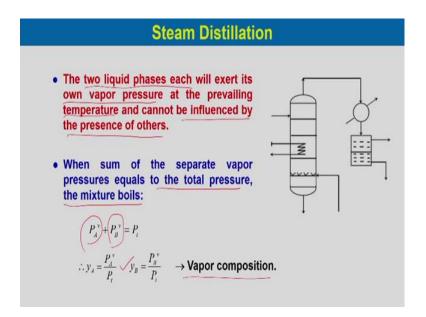
Now, use of steam distillation as we said for food industries that is removal of volatile taints and flavors from edible fats and oils. So, removal of volatile taints and flavors from the edible fats and oils; this is one of the application of the steam distillation. Second is separation of thermally unstable substance from a mixture. So, if you have a thermally unstable substance which will degrade at high temperature, but can be separated by steam distillation; like separation of ammonia and VOCS from waste water it can be done using the steam distillation.

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Now, if a layer of immiscible high boiling liquid A and water B are boiled then the phase rule for three phases and two component can be written. So, F is equal to C minus P plus 2. So, in this case we have two components and so, P is three phase. So, it is 3 plus 2 so, it will give 1 degrees of freedom. So, hence if the total pressure is fixed then the system is fixed.

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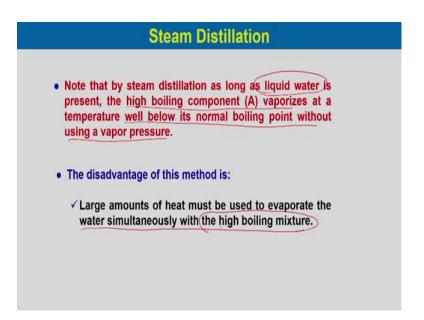


The two liquid phases each will exert its own vapor pressure at the prevailing temperature and cannot be influenced by the presence of the other. So, in this case as we

have two different liquids and they will prevail you know exert their own vapor pressure and at the prevailing temperature. And, that cannot be influenced by the presence of any other component. When the sum of the separate vapor pressures equals to the total pressure the mixture boils.

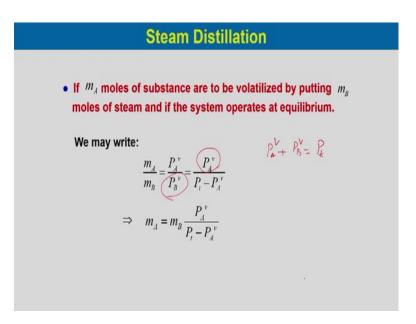
So, basically if we at their individual vapor pressure and that will you know would be equals to the total pressure then the; you know mixture would start to boil. So, we can write P A v for the vapor pressure of component A plus vapor pressure of component B P v B would be equal to the total pressure. Therefore, y A would be equal to P A v divided by P t and y B would be equal to P v B by P t; so that is the vapor composition.

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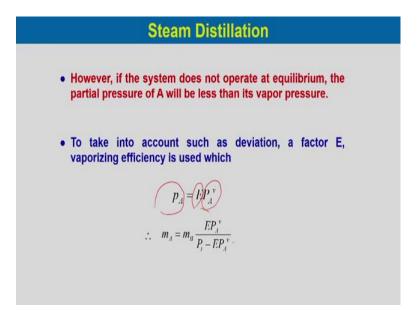
Note that by steam distillation as long as liquid water is present the high boiling component A vaporizes at a temperature well below its normal boiling point without using a vapor pressure. So, in case of the steam distillation as long as the liquid water present or water present as liquid the high boiling component A vaporizes at a temperature well below its normal boiling point without using its vapor pressure. The disadvantage of this method is large amount of heat must be used to evaporate the water simultaneously with the high boiling mixture because you need to get the water as well as the mixture.

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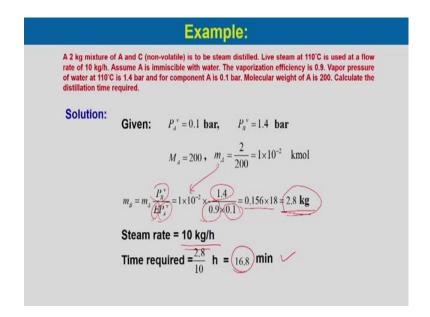
Now, if m A moles of substance are to be volatilized by putting m B moles of steam and if the system operates at equilibria or equilibrium then we can write m A by m B would be equal to P A v by P B v would be equal to. So, this is vapor pressure of component A divided by P B v we can write from the vapor pressure of the individual component plus P A v plus P B v is equal to P t total pressure. So, from here we can write P A v divided by P t minus P A v. So, if you rearrange it would be m A would be equal to m B into P A v divided by P t minus P A v.

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However, if the system does not operate at equilibrium, the partial pressure of A will be less than its vapor pressure. So, to take into account such a deviation a factor E that is vaporizing efficiency is used which is p A partial pressure of A would be equal to E into P A v that is the vaporizing efficiency E into the vapor pressure of component A. So, that would be equal to the partial pressure of component A. Therefore, m A would be equal to m B and instead of partial pressure we can write E P A v divided by P t minus E P A v. So, now let us take an example to solve steam distillation problems.

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A 2 kg mixture of A and C that is non-volatile C is non-volatile is to be steam distilled. Live steam at 100 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 at 110 degree centigrade is 1.4 bar. And, for component A is 0.1 bar at that temperature 110 degree centigrade. Molecular weight of A is 200. Calculate the distillation time required. Now, it is given that P A v is 0.1 bar and P B v is 1.4 bar, molecular weight of A is 200 and m A small m A we can calculate would be equal to 2 by 200 which is equal to 1 into 10 to the power minus 2 kilo mole.

So, we can calculate m B would be equal to m A P B v divided by EP A v. So, partial pressure you know vapor pressure of component B divided by vaporizing efficiency into the vapor pressure of component A. So, by substituting this m A and then the you know vapor pressure both the components and then the vaporizing efficiency is 0.9. So, we

would obtain 0.156 into 18 which is equal to 2.8 kg, steam rate which is given is 10 kg per hour and hence we can calculate the time required. So, total 2.8 kg so, 2.8 by 10 hours; so which would be equal to 2.8 into 60 by 10 which is 16.8 minute; so, this way we can calculate the time required for the distillation for a particular separation.

So, thank you for attending this lecture and we will continue our discussion of distillation operation in the next class.