Principles and Practices of Process Equipment and Plant Design

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Module - 02
Lecture - 08
Distillation

Well, good day to all of you. The basic introduction on the mass transfer processes and also some inside into the equilibrium considerations, which was a recap of the thermodynamics course, were discussed in the previous classes. So, today we are going to take up each process and discuss it in detail.

Now, since distillation is the most widely used separation process in the industry. So, first, we will be starting with distillation.

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Now, I had already mentioned that distillation, is a process of separating miscible, binary, or multicomponent mixtures into two or more fractions. It can be two fractions. It can be more than two fractions.

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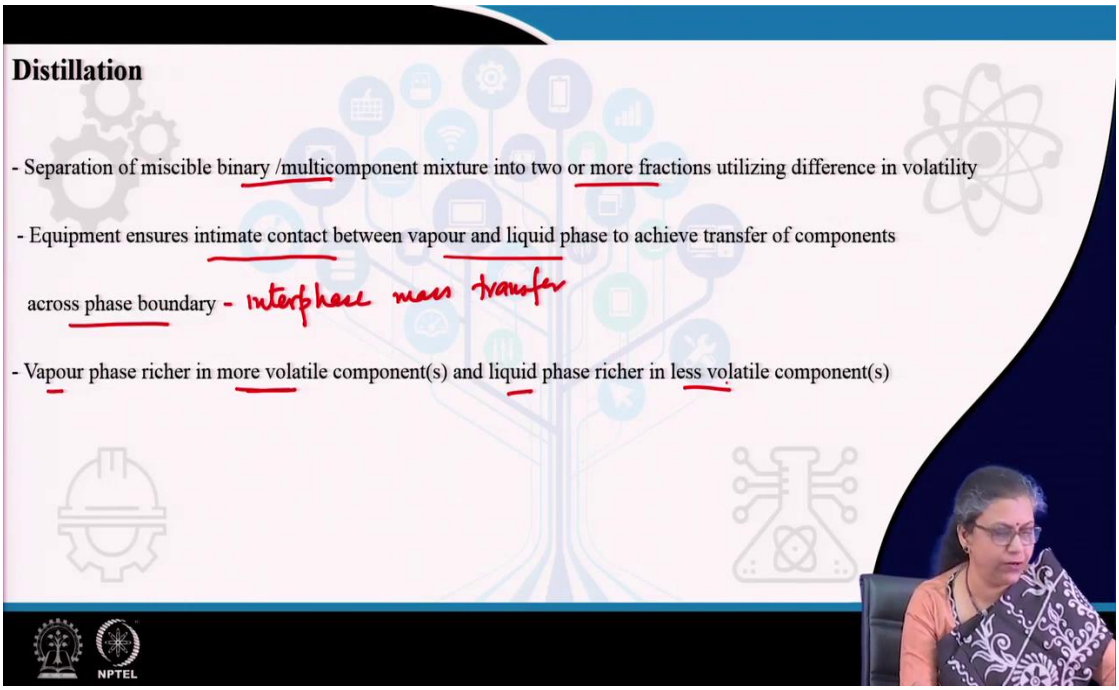

So, therefore, it is the separation of miscible or multicomponent mixtures into two or more fractions by utilizing the difference in the volatility of the components. In the distillation, two phases are first brought into intimate contact.

So, therefore, in this case, the second phase which is the vapour phase if we have the liquid feed, it is a liquid phase if you have the vapour feed and the second phase has been created from this first phase by transfer of energy. After the creation of the two phases, the equipment ensures intimate contact between the vapour and liquid phase.

What does an intimate contact indicate? There will be the transfer of components between the two phases and this transfer of components will occur by inter-phase mass transfer.

So, therefore, in this particular case, the mass transfer occurs due to interface mass transfer across phase boundary. As a result of this interface mass transfer, the vapour phase becomes richer in the more volatile components, or more volatile components cross the phase boundary from the liquid and into the vapour phase. The less volatile components cross the boundary from the vapour to the liquid phase.

As a result after the end of the distillation process, what do we get? We get a vapour phase richer in the more volatile component and the liquid phase, which is richer in the less volatile component.

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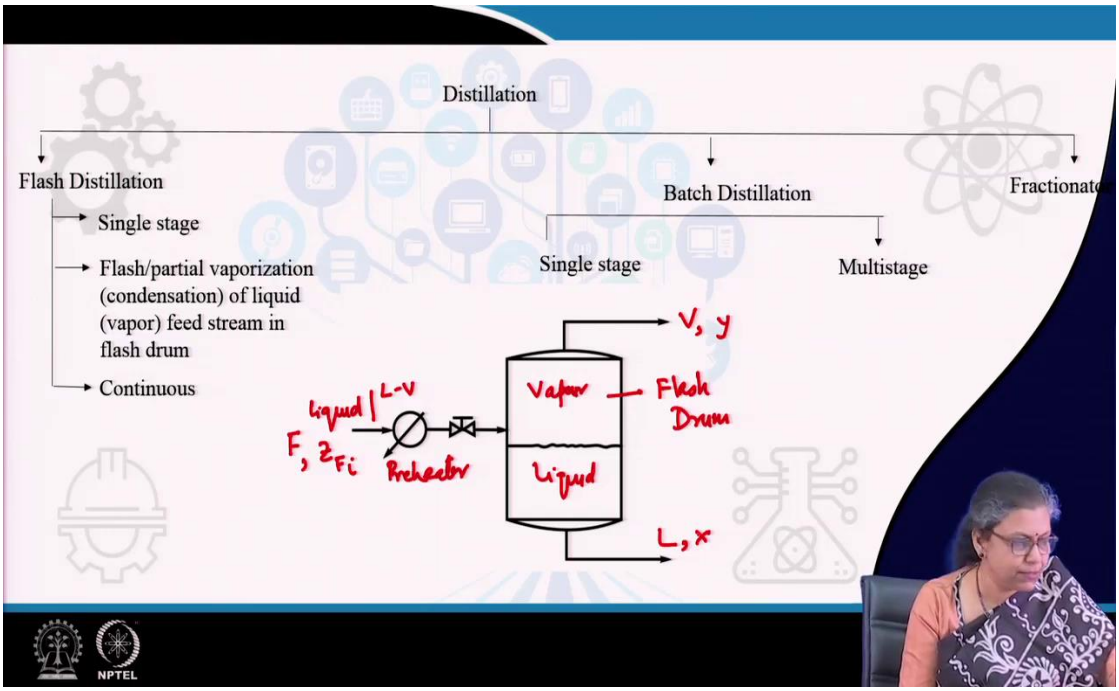

Now, distillation can be of different types. It can be a single-stage distillation, it can be a multistage distillation, it can be a batch process, it can be a continuous process.

Single-stage distillation is the most simple equipment of distillation. Inside a single-stage distillation column, the distillation takes place in a flash drum. We can have a feed of liquid phase or a mixture of vapour and liquid phase. We can also have a vapour phase. In this particular flash drum, the liquid feed or the vapour feed or the liquid-vapour mixture feed can be introduced. Generally, we denote the feed flow rate as F and the composition as z_F for each i^{th} component. The reason for selecting it as z and not x or y is just to show that the feed can exist in any of the phases. When it is only a liquid phase naturally z_{Fi} becomes equal to x_{Fi} and when it is only a vapour phase z_{Fi} becomes equal to y_{Fi} .

Generally, the liquid feed is passed through a preheater, and from the preheater. It gets heated to an extent where it is almost in saturated conditions. Here, we try to ensure that the phase separation does not occur. After that, the feed is throttled across the throttling valve. Then, it is introduced into the flash drum naturally. As inlet is under saturated

conditions and here in the flash drum it separates into two phases; the vapour phase which goes out from the top and the liquid phase which is drawn out from the bottom.

The composition of the vapour phase (V) is denoted by y. The composition of the liquid phase (L) is denoted by x. This particular process is always continuous and this is the process that we use. This is the simplest distillation process.

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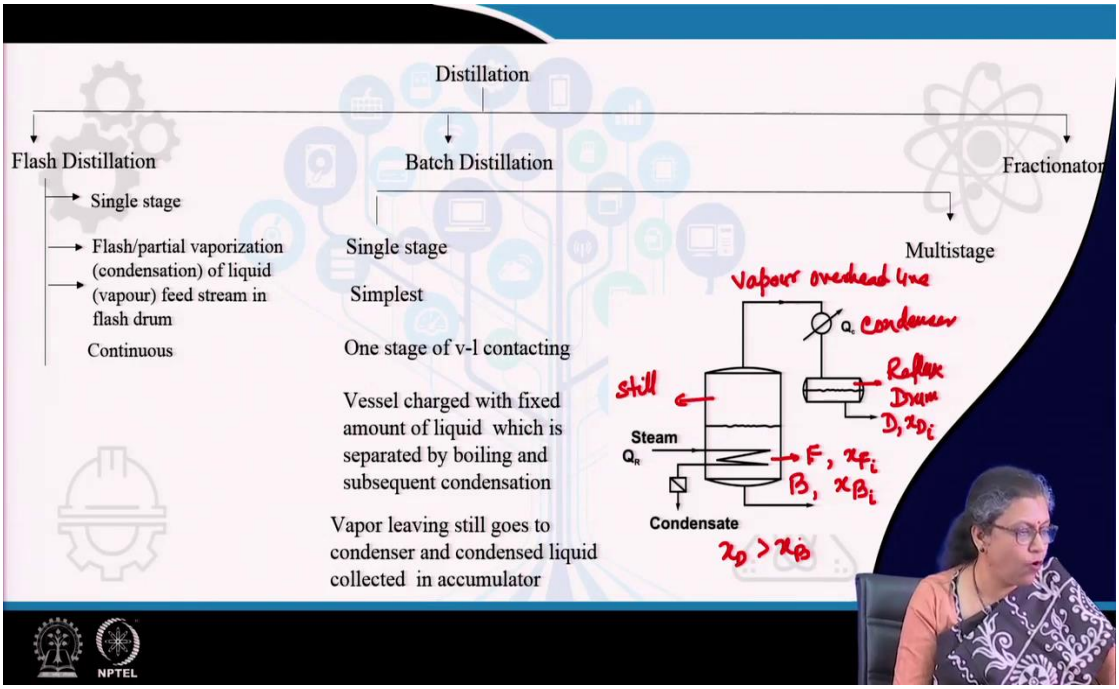

Now, after the flash distillation process, there is the other distillation process among which is the next simple process is the batch distillation process. Batch distillations processes are gaining increasing importance nowadays, due to the increasing application of chemical engineering principles in the production of fine chemicals, pharmaceuticals, food industries, etc.

So, therefore, if you go to an oil refinery you will find fractionators. While in these particular industries (fine chemicals, pharmaceuticals, food industries) where small-scale production is performed and their product quality must be controlled. Under those particular circumstances, we generally go for the batch distillation process.

Now, in batch distillation, as it's quite evident that it is a batch process. So, therefore, in its simplest form, what do we have? We have a still into the liquid of known composition

is introduced and then this is heated. It can be heated either by the introduction of coils or maybe the vessel can be jacketed.

A condensing liquid from maybe some other part of the plant or maybe condensing steam is usually used for providing the heat. Now, once it is heated then naturally what happens? The vapour forms, and it rises up through the vapour overhead line, and from the vapour overhead line it goes to the condenser.

In the condenser, the vapour is condensed and then it is collected in the reflux drum, and from the reflux drum, we collect the product. It is usually known as a distillate with the composition of x_D. The inlet liquid flow rate is say F, and the composition is denoted by x_F. In the end, whatever is remaining is denoted as x_B. For binary feed, x_D, x_F, and x_B refer to the more volatile component in the distillate, feed and bottom (residue) respectively. If it is a multi-component feed then for each component we denote the composition of the ith component by a subscript ‘i' associated with each particular composition.

Now, for a binary mixture as I have told you that since the compositions refer to the more volatile component. So, it is quite natural that x_D is greater than x_B ($x_D > x_B$) and in this particular way, we attain the separation. Now, this is the simplest as I have told you. This is a single-stage vapour-liquid contacting.

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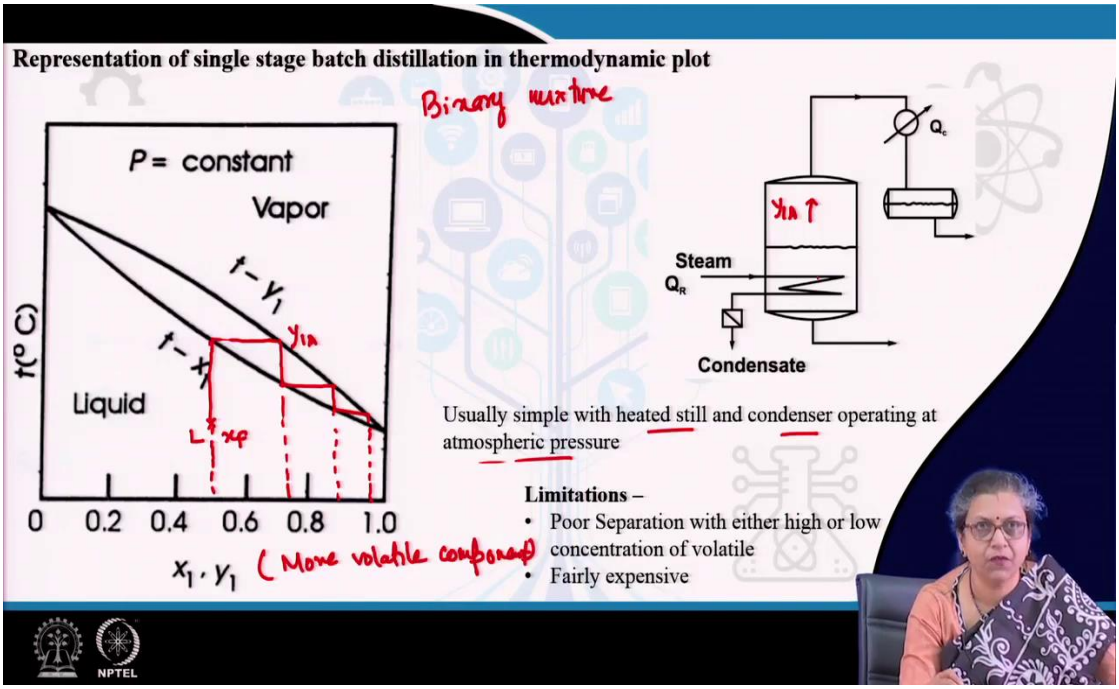

To understand this better just I had wanted to show you the process on a thermodynamic plot. Suppose, we have started from this part we keep on heating. It till we reach the saturated conditions. From the saturated conditions what happens? The vapour forms, this is the liquid feed and its composition is say x_F. This particular plot is definitely for a binary mixture.

It is quite evident, otherwise, we could not have done it and this is plotted in terms of the more volatile component. So, rather x-y they did not, correspond to the composition of the more volatile component and so, therefore, the moment it reaches the saturation state what happens?

We get the vapour which is in equilibrium with this composition. So, therefore, the vapour which is rising from here the composition of this vapour is denoted by this particular say it is y_{1A}. So, the vapour is rising from here (through the top portion of batch distillation still). Then it goes up and it is condensed. When it is condensed say the composition is the same. So, therefore, we had started from this particular composition and the distillate which is richer in the more volatile component. Now, this is very simple this is the simplest type of distillation where it consists of a heated still and the condenser which is operating at atmospheric pressure. But it has some limitations which you can realize by yourself.

You can very well understand that the maximum separation that we can get from here is the equilibrium composition in equilibrium with the feed composition. You also know that under practical situations it is going to be lesser than this. Also, along with that, we know it is fairly expensive.

So, therefore, if you want to enhance the distillate composition or if you want to make the distillate richer in the desired component or the more volatile component what we can do? The distillate can distillate further and for that, we need to repeat the distillation process. What do we get again from this liquid? We get a vapour that is richer in the more volatile component and it is in equilibrium with the feed liquid.

The moment we condense the next stage vapourized product we find that we have achieved additional enrichment. If we keep on repeating this then definitely we would get finally, a product which will be almost comprising of the almost pure more volatile component. So, therefore, redistilling is one particular way by which we can enhance the purity of the distillate.

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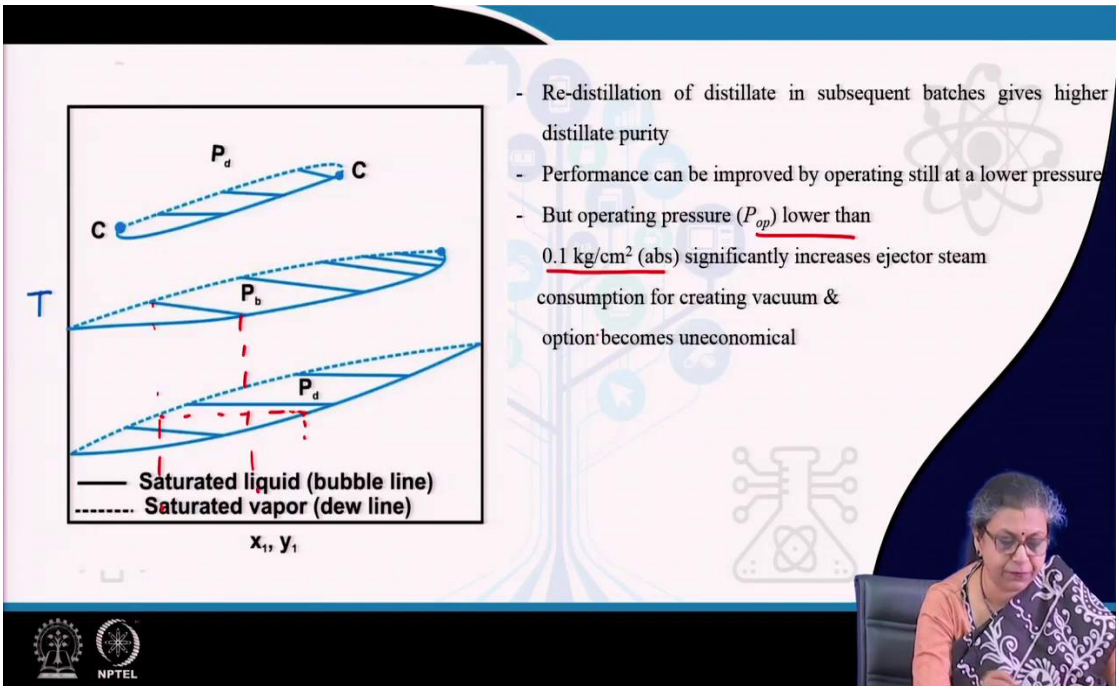

Apart from that what else we can do to improve the performance? See, it is quite evident that suppose, we go for lower and lower pressure. This particular T-x-y diagram is constructed, say at a pressure of 1 atmosphere or 1.5 atmospheres; suppose, we decreased the pressure what do we find when we decreased the pressure?

Firstly, is the difference between the bubble point and the dew point increases for any particular composition or in other words the T-x-y curve the there is a greater gap between them which shows that in this particular case the separation that we get and at lower pressure we get much more separation.

So, we can operate at a lower pressure and improve the performance of the batch distillation. But in this case, you have to remember one thing suppose, we go for a very low pressure say for a pressure lower than say 0.1 kg per cm^2 (abs).

What do we find? We find that under that condition the still it has to be operated under vacuum. Usually, the vacuum is created by an ejector which consumes steam and we find that the steam consumption is increased significantly when we go for very low pressures or rather pressures much lower than 0.1 kg per cm^{2,} and under that condition, this option becomes uneconomical.

So, under that condition see, as I have told you that nowadays batch distillation is gaining importance.

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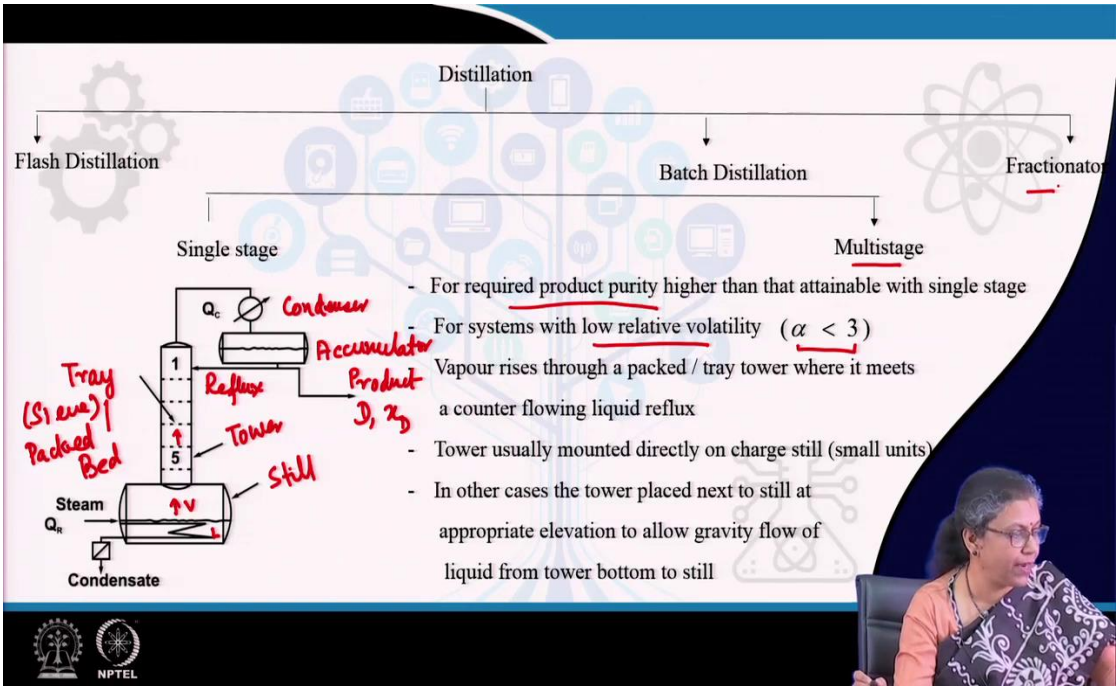

So, therefore, for increasing or rather improving the performance what we can do? We can opt for a multistage batch distillation column. Usually, this is opted when the required product purity is higher than that attainable with a single stage. In a single-stage, the best product that we can attain is in equilibrium with the feed composition.

If we want the product purity higher than that then definitely we have to opt for a multistage. The way I have shown you is that if the difference between the T-x and T-y curve is very close or in other words for low relative volatility, a thumb rule is for alpha < 3, for these particular systems we opt for multistage batch distillation. What is this multistage? We have still over which we have to put a column. Now, this is a tower or a column.

Now, here we can either have trays or we can have packings normally, this column is not very big. Usually, for such a column, we either use sieve trays, or other words we can also use a packed bed here. The details of the sieve trays, packed bed, etc. will be discussed after we finished the mass transfer operations. Then in a consulate form, we are going to

discuss the different tray and packed towers. There you will be getting more idea regarding these two right.

So, therefore, what happens? It is the same thing, steam is there. Steam provides the heat and then the vapour rises from here the liquid remains in still. Now, what it does? It rises through the tower, it reaches the condenser. It gets condensed then the condensed liquid comes and it is collected in the accumulator or other words it is known as the reflux drum.

Now, from the accumulator, we get one product that is richer in the more volatile component some amount of this particular product we take it out say the flow rate is D or rather the yield is D and the composition is x_D. Some portion of this product we recirculate back into the column this particular portion is known as the reflux.

Now, this reflux is introduced. What is this reflux? It is richer in the more volatile component. So, definitely when the vapour is flowing up and it comes in contact with the reflux then what happens. There is an additional mass transfer between the reflux and the vapour. So, when there is an additional mass transfer what happens from the reflux? More volatile components diffuse in the vapour phase from the vapour less volatile components come into the liquid phase.

As a result, we get enhanced enrichment of the vapour phase, and in this particular process just by the repeated distillation that I have shown you, due to this additional enrichment we can get much better or rather much better product quality from the multistage batch distillation as compared to the single-stage batch distillation. Now, usually, these are small-scale units and for most cases, we find that the tower is just mounted directly on the chart stills.

Vapour rises by gravity and generally, the condenser and accumulator are kept at such an elevation that the liquid is reintroduced into the tower and it falls again by gravity. It can also happen for bigger units that the tower is just placed next to the still but at an appropriate elevation such that gravity flow of liquid from the tower bottom to the still can happen.

The other distillation processes are also used. Now the most common process as I have told you is fractionators.

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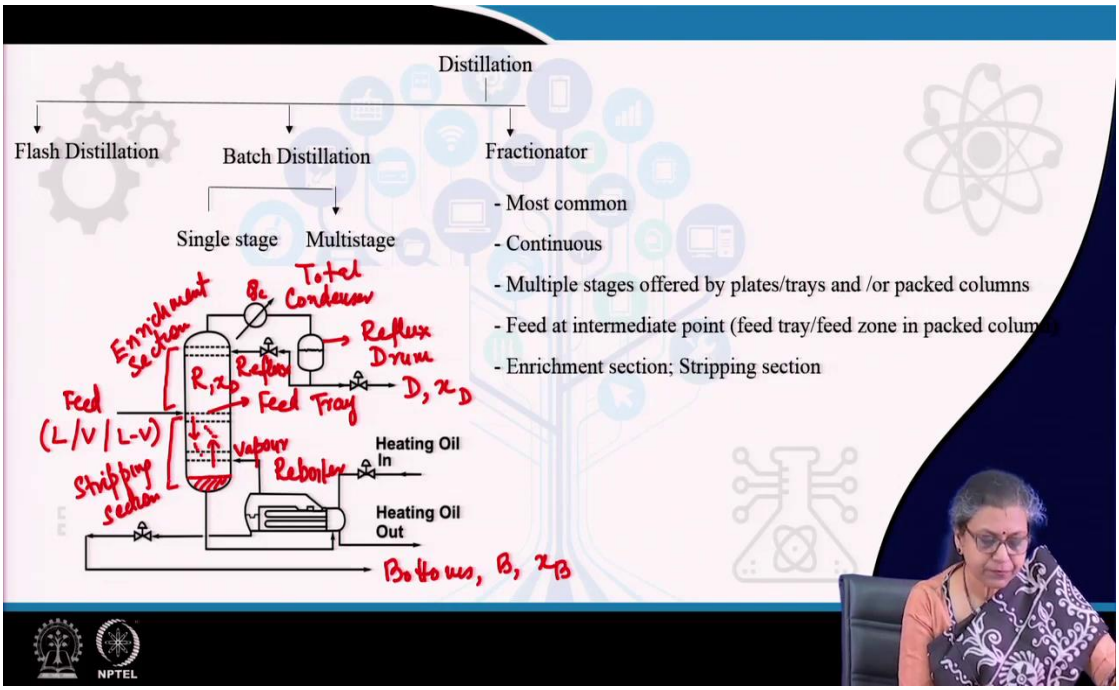

These fractionators are the most common and they are continuous processes here. There is a cascade of multiple stages that we had already discussed. Now, this cascade of multiple stages they are provided either by trays. Usually, big distillation columns whose diameters are greater than 1 meter or so, are provided with trays.

For improving vapour-liquid mass transfer and for ensuring smaller bubbles of the vapour, the trays are perforated and the perforations can be of different-different attachments or accessories like bubble caps, valve trays, etc. These arrangements improve the mass transfer.

Now, in this particular column, the feed is usually introduced in a central portion which is usually known as the feed tray. For a packed column, this is known as a feed zone. The feed may be in different forms like in liquid, vapour, or may be in a mixture of vapour-liquid states. Now, once it is introduced, the vapour part rises just like in the multistage batch distillation. Vapour rises through the overhead vapour line, it reaches the condenser. In the condenser cooling occurs and vapour gets condensed and then it collects in the reflux drum which is also known as the accumulator.

From the reflux drum as I have said a part of the product is drawn out. It is known as the distillate (D). The composition of distillate is denoted by x_D. It is re-introduced into the

column as reflux which is denoted as R. The composition, say it is the same composition when this reflux is introduced. Then again this reflux comes in contact with the rising vapour and it enriches the vapour. So, therefore, by this particular process, we get the high purity of the product. So, the section above the feed where enrichment of vapour occurs. This is known as the enrichment section. In this section, the reflux needs in a counter-current fashion with the vapour and enrichment occurs.

Now, what happens below the feed tray? Here the liquid comes down, this liquid if it has to be enriched it has to come in contact with a counter-flowing vapour in this case. So, from where is this vapour generated? The liquid flows down and it comes to the bottom-most tray. From the bottom-most tray, it is withdrawn and then it is sent into a reboiler. What does the reboiler do? The reboiler partially vapourizes the liquid which comes. We can use different sorts of heating mediums. It can be a shell and tube heat exchanger. There can be different designs of the reboiler which will be dealing in detail when we discussed the distillation column.

We can have thermosyphon reboilers, we can have kettle-type reboilers. In this reboiler what happens vapour is generated. That vapour is again reintroduced into the column just below the bottom tray. Just above the liquid hold up in the bottom of the tray. So, this vapour what it does? This vapour rises through the column and it meets the liquid which is falling down the column.

So, therefore, there is a counter-current flow in the bottom section. There is again mass transfer between the vapour and the liquid occurred and as a result, more volatile components move from the liquid to the vapour phase. Less volatile components move from the vapour to the liquid phase. As a result mass transfer occurs vapour becomes richer in the more volatile component then it rises up. Then in the enrichment section, the enrichment of vapour to the more volatile component continues as it comes in contact with the reflux.

Finally, with all this enrichment we get an almost pure product from the feed that we have started. What will be the purity that will get from the feed composition, and the flow rate, with which we have started, that we can achieve by deciding upon the number of stages inside the column.

So, depending upon the purity, we decide the number of stages initial calculations. Assume that on each tray the vapour and liquid which are leaving are in equilibrium.

That means we perform the calculations assuming ideal contact. We know fully well that ideal contact or equilibrium trays do not exist. So, after we have found out the number of ideal trays then we consider a fraction of overall efficiency and we then compute to find out the actual number of trays, which will be required to achieve that particular separation. So, therefore, the section where the vapour is stripped of the less volatile components. This section is known as the stripping section.

So, therefore, the distillation of the fractionator column is in its most simple form. This has got a feed tray, enrichment section above, and stripping section below this and then the liquid which enriches the vapour is supplied from the vapour which is condensed and collected in the reflux drum and this is provided as reflux. In the same way, the vapour, which forms in the reboiler, is once more introduced here and that also provides the additional separation. Now, it's important for you to remember that while mostly we operate with total condenses, what does this total condenser mean? Total condenser means whatever vapour comes out from the top of a column, usually, the entire vapour is condensed. Part of the condensate is sent as reflux and part of is withdrawn as distillate.

On the other hand, in the reboiler, we find that we undergo partial vapourization just to obtain the vapour, which is required to be introduced in the distillation column the remaining portion of it is withdrawn as a bottoms product flow rate B, and the bottom composition as x_B.

So, therefore, the reboiler also serves as a partial vapourization unit where the liquid is partially vapourized, to ensure vapour and the liquid are in contact and the vapour is introduced back into the column and further, the liquid part is withdrawn out.

So, therefore, the reboiler also acts as a single-stage. Therefore, when you are calculating the number of ideal stages, we may have to remember that the reboiler also accounts for one particular stage here.

The reboil stripper is nothing the reboil stripper, where we do not have the enrichment section, feed is introduced from the top, and then it flows down. So, we usually have a stripping section.

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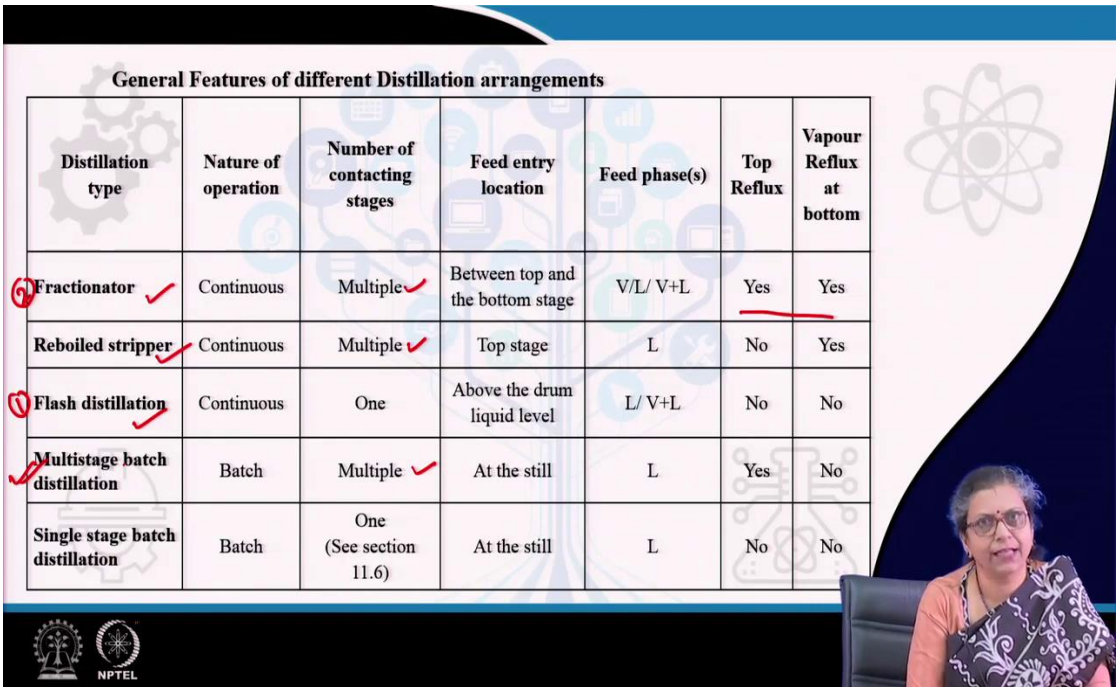

So, for all these cases the fractionator, the reboil stripper, the flash distillation are continuous processes. Fractionator, reboil stripper, and multistage distillation column have multiple stages. Flash and the simplest batch distillation have one stage each. While we discuss the batch distillation you will understand that a single-stage batch distillation performs an enrichment of slightly greater than a single-stage, because when the vapour rises it comes in contact with the pull part above the still and there is some amount of vapour condenses and falls down. So, therefore, the enrichment is almost equal into 1.1 stage. Feed entry location about single-stage batch distillation is at the still. In the fractionator, feed entry is in between the top and bottom stages. In the flash distillation definitely, feed is introduced into the flash drum in the zone just above the liquid level in the flash drum.

The feed conditions are different in different-different distillation types. It is mentioned in the above table. We have already discussed these things.

We require top reflux whenever we want enrichment and vapour reflux for additional enrichment. Both these types of reflexes are there only in a fractionator. We get the

maximum amount of enrichment or maximum amount of separation and therefore, a fractionator is the most commonly used distillation column in industries.

Very recently batch distillation units, specifically the multistage batch distillation units are being increasingly used in pharmaceuticals, cosmetics, food products, fine chemicals, and other similar industries.

You have got an overview of the different distillation processes. What I plan is that we will take up each distillation individually and we will be discussing them.

So, first, we will be taking a flash distillation and how to design the flash drum, what are the inputs we require, what are the outputs we require, what are the control, controls which are required for a distillation drum. Although it is very simple equipment, you will find that there are certain details which need to be considered.

After flash distillation then we are going to discuss the fractionator and after the fractionator, we will be discussing single-stage batch distillation and we will also be touching upon multistage batch distillation columns or batch distillations still along with the column.

So, with this, we end here. In the next class, we go ahead with our discussion on the design of a flash drum. What are the things that have to be considered and how do we proceed with it.

Thank you very much.