Principles and Practices of Process Equipment and Plant Design

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Module - 02
Lecture - 15
Batch Distillation

So, hello everybody. After covering flash distillation, rectification or rather fractionation, today we are going to discuss Batch Distillation Operations. Now, normally what we find is we feel that batch distillation it is very simple and therefore, it should be easy, but rather it is just the other way around which is particularly because it is an unsteady state operation.

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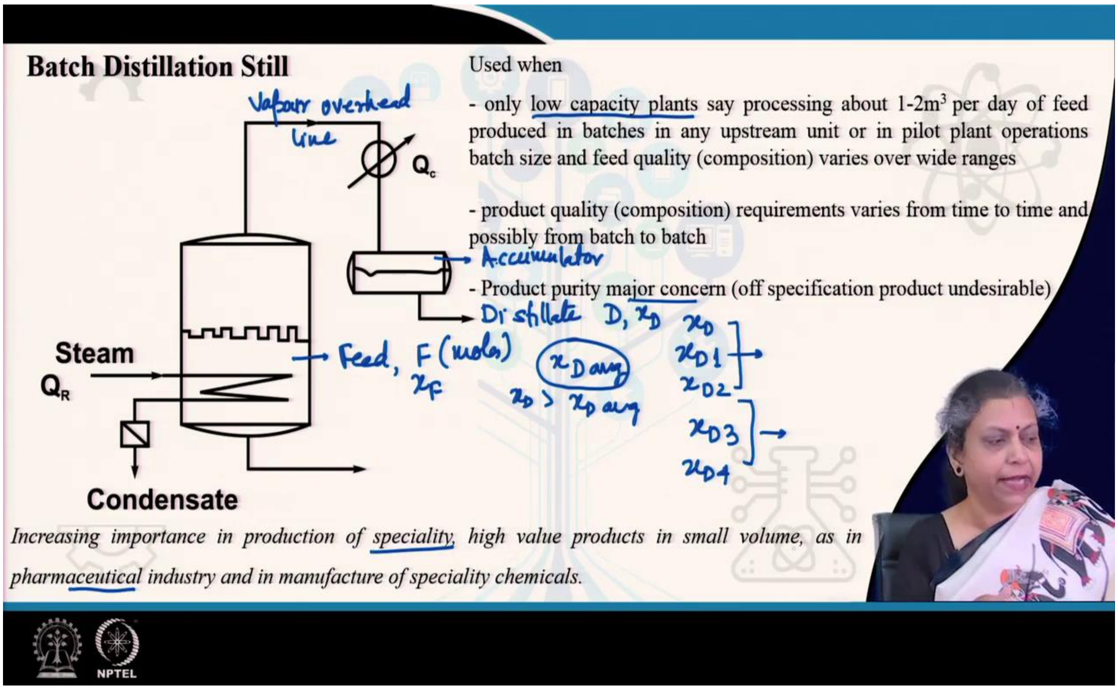

Batch Distillation Still

Used when

- only low capacity plants say processing about 1-2m^3 per day of feed produced in batches in any upstream unit or in pilot plant operations batch size and feed quality (composition) varies over wide ranges

- product quality (composition) requirements varies from time to time and possibly from batch to batch

- Product purity major concern (off specification product undesirable)

Increasing importance in production of speciality, high value products in small volume, as in pharmaceutical industry and in manufacture of speciality chemicals.

So, naturally, in that case, most of the equations they will be written in terms of differential terms and that adds to the complexity of the design. But anyhow batch distillation is for operation it is not a very complex operation. In this case, what do we have? Initially just as we had mentioned in the introduction, there is a still in that particular still. It is just with a fixed amount of a feed.

So, therefore, here some amount of feed is there and say the total amount of the feed-in say moles it is F and in the composition is x_F. Since it is a liquid feed, we take the composition as x_F. This feed is heated by a heating medium. In this case, what is the heating medium? It is the steam that supplies heat and the condensate of this steam is collected. As the feed gets heated then gradually, it reaches its bubble point.

Once it reaches its bubble point vapour start generating. What happens to these vapours? This vapour starts rising, it rises through the vapour overhead line. This was mentioned in the introduction part as well. They rise up in the vapour overhead line, from there they come to the condenser. In the condenser, cooling occurs, the vapours get condensed in the condenser. Then they come and they are collected in the accumulator. It is usually termed as the accumulator. In the accumulator, they get collected and after that, the distillate is drawn from here (from the bottom of the accumulator). The distillate amount over a particular time duration is D, and its composition is given by x_D. Distillation keeps on proceeding in this particular way. Now, as distillation proceeds what do you expect is going to happen? Gradually, initially, the more volatile components will start vaporizing and they will be moving. Then, with time gradually the more volatile components get depleted in the feed, the feed becomes richer in the less volatile component. So, naturally with time what happens? The distillate also becomes richer in the less volatile component. What I mean to say is that the distillate composition along with the feed composition keeps on varying with time. We continue the distillation till we get the product specification that has been specified.

Say suppose a product has been specified that we would like to have some particular distillate composition which I am denoting as $x_{D,avg}$. We have to see that initially the vapour that is richer in composition as compared to $x_{D,avg}$. Then, gradually the composition is going to be depleted in the more volatile component.

It was mentioned earlier repeatedly that we denote these compositions which refer to the composition of the more volatile component, in the case of binary mixtures or in case of ternary mixtures as well. There can be instances when the composition of the desired component is denoted by x's, but mostly it is the more volatile component at least for this particular class.

So, therefore, initially, we will start with $x_D > x_{D,avg}$. Then, gradually as distillation proceeds, x_D will be depleted. It will become less and less till maybe it will fall below $x_{D,avg}$. We continue distillation till the average product composition meets our specifications. The moment it has met, we stopped the distillation process.

Now, this distillation was not very much practised in chemical industries in earlier times. Particularly in the oil industries etc, where normally there was a large scale operation with a huge amount of feed and the need for the continuous operation was very important. But gradually, the need is increasing, particularly with the importance of low capacity plants. At the moment especially in the production of I should say pharmaceuticals, high speciality chemicals, and food industries etc.

So, therefore, now more and more batch distillation operations are being performed in industries. Now, there are certain things that you have to remember that batch distillation will be opted only for low capacity plants otherwise it becomes un-economical. Now, there are certain particular situations where batch distillation is preferred. For example, suppose you get feed from different sources or maybe you get feed from the same source, but the composition of the feed keeps on varying.

Now, if this has to be accommodated in a fractionator then in that case naturally you will the design has to be very flexible and there is no guarantee that for all the feed conditions you get the product composition that you desire. So, when the amount is small you would prefer the batch distillation. It is mainly preferred due to its flexibility of operation.

There is the other thing also may be in the same batch, you can use it for different products. You may have different clients who would want different specifications of the product. It can happen to the same client at different instances due to the different applications. They would want the different specifications of the product.

So, for that particular situation, batch distillation is going to be preferred. You can monitor the product composition and accordingly, you can provide the product specification that is desired.

Particularly, it is important when product purity is a major concern and when off-specification products are extremely undesirable. So, you can see that well we have reached the $x_{D,\ avg}$ and therefore, we will not proceed with the distillation.

But normally for when the product purity is a major concern, then we would like to operate with a constant distillate composition that is usually done in multistage batch distillation systems, whereas I had mentioned in the introduction and we will also be discussing at the end of the class.

Just above the still, we have a column, and the column is operated in such a way that we get a constant distillate output. For this, what do we need to know? We need to vary the reflux ratio. So, therefore, when product purity is a major concern, we would like to opt for batch distillation.

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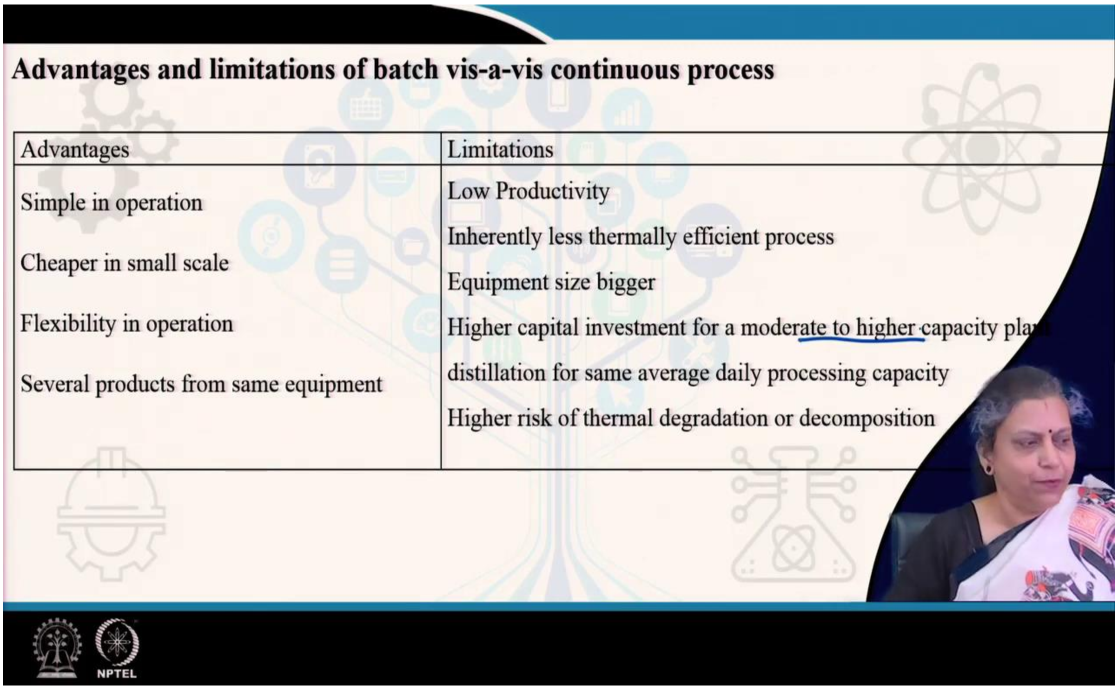

Advantages and limitations of batch vis-a-vis continuous process

Advantages	Limitations
Simple in operation Cheaper in small scale Flexibility in operation Several products from same equipment	Low Productivity Inherently less thermally efficient process Equipment size bigger Higher capital investment for a moderate to higher capacity pla distillation for same average daily processing capacity Higher risk of thermal degradation or decomposition

So, just summarizing the advantages of a batch process, vis-a-vis a continuous process, we now definitely see the first thing it is very simple in operation. There are not many control parameters. Definitely, for small scale production, it is cheaper. But this does not apply when we go for a larger scale because we have to remember that the size of the equipment is larger compared to the size of a continuous operation for the same particular capacity.

Why? Because the entire feed has to be fed in the still itself and then the operation has to start. So, for a smaller scale, it is definitely cheaper, but this does not apply when we go for a moderate or high capacity plant. Flexibility in operation, we have already discussed, quite naturally because you can use the same still for different variations or rather for different compositions of the feed.

You can also use this for different compositions, when different product specifications are required and from the same equipment. You can get different products. For example, in this particular case, we find that maybe initially you start, you get some particular product say suppose which has a composition of say x_{D1}.

So, we start with some particular x_D. We proceed till some particular x_{D2} such that the average composition becomes x_{D1}. So, we have one particular accumulator where we keep on collecting till the average composition is x_{D1}. Maybe you need another product that has a composition of say x_{D3}. So, for that what we do?

We have collected, in one accumulator we have collected product whose average composition is x_{D1}. Then, we direct the vapour to another accumulator. In that particular accumulator maybe we start collecting from x_{D2} to x_{D4} such that the average composition is x_{D3}.

So, in this particular way, depending upon the number of products we can add the specifications. We can collect the products in different accumulators and we can get different products from the same feed in the same operation. This is not possible in a single piece of equipment in a continuous process.

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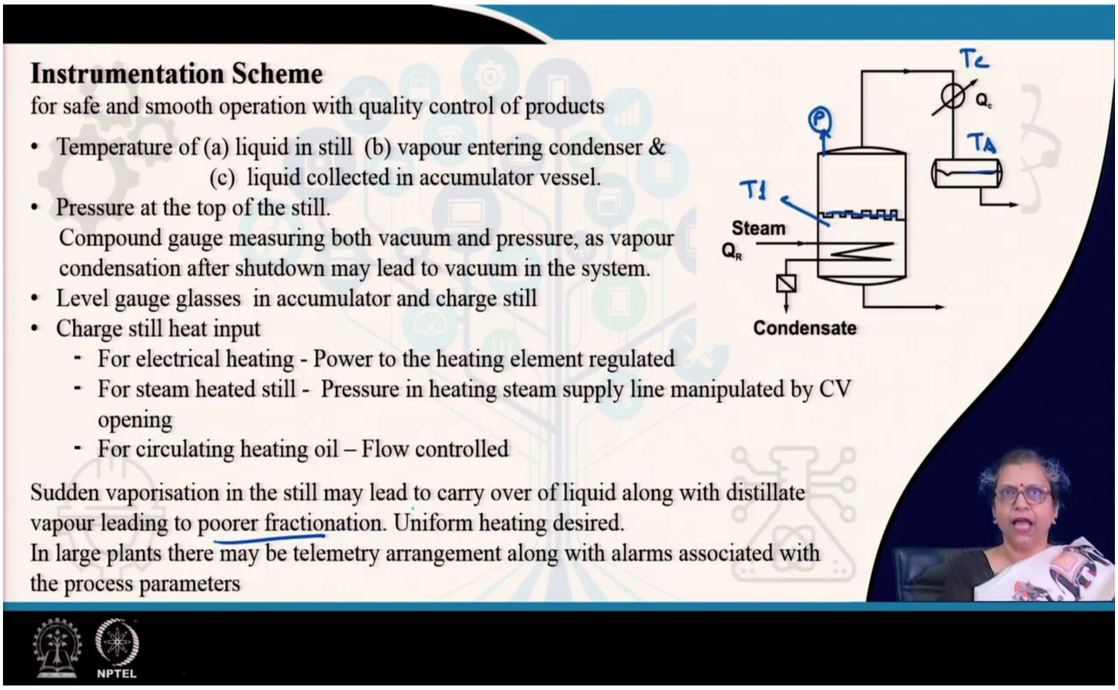

Instrumentation Scheme

for safe and smooth operation with quality control of products

- Temperature of (a) liquid in still (b) vapour entering condenser & (c) liquid collected in accumulator vessel.
- Pressure at the top of the still. Compound gauge measuring both vacuum and pressure, as vapour condensation after shutdown may lead to vacuum in the system.
- Level gauge glasses in accumulator and charge still
- Charge still heat input
  - For electrical heating - Power to the heating element regulated
  - For steam heated still - Pressure in heating steam supply line manipulated by CV opening
  - For circulating heating oil – Flow controlled

Sudden vaporisation in the still may lead to carry over of liquid along with distillate vapour leading to poorer fractionation. Uniform heating desired.

In large plants there may be telemetry arrangement along with alarms associated with the process parameters

Now, even if it is simple, there has to be some particular controls for the quality control of the products and also for a safe and smooth operation. What are the things that we need to control? Just like all other operations, distillation operations, the temperature has to be controlled, the pressure has to be controlled, the level in the vessels have to be controlled. Where should you control the temperature?

Definitely, in the still, the temperature has to be controlled. This is one place where it has to be controlled. The pressure also has to be controlled at the top of the still. Based on this temperature, pressure, the equilibrium data will be generated and we can start designing the batch column.

The other place where it is necessary to control the temperature is the vapour that is entering the condenser. Here is the temperature has to be controlled and also it has to be controlled in the liquid which is collected in the accumulator.

So, therefore, here also the temperature has to be controlled. Based on these two temperatures you know very well that the temperature of the vapour which is entering the condenser that is normally at the dew point and the liquid which is leaving, it is at the bubble point. So, just like we have discussed in your fractionator in this case also, the condenser provides the latent heat for the vapour to be cooled from its dew point to its bubble point.

Once you know the accumulated temperature, (if the accumulated temperature supposedly is going to be at the bubble point) that this feed should be cooled before it is stored. So, the amount of cooling that is required can be figured from that.

Levels have to be controlled. Level has to be controlled inside still and also inside the accumulator. So, you need level gauge glasses in these two places. Now, normally if you have a very small plant, then for all these controls, they can just add the column itself and an operator can come and he can keep on measuring. If we have a larger plant then there can be a telemetry arrangement along with alarms etc associated to control and regulate the process parameters.

Another very important thing has to be controlled here. See the entire vapour which is generated, the entire distillation which is going on, that depends upon the heating which is being added to this particular still.

So, therefore, the heating rate should be controlled. It should be as uniform as possible because suddenly if a huge amount of heat is given, there will be a lot of vaporization. There is every chance that this vapour will entrain some amount of liquid and it takes along with it and that will reduce the efficiency of the distillation. It will result in poor fractionation.

So, therefore, uniform heating is very much necessary. The heating rate is necessary because that tells you the duration of the startup period and also the rate of distillation. How do we control this particular heat input? This can be heated either by electrical heating, in that case, the power to the heating element can be regulated.

We can use steam just as we have shown here. We can use steam by heated stills, in that case, the pressure in the heating steam supply line, that can be controlled utilizing a control valve. We may have circulating heating oils, maybe from some other part of the plant in that case we control the flow and in that way we regulate the heating.

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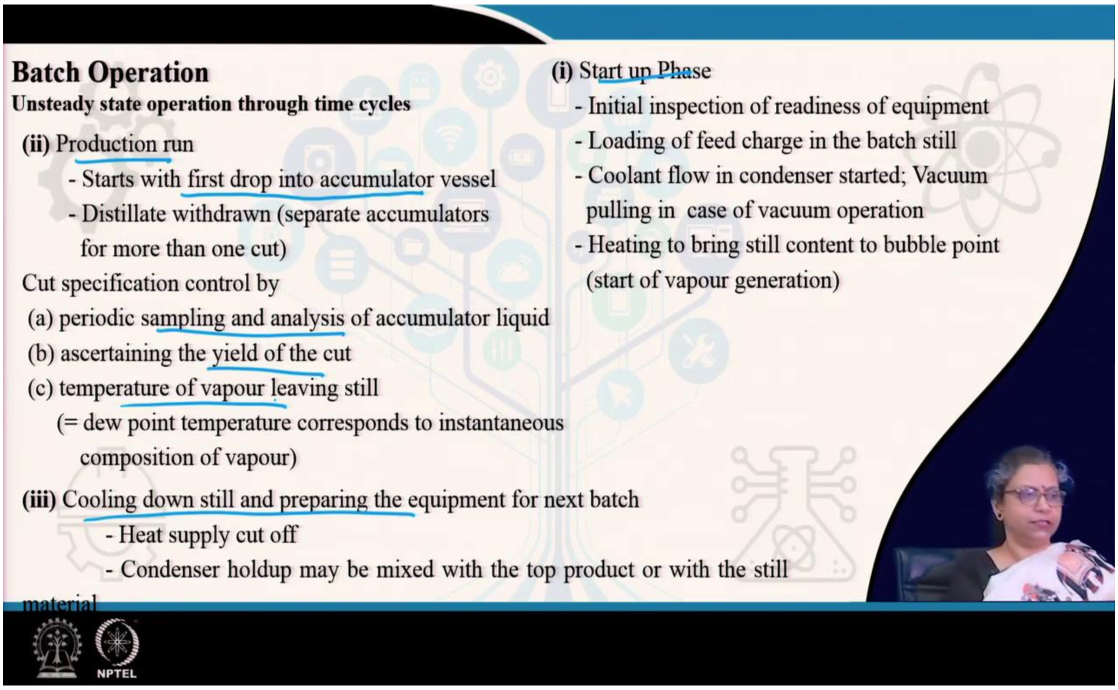

Batch Operation

Unsteady state operation through time cycles

(i) Start up Phase

- Initial inspection of readiness of equipment
- Loading of feed charge in the batch still
- Coolant flow in condenser started; Vacuum pulling in case of vacuum operation
- Heating to bring still content to bubble point (start of vapour generation)

(ii) Production run

- Starts with first drop into accumulator vessel
- Distillate withdrawn (separate accumulators for more than one cut)

Cut specification control by

(a) periodic sampling and analysis of accumulator liquid

(b) ascertaining the yield of the cut

(c) temperature of vapour leaving still

(= dew point temperature corresponds to instantaneous composition of vapour)

(iii) Cooling down still and preparing the equipment for next batch

- Heat supply cut off
- Condenser holdup may be mixed with the top product or with the still material

Well, when we go to discuss batch distillation, so along with the instrumentation. It is also very important that we understand the operation of a batch process. It is an unsteady state operation. Naturally, it has to go through time cycles. What do we have? We initially have a start-up phase, and then we have a production, and then definitely the ending phase.

In the startup phase, naturally, what you will do? You will be first inspecting the readiness of the equipment, and then you will load the feed in the batch still and you will also start the coolant flow in the condenser. What does this do? This ensures that if you have some extremely volatile component then you do not lose it whatever goes into the condenser from the beginning is condensed and can be collected in the accumulator.

So, we start the coolant flow rate in the condenser as well. If it is a vacuum operation, then the vacuum pulling also has tried to start from the very beginning itself, so that suddenly if the vacuum is started at some intermediate point then it may lead to excessive vapourization. After that, we have started everything and we start heating the still.

As long as the heating continues and vapour is being generated, but nothing is coming and collecting in the accumulator, it is the start-up phase. Do we continue heating, during the heating what happens? The vapour gets generated and this particular vapour heats the upper part of the accessories the pipings etc. Then why it gets heated? Some amount gets condensed, it comes back in the still and then again it starts heating. So, in this particular way, the entire system gets heated.

After that, a sufficient amount of the entire vapour has gone, and then it gets condensed and the moment the first drop of the liquid accumulates enters the accumulator vessel. we start the production run. In the production run, definitely, we are producing what we want from the batch distillation process.

So, during this production run, actually the still gets heated and vapours are produced. The vapours are condensed, and then they come and then they are collected in the reflux drum, and if we required different products, products with more cuts, each particular product that we are collecting they are usually referred to as cuts.

So, if we need more than one cut, then naturally we will be collecting them in separate accumulators and we will be controlling the cut specifications either by periodic sampling and analysis of the accumulator liquid. We can also ascertain the yield of the cut and based on the previous information on the feed we can decide upon the cut specification.

We can also regulate or we can also measure the temperature of the vapour which is leaving the still because it's important to remember that this particular vapour temperature corresponds to the dew point temperature of the vapour at that particular instant of time.

So, in this way, we continue the production run as long as we are getting the distillate of our specifications or possibly if the bottoms product is specified. We attain the specification of the bottom product. Depending upon the specification, we would like to continue the production run.

When we find that our specifications are no longer being met then we go to the end of phase or the cooling down phase where this still is cooled, and we prepare the equipment for the next batch. We cut off the heat supply line, and then there will be some amount of liquids that will be accumulated in the condenser. It is either mixed with the top product or it is mixed with the still.

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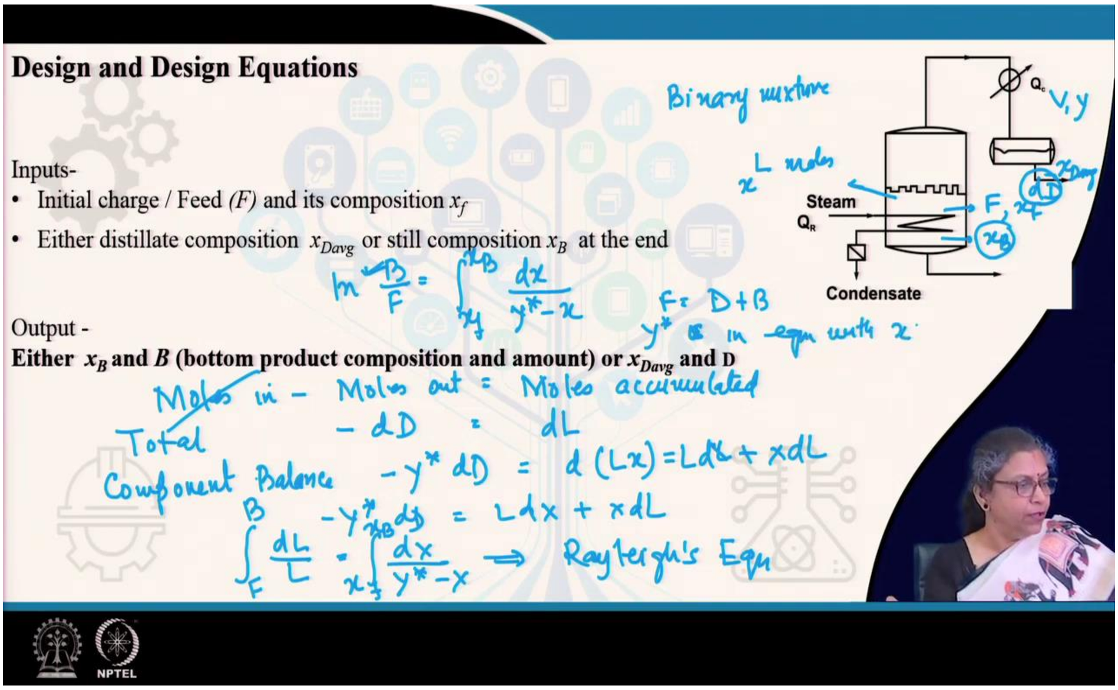

Design and Design Equations

Inputs-
- Initial charge / Feed *(F)* and its composition x_f
- Either distillate composition x_{Davg} or still composition x_B at the end

Output -
Either x_B and B (bottom product composition and amount) or x_{Davg} and D

Binary mixture

x L moles

$$\ln \frac{B}{F} = \int_{x_F}^{x_B} \frac{dx}{y^* - x}$$

$F = D + B$

y^* is in eqm with x

Moles in − Moles out = Moles accumulated

Total $-dD = dL$

Component Balance $-y^* dD = d(Lx) = Ldx + xdL$

$-y^* dD = Ldx + xdL$

$$\int_F^B \frac{dL}{L} = \int_{x_F}^{x_B} \frac{dx}{y^* - x} \Rightarrow \text{Rayleigh's Eqn}$$

So, we have understood the instrumentation and we have understood the cycles of operation of a batch. So, this is a design class. We would like to come to the design equations. As this is an unsteady state process, therefore, the design equations will be written in form of differential equations.

Usually, what are the inputs you tell me? The feed which is there as I have told you, the feed (F) is the input, the x_F is the input. As I have told you either x_{Davg} will be specified or maybe here x_B that will be specified according to which we are going to perform the operations.

Now, the famous Rayleigh equation is usually used for this. So, the Rayleigh equation is nothing, it is simply based on the overall and the component mass balance or the mole balance around the distillation unit. So, in this particular case, what do we have? Any particular instant of time says the amount of liquid which is there that is L moles and the composition of the liquid in here it is x_{in} the still.

Again, I will repeat, this x refers to the more volatile component. I will be deriving the Rayleigh's equation for a binary mixture. So, at the instant, the amount of liquid is L moles and the composition is x. The vapour, which is rising, is V, and its composition is y. This V is condensed here and that is collected as D. At the moment, we are just discussing a single-stage distillation unit. So, therefore, at any instant of time, we find that the amount of distillate that is collected is equal to dD.

So, therefore, if from this just from the basics, the basic mass balance equation can be written as:

$$moles\ in - moles\ out = moles\ accumulated$$

In this particular case, we do not have any moles in. So, therefore, moles accumulated will be equal to minus moles out.

$$-moles\ out = moles\ accumulated$$

Tell me that if I do the total material balance, what is the moles out? dD amount. At that particular instant of time, what is the moles accumulated? dL, for that instant of time. If I do the component balance on the more volatile component, then in that case, in that instant of time what is going to be collected?

$$-dD = dL; \quad total\ balance$$

$$-y^{*}dD = d(Lx) = Ldx + xdL; componenet\ balance$$

This y* is in equilibrium with the liquid composition here. From, there we can write

$$\int_{F}^{B} \frac{dL}{L} = \int_{x_F}^{x_B} \frac{dx}{y^{*} - x}$$

So, therefore, this gives you the famous Rayleigh's equation.

From this particular Rayleigh's equation, if we integrate it and if we write it, then from this we get this is ln B by F this is equals to integral x F to x B dx by y star minus x. So, if we want to find out B then in that case we need to integrate it.

Even if you can find out D also, from the overall material balance you quite naturally F equals to for during the entire time interval F equals to D plus B we can find it out.

$$F = D + B$$

But for finding this out we need to perform this particular integration.

It is important to remember that naturally when we try to design any operation, we assume ideal cases, under ideal cases this y* is in equilibrium with the x at that instant of time.

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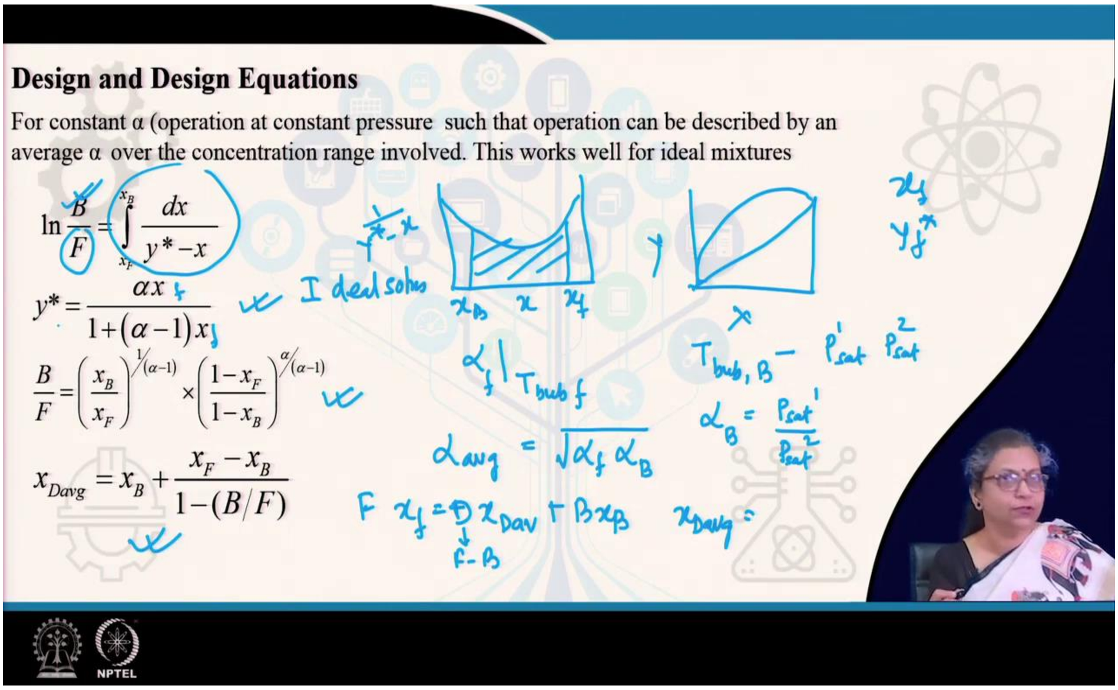

Design and Design Equations

For constant α (operation at constant pressure such that operation can be described by an average α over the concentration range involved. This works well for ideal mixtures

$$\ln\frac{B}{F} = \int_{x_F}^{x_B}\frac{dx}{y^*-x}$$

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

$$\frac{B}{F} = \left(\frac{x_B}{x_F}\right)^{1/(\alpha-1)} \times \left(\frac{1-x_F}{1-x_B}\right)^{\alpha/(\alpha-1)}$$

$$x_{Davg} = x_B + \frac{x_F-x_B}{1-(B/F)}$$

So, now, in order to perform this graphical integration, what you can do? We have already the equilibrium data with us. So, therefore, what we can do? For each particular equilibrium data, we can find out $\frac{1}{y^*-x}$. We plot it as a function of x. We may have got some sort of a plot. To perform the graphical integration, we need to perform it from x_F to x_B.

So, we can find out the area under the curve and from there we can find out B (provided F is given). We can also have an algebraic equation which will which can describe the equilibrium curve. We can use that algebraic equation here y* expressed as a function of x.

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

For ideal solutions, when alpha more or less remains constant, under that condition we know that y* can be related with x in terms of alpha. Therefore, for such situations, we can relate y star and x by this particular equation. And once we can do this, then we can express B/F from this equation. We can do this.

$$\frac{B}{F}=\left(\frac{x_B}{x_F}\right)^{\frac{1}{(\alpha-1)}}\left(\frac{1-x_F}{1-x_B}\right)^{\frac{\alpha}{(\alpha-1)}}$$

$$x_{Davg}=x_B+\frac{x_F-x_B}{1-\left({}^{B}/_{F}\right)}$$

Now, this is applicable for ideal solutions and particularly when alpha does not vary much during the process of distillation. What do you know? Initially, if you are if x_B is not known to you then you calculate the alpha at the bubble point of the feed ($\alpha_f\big|_{T_{bub\,f}}$) and then you use that alpha. At the end of the distillation, you know what is your corresponding T bubble point ($T_{bub,B}$) for the bottoms temperature.

Corresponding to that, you can find out P_1^{sat} of one component, P_2^{sat} of the second component. From these two you can find out the alpha which is nothing but equal to P saturated 1 by P saturated 2, where 1 is the more volatile component.

$$\alpha_B=\frac{P_1^{sat}}{P_2^{sat}}$$

Then, you can compare the alpha at the end of the operations say the alpha of the bottom (α_f) with the alpha of the feed (α_B). If you find that they are not very different there is no problem.

If you find that they are quite different then definitely you have to redesign the whole thing by taking a geometric mean of alpha for the feed and the bottom temperatures.

$$\alpha_{avg}=\sqrt{\alpha_f\alpha_B}$$

So, therefore, in this particular way it proceeds. Now, from the overall material balance, we also get what will be the x_{Davg} in this particular case.

From, the overall material balance,

$$Fx_F=Dx_{Davg}+Bx_B$$

We know, $F = D + B$

So, by using the above two equations and by rearranging the final expression, we can get a final expression like the following:

$$x_{Davg} = x_B + \frac{x_F - x_B}{1 - \left({}^{B}/_{F}\right)}$$

Now, these are the two equations that we use and y* is calculated from this equation.

To start with initially what we do? We know x_F. So, once we know x_F, we find out y_F^* using this equation where this x corresponds to x_F. We find out this y*. If we find that this y* is greater than that which is specified for the distillate we go for this particular process.

If we find that y* is less, we know there is no point in proceeding with this particular process. We will either have to reduce the pressure or else we have to go for a multistage column.

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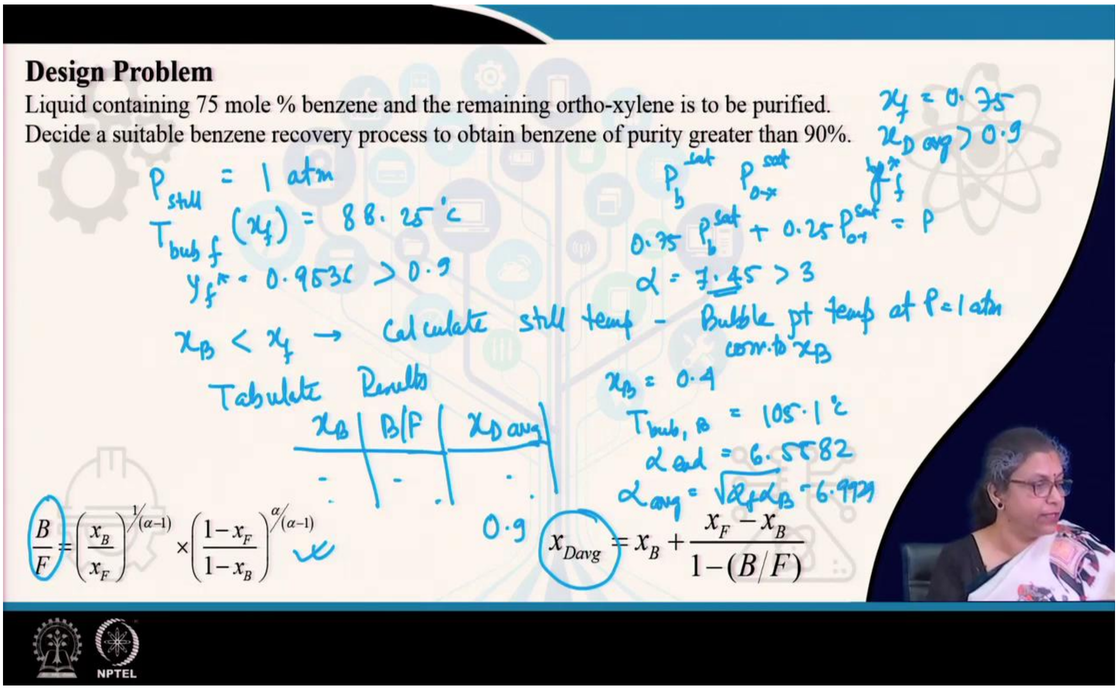

Design Problem

Liquid containing 75 mole % benzene and the remaining ortho-xylene is to be purified.
Decide a suitable benzene recovery process to obtain benzene of purity greater than 90%.

$x_f = 0.75$

$x_{D\,avg} > 0.9$

$P_{still} = 1$ atm

$T_{bub\,f}(x_f) = 88.25\,°C$

$y_f^* = 0.9536 > 0.9$

P_b^{sat} $P_{o\text{-}x}^{sat}$ y_f^*

$0.75\,P_b^{sat} + 0.25\,P_{ox}^{sat} = P$

$\alpha = 7.45 > 3$

$x_B < x_f \rightarrow$ Calculate still temp - Bubble pt temp at P = 1 atm corr. to x_B

Tabulate Results

x_B	B/F	$x_{D\,avg}$
-	-	.
.	.	.

0.9

$x_B = 0.4$

$T_{bub,B} = 105.1\,°C$

$\alpha_{end} = 6.5582$

$\alpha_{avg} = \sqrt{\alpha_f \alpha_B} = 6.9929$

$$\boxed{\frac{B}{F}} = \left(\frac{x_B}{x_F}\right)^{1/(\alpha-1)} \times \left(\frac{1-x_F}{1-x_B}\right)^{\alpha/(\alpha-1)} \checkmark$$

$$\boxed{x_{Davg}} = x_B + \frac{x_F - x_B}{1-(B/F)}$$

Just to give you a very concrete example, see we have started with a liquid that contains 75 mole% benzene and say 25 mole% ortho-xylene which means that your $x_f = 0.75$. Now, we need to design a suitable recovery process for the benzene such that we would have liked to have $x_{Davg}>0.9$. We can do it or not for that we need to find out what is the y_f*, rather y* in equilibrium with the feed composition.

If the alpha for this particular case, we assume that the pressure of the feed is maintained constant at say 1atm. In that particular case, you can find out what will be the T bubble point of the feed for x_f. How do you do it? You know the x_f composition. So therefore, you have to select the P_b^{sat} and the $P_{o\text{-}x}^{sat}$ in such a way that

$$0.75P_b^{sat} + 0.25P_{o-x}^{sat} = P = 1atm$$

So, you have to select. Corresponding to this you can find out the T bubble point of the feed. Now, this is called a trial and error technique. So, whenever you will be given problems in assignments or your assessment tests, definitely the bubble point of the feed will be specified for most of the cases.

So, therefore, we find out that in this particular case, it is around says 88.25℃ and corresponding to this we find that the y_f*, it is around 0.9536 which is much much greater than 0.9. So, this shows that we can proceed with this particular process.

So, what do we do? We initially start with this particular value of alpha (α) that we have found out from here. In this particular case, you can check it for yourself alpha is roughly around 7.45 or something. So, therefore, and one more thing I had told you that when alpha is greater than 3, we can go for a single stage. So, therefore, this is greater than 3 we can confidently go for a single-stage calculation.

How do we go about the calculation? Initially, we assume some particular x_B which is definitely which is less than x_f. Corresponding to that particular x_B, we calculate the still temperature. What is the still temperature? It is the bubble point temperature at P = 1 atm, corresponding to x_B. So, therefore, we calculate it.

Then, using this particular equation we calculate B by F from here.

$$\frac{B}{F} = \left(\frac{x_B}{x_F}\right)^{\frac{1}{(\alpha-1)}} \left(\frac{1-x_F}{1-x_B}\right)^{\frac{\alpha}{(\alpha-1)}}$$

So, therefore, we can calculate B/F. Once B/F is calculated, we can calculate x_{Davg}. Normally, what do we do? We tabulate the results. We assume different x_B's for each particular x_{B}, we plot the B/F and we plot the x_{Davg}. We continue, we keep on continuing this till we get an x D average of 0.9.

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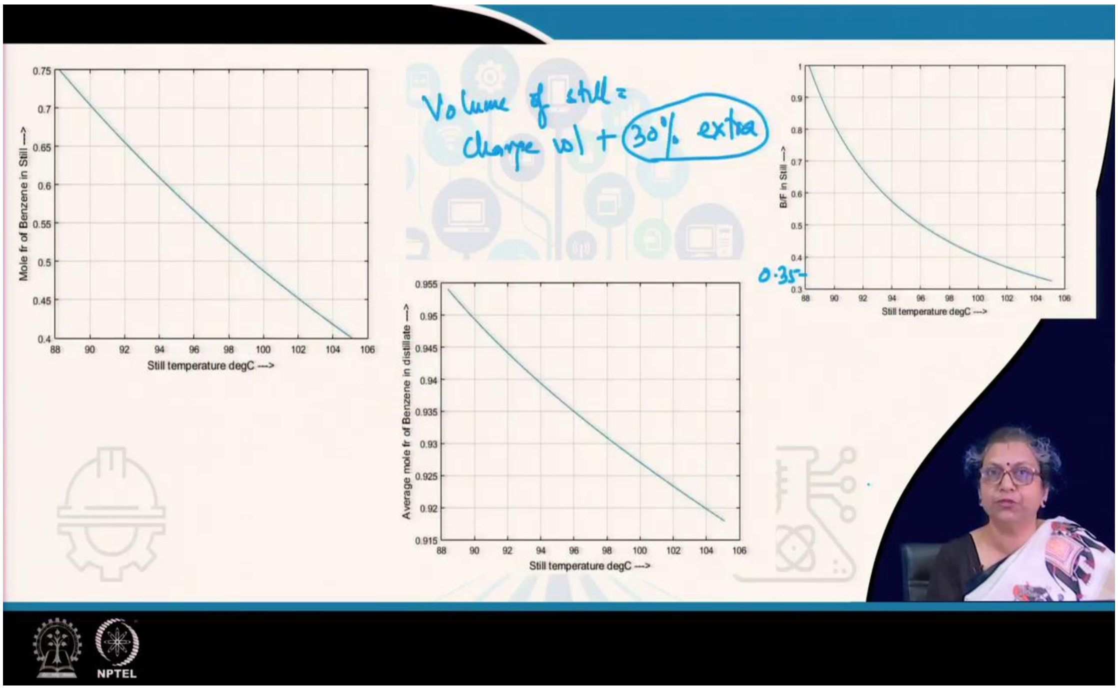

Volume of still = charge vol + 30% extra

I have just done it, and I have shown the curves here which shows that more or less the x_{Davg} of 0.92 at a still temperature of about 104 °C and under this condition, the mole fraction in the still is about 0.4. I find that the B/F in this particular case, is almost equal to 0.35 or something which is not bad.

Now, there are certain things that you have to remember in this particular case.

- Still size that you are supposed to take depends upon the initial charge that you are supposed to fill or the initial feed amount that was supposed to fill there.
- You need to note that at the end of the operation the amount of liquid that remains should be sufficient to submerge the heating elements.

So, therefore, if we have a very less amount of B/F in the still then it will not be proper. Normally, what do we assume? We assume that the volume of the still will be equal to the charge volume plus some about say 30 % extra to provide for the vapour space. Then, very frequently we also have a demister pad. So, these things have to be accounted for.

If we have a foaming liquid, then this particular space has to be given extra. In the end, whatever volume you have, that volume of the bottom should be sufficient such that the heating elements can be submerged there.

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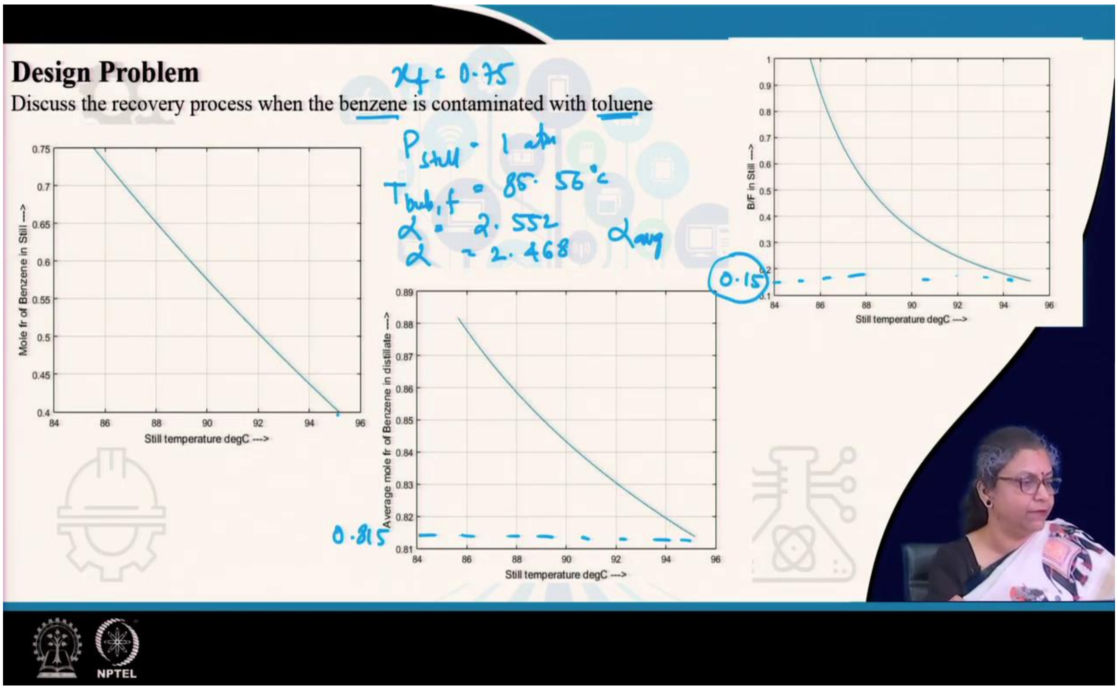

Design Problem

Discuss the recovery process when the benzene is contaminated with toluene

$x_f = 0.75$

$P_{still} = 1$ atm

$T_{bub,f} = 85.56$ °C

$\alpha = 2.552$

$\alpha = 2.468$ α_{avg}

Just I thought that I will repeat the process when the benzene with the same composition may be from some other source we have got the feed this time with the same composition of benzene, but this time it is contaminated not with o-xylene, but same with toluene. So, in this case, what can be performed this particular operation. So, in this particular case, again we start with the same thing.

We find that the P still in this particular case is 1 atm. We have kept the same thing. Corresponding to this the $T_{bub,f}$ is around 85.56 °C and for that α is around 2.552.

So, with this α, we find that in this case also we are going till $x_B = 0.4$. We find that the maximum purity is around 0.815. We are unable to use the single-stage batch distillation still to recover 90 % benzene when it is contaminated with toluene.

The B/F is also around 0.15 which is a bit low.

So, therefore, this suggests that when benzene is contaminated with toluene when the alphas are not very far off rather, it is not much higher than 3 in that particular case the single stage is not a very good option. For such cases either we have to reduce the pressure which is not always economical, for such situations we have to go further multistage column.

One small thing I would like to mention, which I had forgotten to mention this particular case. See when I was doing this initially I had assumed an alpha of, I had assumed an alpha of 7.45. At the end what I found was that for x B equals to 0.4, the T bubble point for the bottoms was equal to 105.1 °C.

And therefore, the alpha at the end we if you calculate you will find that is equal to 6.5582. As a result, we had used the alpha average in this particular case. We had done the entire calculations with an alpha of 6.9929 and the graphs have been generated from that particular α.

For the case of toluene also, we had made the check. It was not extremely much, they were not extremely different initial alpha was say 2.55 to the end alpha was 2.468. So, therefore, we have in this case also the curves had been generated with an alpha average, but if you can do it with the initial alpha also it is not bad.

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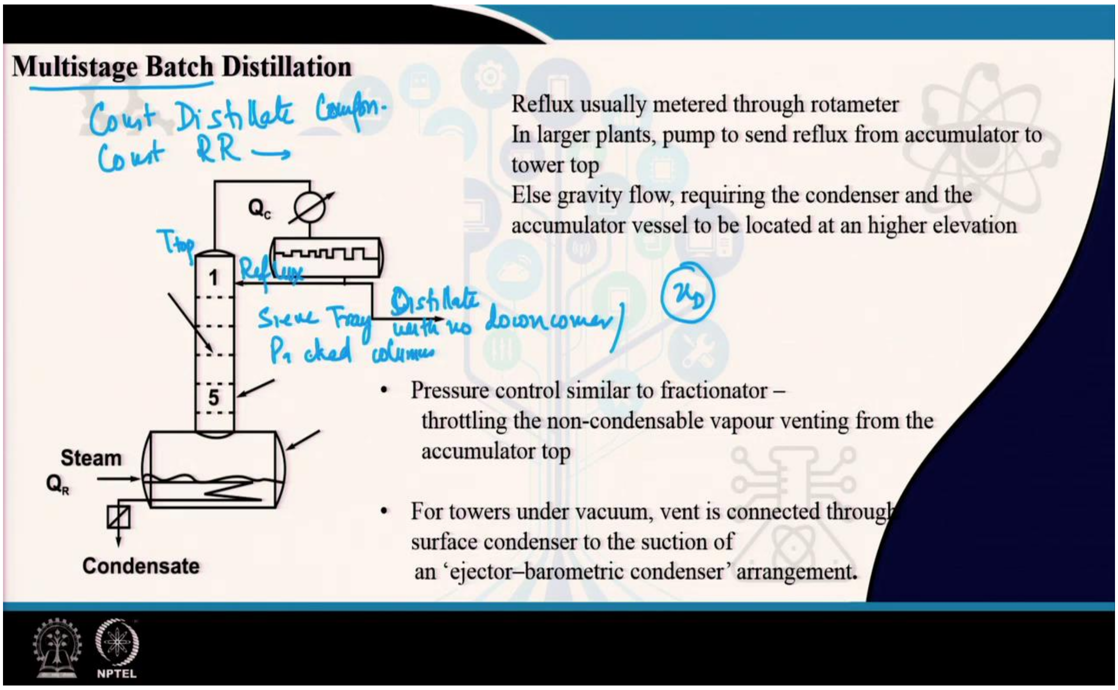

Multistage Batch Distillation

Const Distillate Compn.
Const RR →

Reflux usually metered through rotameter
In larger plants, pump to send reflux from accumulator to tower top
Else gravity flow, requiring the condenser and the accumulator vessel to be located at an higher elevation

- Pressure control similar to fractionator – throttling the non-condensable vapour venting from the accumulator top
- For towers under vacuum, vent is connected throug surface condenser to the suction of an 'ejector–barometric condenser' arrangement.

But definitely under this condition, we find that we have to go for a multi-stage distillation column. We have a tower above the still. In this tower either we have sieve trays with no downcomers. Since, we need very few trays, so, therefore, tray efficiency is not of concern.

So, we either use a sieve tray with no downcomer or we used packed columns in this particular case. So, the vapour which is generated rises up, reaches the condenser comes to the accumulator just like a fractionator. We withdraw some amount and we introduced it as the reflux.

The rest amount is withdrawn as a distillate. This reflux comes in contact with the rising vapour enriches the vapour further. So, therefore, this particular system is similar to the fractionator with only the rectification section.

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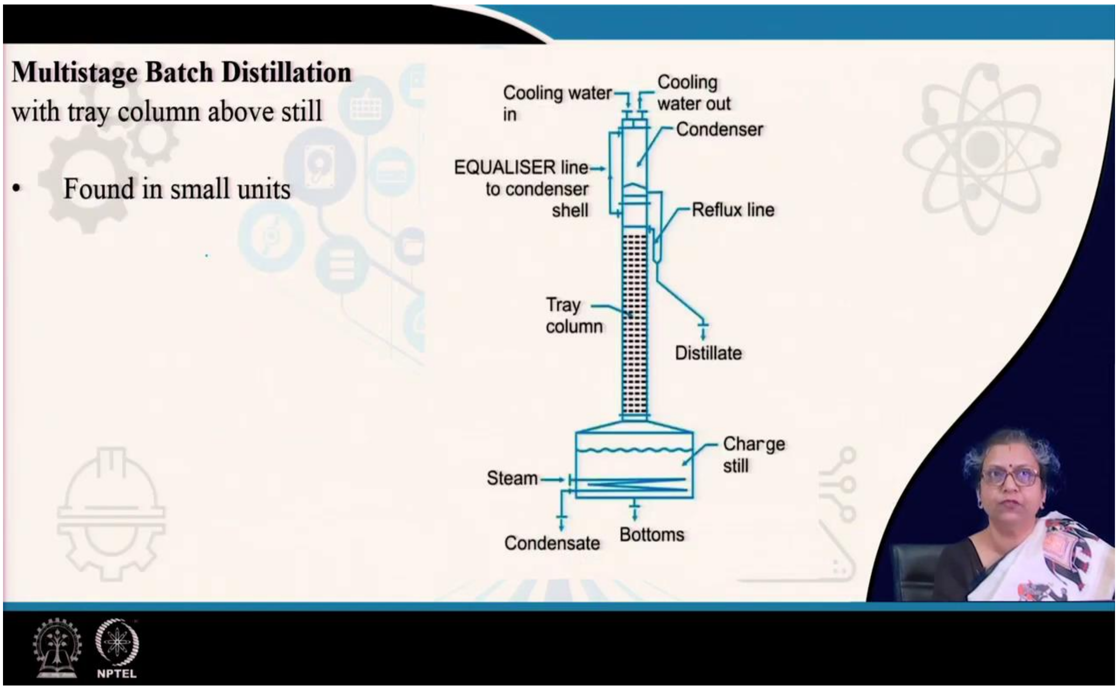

Multistage Batch Distillation
with tray column above still

- Found in small units

So, we usually for small units what do we find? Such small units the condenser, and the column are located just above the still. For larger systems, it is usually located by the side.

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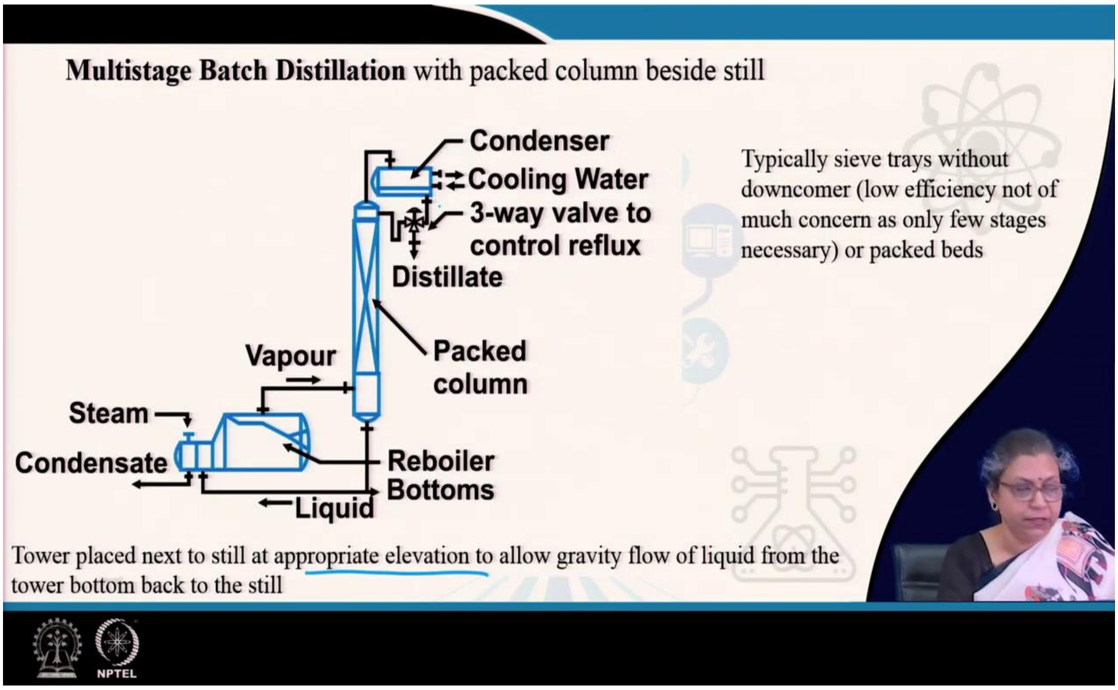

Multistage Batch Distillation with packed column beside still

Typically sieve trays without downcomer (low efficiency not of much concern as only few stages necessary) or packed beds

Tower placed next to still at appropriate elevation to allow gravity flow of liquid from the tower bottom back to the still

But when it is located at the sides, it must be located at an appropriate elevation, such that when the liquid is falling down from here it can enter this still by gravity flow.

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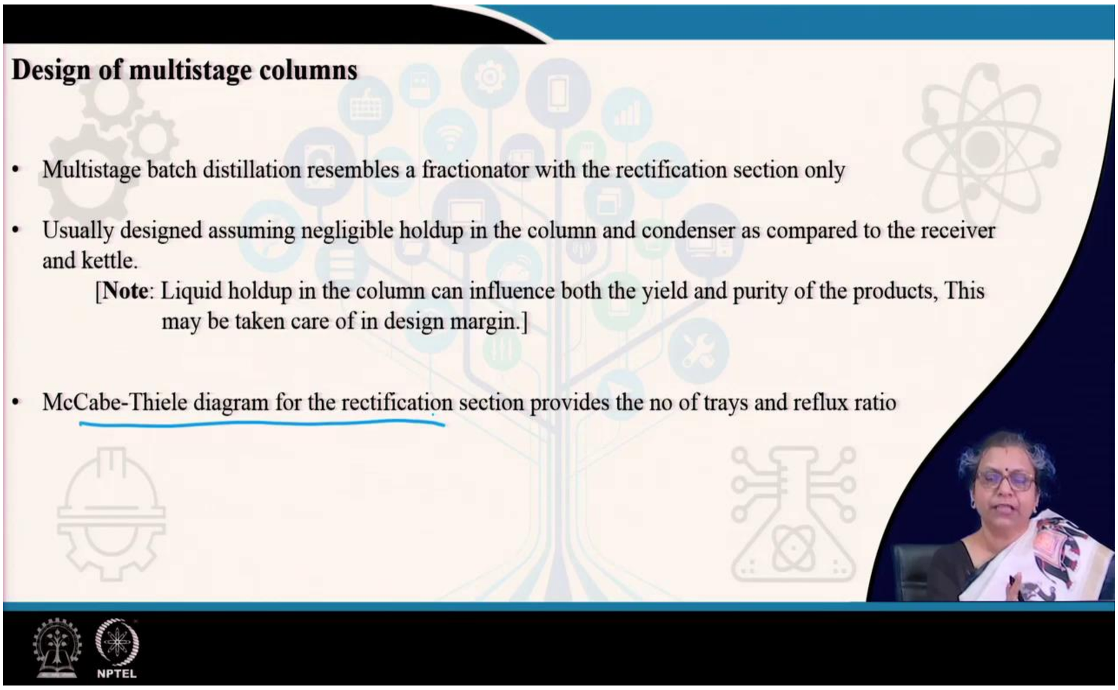

Design of multistage columns

- Multistage batch distillation resembles a fractionator with the rectification section only
- Usually designed assuming negligible holdup in the column and condenser as compared to the receiver and kettle.
  [**Note**: Liquid holdup in the column can influence both the yield and purity of the products, This may be taken care of in design margin.]
- McCabe-Thiele diagram for the rectification section provides the no of trays and reflux ratio

Since it is the rectification section of a fractionator. So, normally we use the McCabe-Thiele diagram for the rectification section to obtain the number of trays and the reflux ratio.

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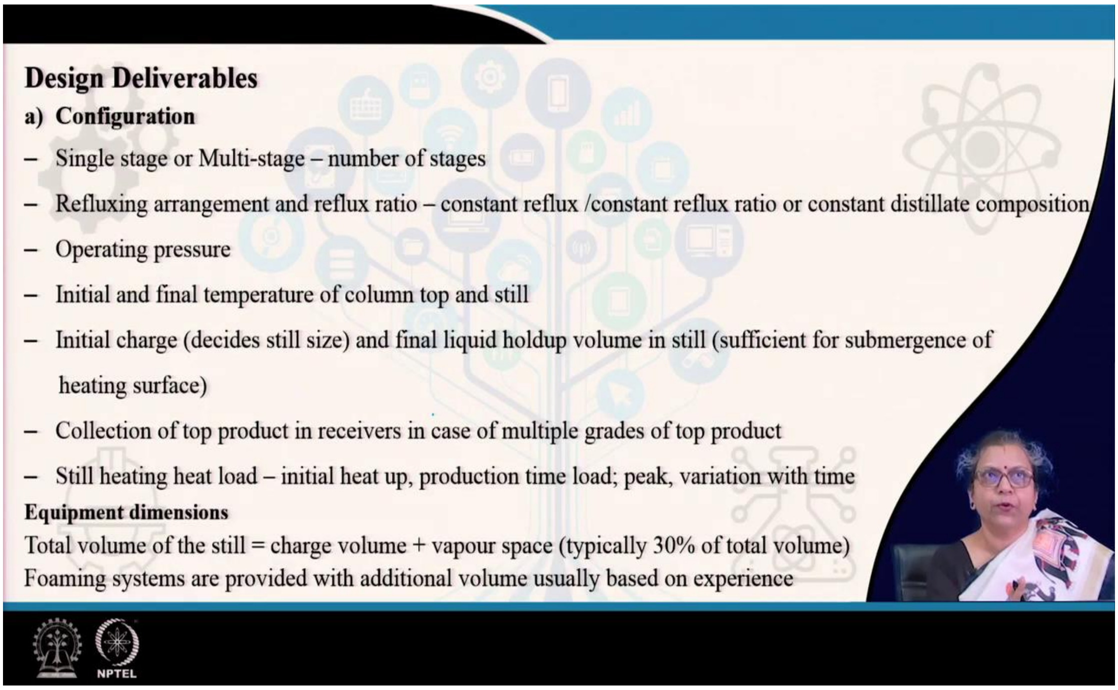

Design Deliverables

a) Configuration

- Single stage or Multi-stage – number of stages
- Refluxing arrangement and reflux ratio – constant reflux /constant reflux ratio or constant distillate composition
- Operating pressure
- Initial and final temperature of column top and still
- Initial charge (decides still size) and final liquid holdup volume in still (sufficient for submergence of heating surface)
- Collection of top product in receivers in case of multiple grades of top product
- Still heating heat load – initial heat up, production time load; peak, variation with time

Equipment dimensions

Total volume of the still = charge volume + vapour space (typically 30% of total volume)

Foaming systems are provided with additional volume usually based on experience

I would like to mention that with the reflux, the column is operated under two conditions. One of them is that when the distillate product has to be it is extremely, it is very important. For that particular case we keep the distillate composition the same, we vary the reflux ratio, and we control it by controlling the top temperature because the top temperature is going to if this is kept constant then x_D is kept constant.

So, this is kept constant by wiring the reflux ratio and we continue this particular operation till the point we get. We find that the reflux ratio has reached an uneconomically high value. But the easier thing to do is when we can keep the reflux ratio constant. So, under that condition, the x_D keeps on varying. We keep the reflux ratio constant and we continue the operation till x_D falls below a specified value.

So, therefore, when we are going for multi-stage batch distillation, the operation can operate by two ways. It can be by keeping the constant distillate composition and varying are or it can be by maintaining a constant reflux ratio where the distillate composition varies. This is easier to do and therefore, this is practised. Only when there is a strict specification on the distillate composition, we go for this particular operation.

So, I think this much for today. We have discussed a single-stage batch distillation, and we had also done a problem just to show the application of Rayleigh’s equation. I have also touched upon the concepts of multi-stage batch distillation.

Thank you very much.