Plant Design and Economics
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Lecture No -36
Introduction to Separation Systems

Welcome to module 8 of plant design and economics. Almost always separation systems will follow reactor systems. So after discussing reactor network synthesis in module 7, we will discuss synthesis of separation system in module 8 and module 9. So today we start with separation systems synthesis part 1.

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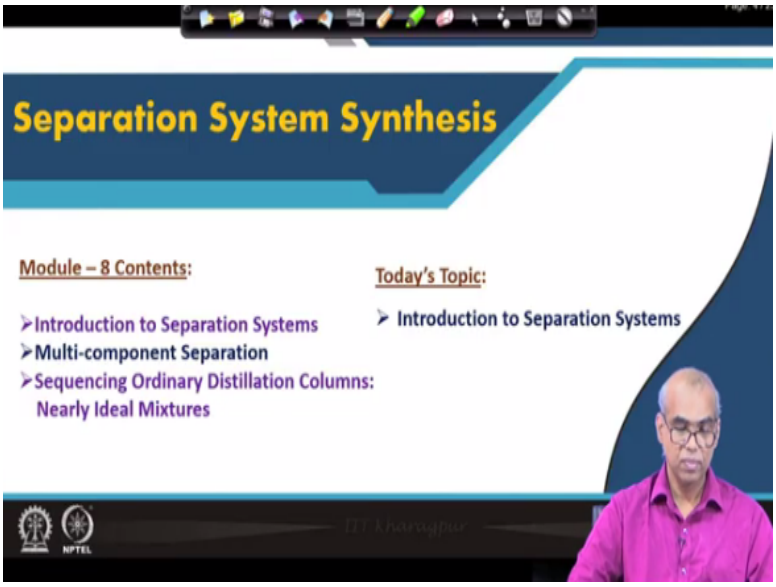

So in this module we will start with the brief introduction to separation system. Then we will talk about multi component separation in particular multi component distillation methods, shortcut methods and then we will talk about sequencing of ordinary distillation columns for nearly ideal mixtures. Today we start with introduction to separation systems.

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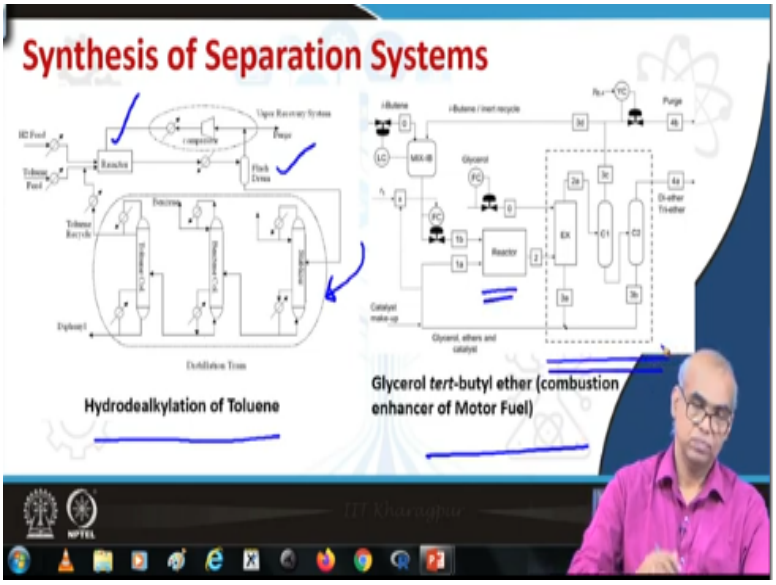

Now, look at this two flow sheets. This is for hydrodealkylation of toluene where Benzene is produced from toluene and hydrogen. We have discussed this in our previous modules. Now the effluence from the reactor undergoes a phase separation and then the separated liquid phase passes through a distillation tray, a sequence of distillation column. So a tray of separation system follows a reactor here.

Again this is another example, another flow sheet for production of glycerol tert-butyl ether which is used as a combustion enhancer of motor fuel. Again you will see that after reactor a separation tray follows. So, synthesis of separation system must be considered after we have synthesizing the reactor network.

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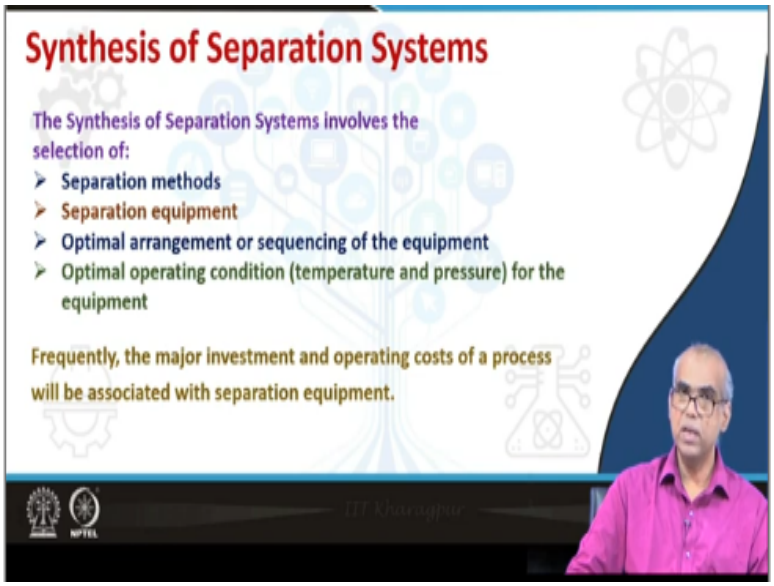

So, what do you mean by synthesis of separation systems? The synthesis of separation system will involve the selection of appropriate separation method, the appropriate separation equipment, the optimal arrangement or sequence of these equipment, optimal operating conditions such as temperature and pressure. Frequently, the major investment and operating cost of a process is associated with separation equipment.

So synthesis of suitable separation equipment is extremely important for process economics of any chemical process industry. So similar to reactant network synthesis, we are interested in determination of separation method, separation equipment, optimal arrangement or the sequence of the equipment as well as optimal operating conditions for this equipment.

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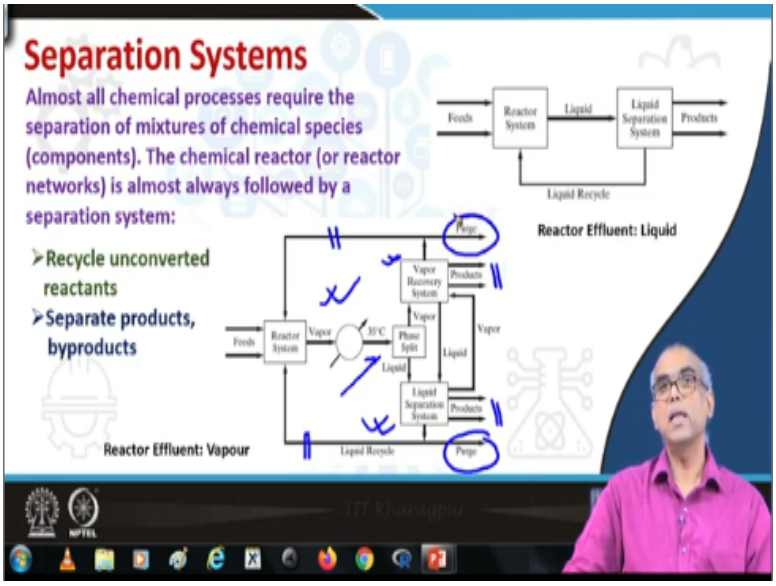

As we say, almost all chemical process require the separation of mixture of chemical species or components. The chemical reactor is almost always followed by separation system. This is because you would like to recycle unconverted reactants. So the unconverted reactants present in the effluent stream has to be separated and recycled back or improved process economics.

You also need to treat the effluence stream to separate products and by-products. So this flow sheet shows you a liquid reactor effluent goes through a liquid separation system from which a liquid stream recycle goes to the reactor and we are withdrawing products. You can also have a vapour effluent from the reactor which can be phase plated and the liquid stream can go through liquid separation system and the vapour phase can go to vapour recovery system.

From both liquid separation system and vapour recovery system, you can take out products and you can send the recycle stream, liquid recycle stream or the vapour recycle stream to the reactor system. We will also have purge stream to avoid building up of any inert species in the recycle loop.

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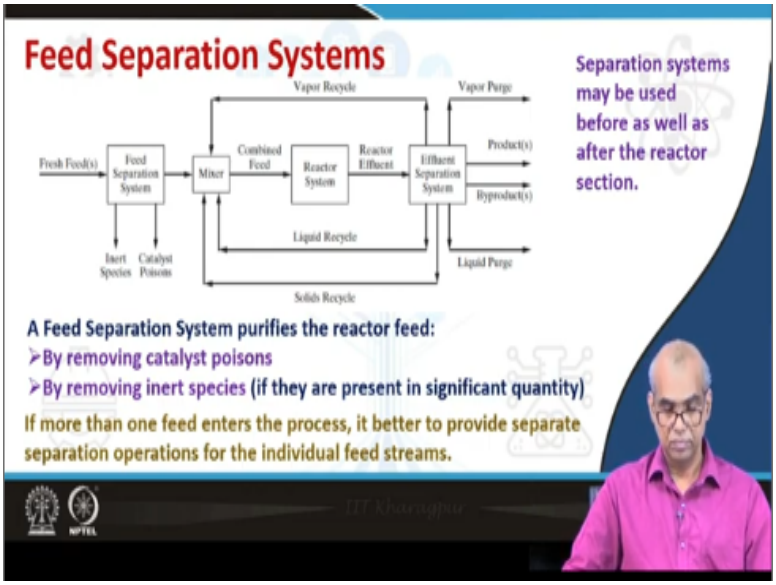

You may also require separation systems for the feed stream. In the previous flow sheets, we have seen the separation systems present after the reactor system. But separation system may be used for the feed stream as well. So my flow sheet or my process can have separation systems before as well as after the reactor systems. A feed separation systems often necessary to purify the feed stream.

Why do I need to purify the feed stream? Because we may have to remove any species that are present in the feed stream which may poison my catalyst or I may also be interested in removing inert species if they are present in significant quantity. If more than one feed streams enters the process, it is better to provide separate separation operations for the individual feed streams.

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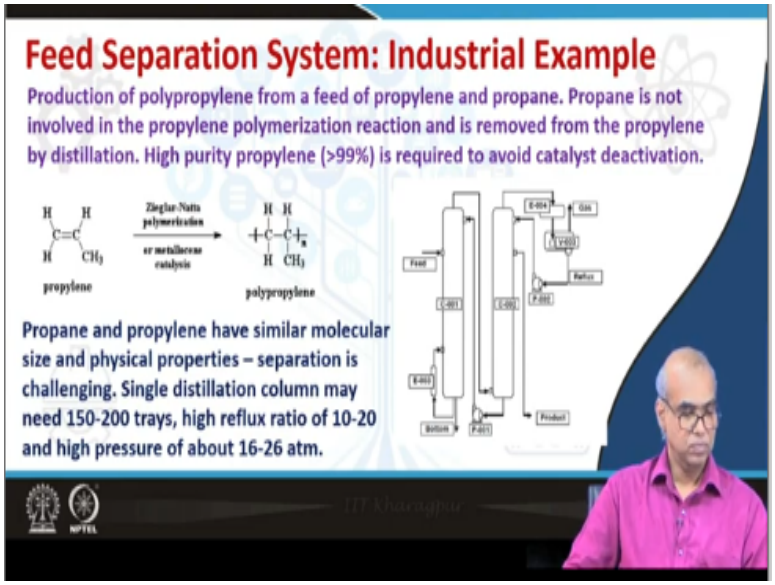

Now, we will take certain examples, certain industrial examples where the feed separation systems are used in practice. As the first example, let us consider production of polypropylene from a feed of propylene and propane. Propane does not participate in the polymerisation reaction, it is the polymerisation of propylene that gives us polypropylene. Propane is removed from the propylene by distillation.

High purity propylene with purity higher than 99% is required to avoid catalyst deactivation. So it is extremely important that we remove propane from the propylene. The catalyst reaction scheme is shown. Now, when it comes to separation of propane and propylene, both the species have very similar molecular size and physical properties. So, this makes separation of these two components very difficult.

Single distillation column if I use, that may require 150 to 200 trays, very high reflux ratio of about 10 to 20 and the high pressure of above 16 to 26 atmosphere. Now, see if I consider the average trays spacing is about 2 feet, so 150 to 200 trays will give me a height of distillation column which is about 300 feet or 400 feet. Now if I use 400 feet or even 300 feet high distillation column, that may lead to structural instability.

So typically, we should avoid as far as possible distillation column with more than 100 trays so that the structural instability associated with very tall towers is avoided. So this particular distillation can be done instead of single unit, we can do it in 2 units. So this is an industrial example where the separation at the feed stream, so separation of the feed stream before the feed enters the reactor is important.

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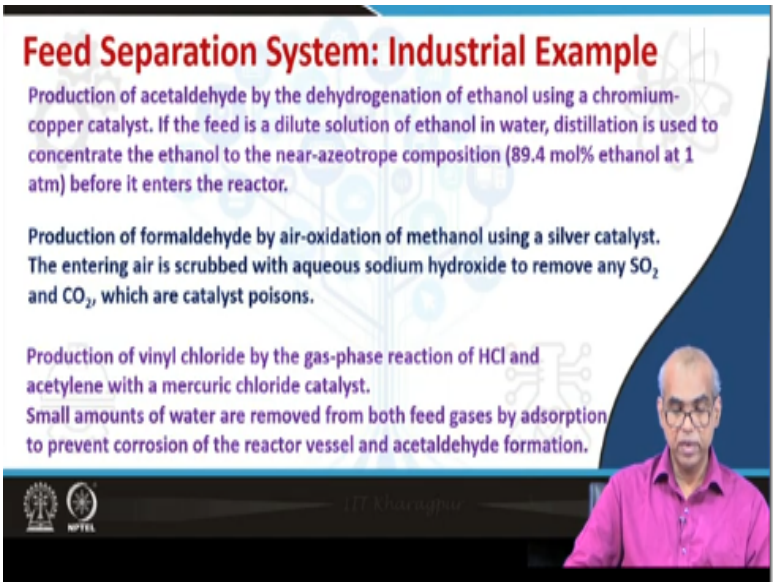

Another example, production of acetaldehyde by dehydrogenation of ethanol using a chromium copper catalyst. If the feed is a dilute solution of ethanol in water, distillation is used to concentrate the ethanol to the near azeotrope composition, which is 89.4 mol percentage ethanol at 1 atmosphere. So before the feed enters the reactor, we can perform distillation to concentrate the ethanol water mixture to the near azeotrope composition.

Let us consider another example, production of formaldehyde by air oxidation of methanol using silver catalyst. The entering air is scrubbed with aqueous sodium hydroxide to remove any sulphur dioxide and carbon dioxide that may be present. Note that sulphur dioxide and carbon dioxide are catalyst poison. So before the feed enters the reactor the feed is purified to remove sulphur dioxide and carbon dioxide.

Let us consider another example, production of vinyl chloride by the gas phase reaction of hydrochloric acid and acetylene with a mercuric chloride catalyst. Small amounts of water are removed from the both the feed gases by adsorption to prevent corrosion of the reactor vessel and acetaldehyde formation.

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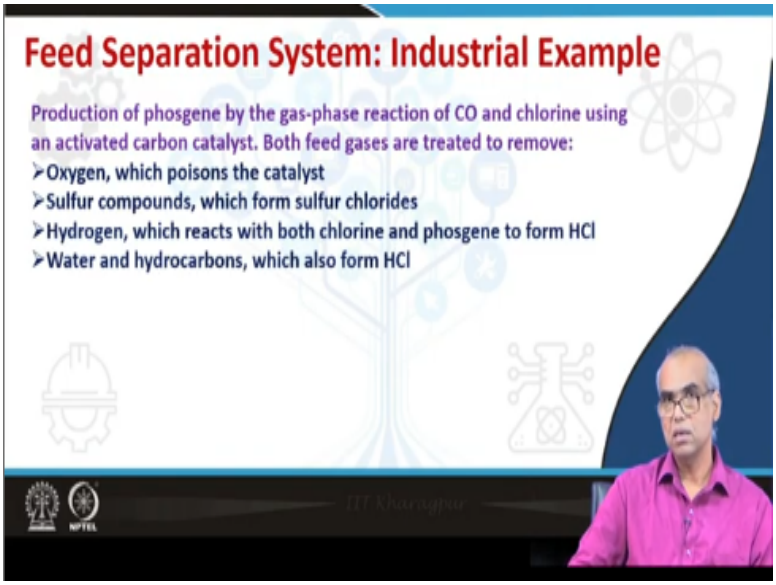

One more example, where feed is purified before it enters the reactor. Production of phosgene by the gas phase reaction of carbon monoxide and chlorine using an activated carbon catalyst. Both feed gases are treated to remove oxygen because oxygen may poison the catalyst, sulfur to remove sulphur compounds otherwise it will form sulfur chlorides, to remove hydrogen from the feed otherwise hydrogen will react with chlorine and phosgene to form HCl and to remove water and hydrocarbons because these also form HCl.

So we have seen several examples, industrial examples where the purification of feed is important before it enters the reactor system.

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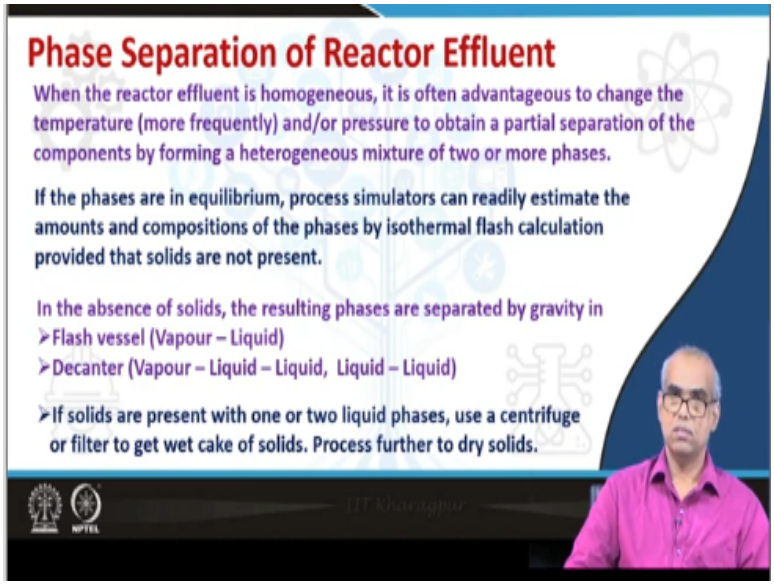

Now let us consider phase separation of reactor effluent. When the reactor effluent is homogeneous, it is often advantageous to change the temperature or pressure for partial

separation of the components by performing heterogeneous mixture of two or more phases. Generally, this phase separation is done by change of temperature that is more frequently done, but also this can be effected by change of pressure.

If the phases are in equilibrium, we can take help of process simulators to readily estimate the amount and composition of the phases by isothermal calculations, isothermal flash calculations provided that solids are not present. So, we have process simulators which can readily calculate the compositions and the amounts of species in this different phases which are in equilibrium with each other.

Absence is a very commonly used process simulators which can be used for such purposes. In the absence of solids, the resulting phases are separated by gravity in flash vessel or decanter. Flash vessel, using flash vessel you can separate vapour liquid mixture stream. Using decanter, you can separate vapour-liquid-liquid or liquid-liquid streams. But if solids are present with, if solids are present with one liquid or two liquid phases, we can use a centrifuge or a filter to get wet cake of solids.

And then this wet cake of solids can be further processed to dry solids and also we can separate the mother liquor from the wet solid.

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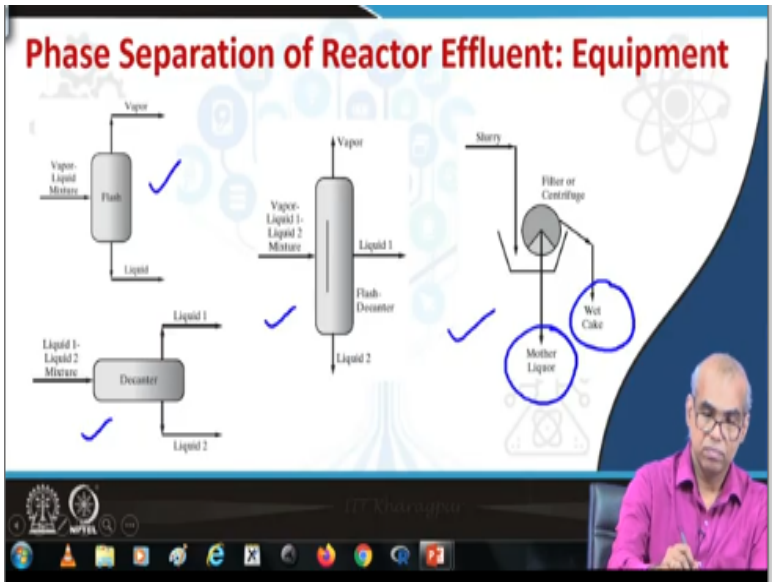

These are some equipment which can be used for phase separations. Flash drum can be used to separate vapour liquid mixture, decanter can be used to separate a mixture of liquid 1 and liquid 2, a flash decanter can be used to separate a mixture which contains 2 liquids and 1

vapour. Filter or centrifuge can be used to separate a slurry containing solids which may be splitted into mother liquor and wet cake. Wet cake can be further processed to obtain dry solid.

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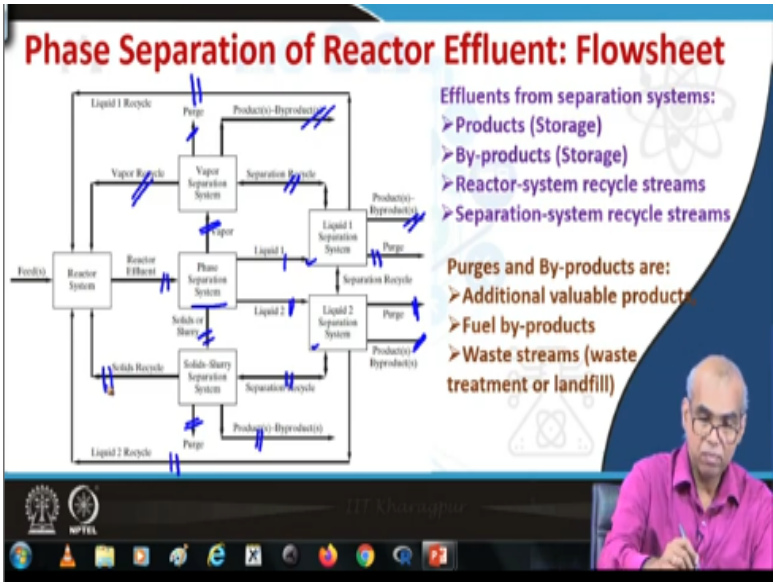

Now this flowsheet gives you details of phase separation of reactor effluence. So the reactor effluent enters the phase separation system. And the phase separation system creates solid steam, vapour stream as well as liquid streams. So note that this phase separation system may not be a single piece of equipment, it may be a network of phase separating units. Now the liquid streams, there are 2 liquid streams shown in the flow sheet.

Both the liquid streams goes to individual liquid separation systems from which we can take out products or by-products. From both the liquid separation systems, recycle stream goes to the reactor and also purge streams are there to avoid building any inerts in the recycle stream. Also, separation recycle streams are there from both the liquid separation system. Now look at the vapour separation system.

From the vapour separation system also we have product or by-product stream, purge stream as well as vapour recycle. The same thing for solids slurry separation system, we have product by-product steam, we have purge stream as well as solid recycle stream. So effluence from separation systems are of 4 types; products which goes to storage, by-products, useful by-products will also goes to storage, reactor system recycle and separation system recycle.

Purges and by-products are additional valuable products, fuel by-products or waste streams which goes to waste treatment or landfill.

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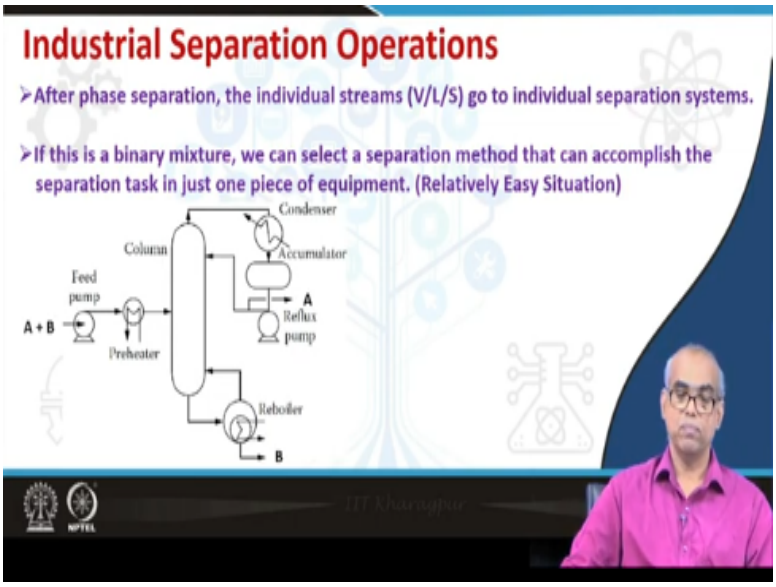

After the phase separation has been done, the individual streams will go to individual separation systems. So that is what we have seen in the previous flow sheet that after phase separation, the vapour stream went to vapour recovery system, liquid stream went to liquid separation system and solid system, solid stream went to solid separation system. So the individual streams from the phase separations go to individual separation systems.

If this is a binary mixture, then you can select an appropriate separation systems that can accomplish the separation tasks in just one piece of equipment and then it gives us a relatively easy situation. For example, if the individual stream that I am talking about is a binary liquid mixture and let us consider that they are not close boiling mixtures, we can very easily consider our binary distillation process to separate this mixture of A and B into relatively pure components A and B.

It is possible to accomplish these separation in a single piece of equipment, for example here in a single distillation unit.

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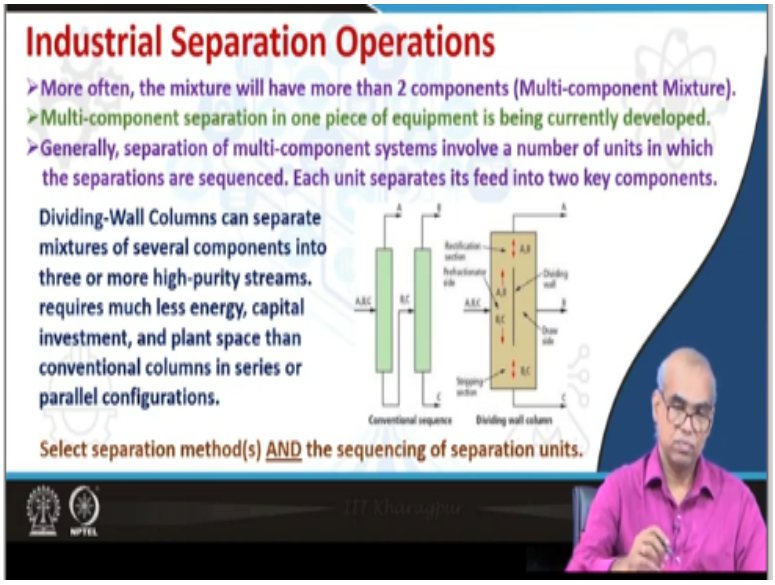

But more often than not, the mixture for industrial applications will be multi component mixture, meaning will have more than two components. Multi component separation in one piece of equipment is being developed correctly. For example, a dividing wall column which is a special type of distillation column can separate mixtures of several components into three or more high purity streams.

So this is a schematic of dividing-wall column. Look at the dividing-wall. Now this dividing-wall column which is a special type of distillation column can be used to separate a three component mixture of A, B and C into 3 different streams A, B and C. If you look at the divide-wall column, you see the rectification section handles the separation of A and B and the stripping section handles the separation of B and C.

So the presence of this divide column effectively performs the job of 2 conventional distillation column. So, such arrangement requires much less energy, less capital investment and also requires less plant space than the conventional columns in series or parallel configuration. So this is an example of process intensification. So while such systems are being developed, but more commonly the current separation of multi-component systems involve a number of units in which the separation are sequenced.

Each unit separates its feed into 2 components. For example, if I have a mixture of 3 components A,B and C, I will require 2 conventional distillation column, where in the first distillation column I have separated A from the mixture of B and C, B and C mostly goes to the second distillation column as feed and where B and C are separated. So this is how it is

commonly done. So, we need to select the separation method as well as the sequencing of separation units.

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Let us now talk about separating agents. Mixing of chemical species is spontaneous process. However, separation of a mixture of various chemical species is not a spontaneous process. We need separating agents. So separation requires an expenditure of some form of energy or mass. So when you use some form of energy we call it energy separating agent and when you use mass we will call it mass separating agent.

So energy separating agent or mass separating agent is required to cause a separation. 4 common industrial techniques are there to achieve separation. First the creation of a second phase by heat transfer, shaft work or pressure reduction. So this is an example of energy separating agent. For example distillation process, cooling crystallization where we introduce energy.

Expenditure of energy is involved whether we heat the feed stream or we cool down the feed stream in case of cooling crystallization. The introduction into the system of a second fluid phase. So introduce a mass, so mass separating agent is involved. For example, a liquid-liquid extraction. In case of crystallization, we have something called anti solvent crystallization. Suppose a solute is dissolved in solvent A.

Now, I add another solvent B to the system where these solvents A and B are soluble but the solute which is soluble in A is not soluble in the added solvent B. So the solubility of the

solute in the mixed solvent reduces. So there will be generation of super saturation and crystallization will occur. So this is also an example of mass separating agent where the anti solvent the second solvent that you have added is the mass separating agent.

The addition of a solid phase on which selective absorption can occur. This is clearly a case of mass separating agent. And finally the placement of a selective membrane barrier. Here also this is energy separating agent because mechanical energy is involved in partition of species through this membrane barrier. Except the membrane separation process, all other separation process such as creation of separation phase or introduction of second fluid phase into the system or addition of a solid phase on a selective absorption take place.

In all these 3 cases the extent of mass transfer is limited by thermodynamic equilibrium between the phases. But in case of membrane based separation process the exiting phases are not in equilibrium with each other. The separation occurs due to the difference in rates of permeation of the species involved.

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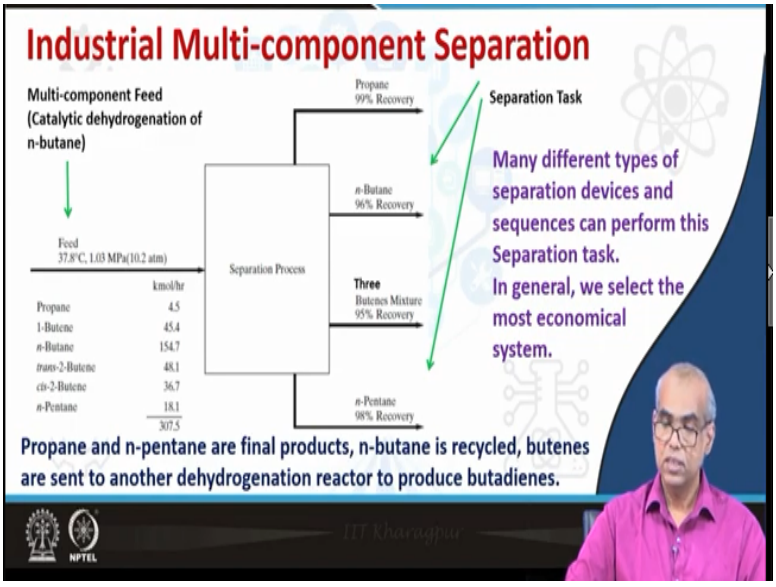

To understand the complexity of multi component separation systems or industrial multi component separation systems, let us take this example. We are considering separation of a multi component mixture of Propane, 1 butene, n butane, trans-2-butene, cis-2-butene and n pentane. This mixture is obtained from catalytic dehydrogenation of n butane. Now this mixture of 6 components has to be separated into a propane rich stream where 99% of the propane in the feed stream has been recovered.

1 n butene rich stream, n butane rich stream where 96% of n butane has been recovered. Then 3 butenes mixtures - 1 butene, trans-2-butene, cis-2-butene, and this butene mixtures must correspond to 95% recovery from the feed. And finally 1 n pentane rich stream where you have recovered 98% pentane from the feed. So this is my separation task. I need this 4 streams.

Propane and n pentane are final products, n butane is recycled to the dehydrogenation unit, catalytic dehydrogenation unit and butenes, you have 1 butene as well as trans-2-butene and cis-2butene, these butenes are sent to another dehydrogenation reactor to produce butadiene. So many different types of separation devices and sequences can perform this separation task and obviously as a process engineer you select the most economical one.

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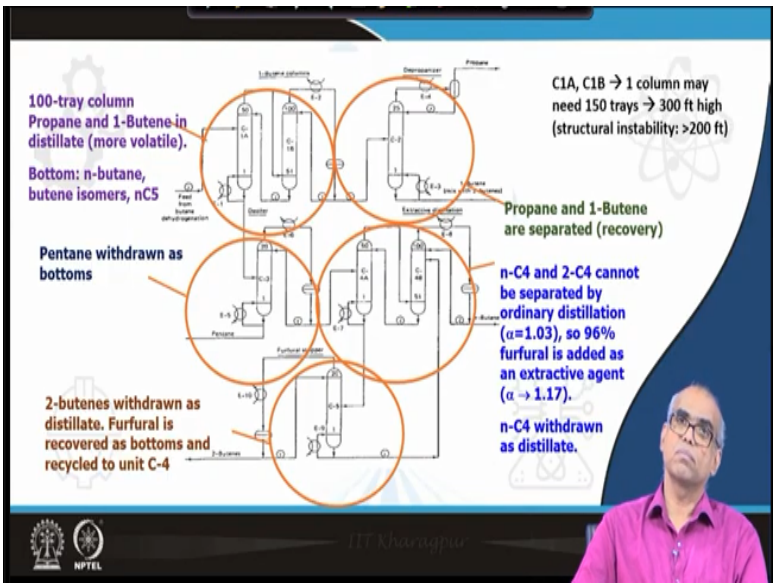

Now, let us consider one separation trays or separation sequence which consists of several distillation columns. Now the feed enters this combinations of 2 distillation column, C1A, C1B. These are 100 tray column. Propane and 1 butene are obtained in the distillate from this 2 distillation trays. Propane and 1 butane, propane and 1 butene are most volatile propane and 1 butene are most volatile among these 6 components. So propane and 1 butene are obtained in the distillate. So, what will be the bottom one? The remaining of the components.

What are those? n butane, then trans-2-butene and cis-2-butene, these 2 isomers as well as n pentane. Note that the other butane, 1 butene and propane has been obtained as dislet from this C1A, C1B column. So this dislet stream goes to distillation column C2 where the propane and 1 butene are completely separated. So they are recovered. Now the bottom

stream from the C1A, C1B column which essentially contains n butane, cis-2-butene and trans-2-butene isomers and n pentane.

This bottom stream goes to column C3 where pentane is withdrawn as bottoms. So the distillate which is n butane and 2 butene isomers, cis butane and trans butane they go to C4A C4B columns. But nC4 that means n butane and 2 butenes cannot be separated easily by ordinary distillation column because of very low relative volatility of 1.03. So what you do here, instead of performing distillation ordinary distillation, we perform extractive distillation.

So 96% of furfural is added here as extractive agent. This analysis the relative volatility to about 1.17. So it is now possible to separate n butane as distillate. So the bottom which is 2 butenes and furfural is sent to the column C5. So 2 butenes that means cis-2-butenes and trans-2-butenes isomers are withdrawn here as distillate and furfural is recovered as bottom steam from the C5 column.

And this furfural is recycled to the previous C4 column where we are performing the extractive distillation. So, look at the complexity of the separations of those 6 component mixtures. Now this sequence of distillation column will of course be able to perform the separation and it will be economic as well. But still try to note that there is one scope for improvement.

In the problem statement, we have seen that we require butane mixtures. So 1 butene and those two butenes isomers. Because these 2 butenes that we separated here at the end from C5 column is mixed with 1 butene and sent to another dehydrogenation unit for production of butadiene. So perhaps a better sequence will be where we do not require to cause the separation among these butenes that perhaps will be more cost effective and will improve process economics.

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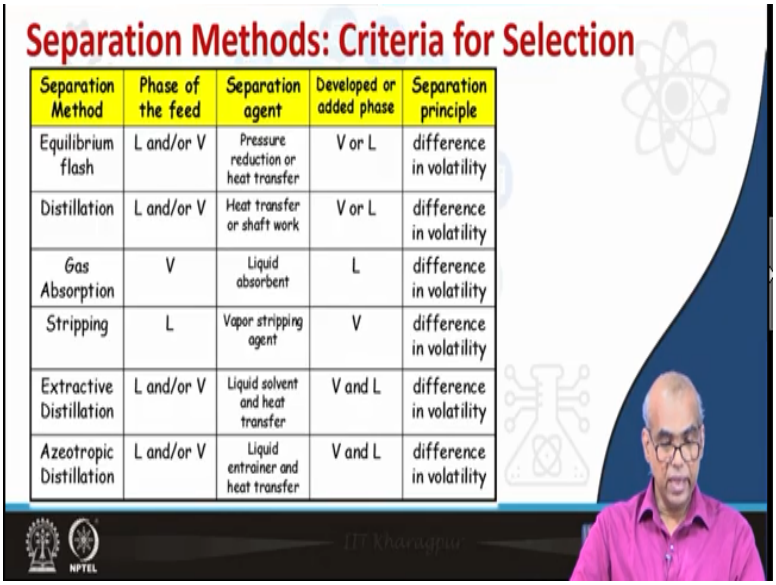

So here we give you a list of separation methods that are available for performing industrial separations. Also along with these phase of the field separation agent are given separation principles are also indicated. Developed or added phases are also indicated. For example for the separation method equilibrium flash, the phase of the feed is generally liquid and or vapour.

Separating agent is pressure reduction or heat transfer. Developed phase is vapour or liquid and the separation principle that is used is difference in volatility. Similarly, distillation gas absorption stripping, extractive distillation, azeotropic distillation all makes use of difference in volatility.

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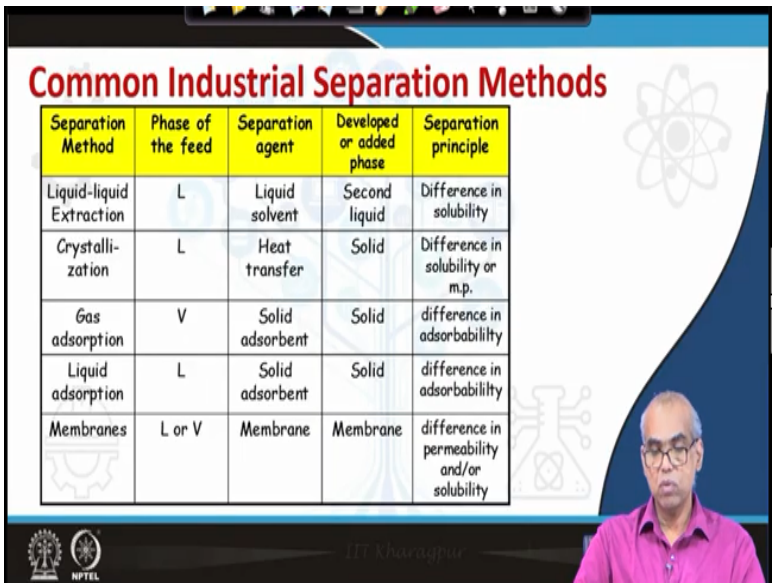

Liquid-liquid extraction makes use of difference in solubility. Crystallization makes use of differences solubility or multi point. Gas adsorption differences adsorbability. Liquid adsorption also makes use of difference in adsorbability. Membranes makes use of difference in permeability and or solubility.

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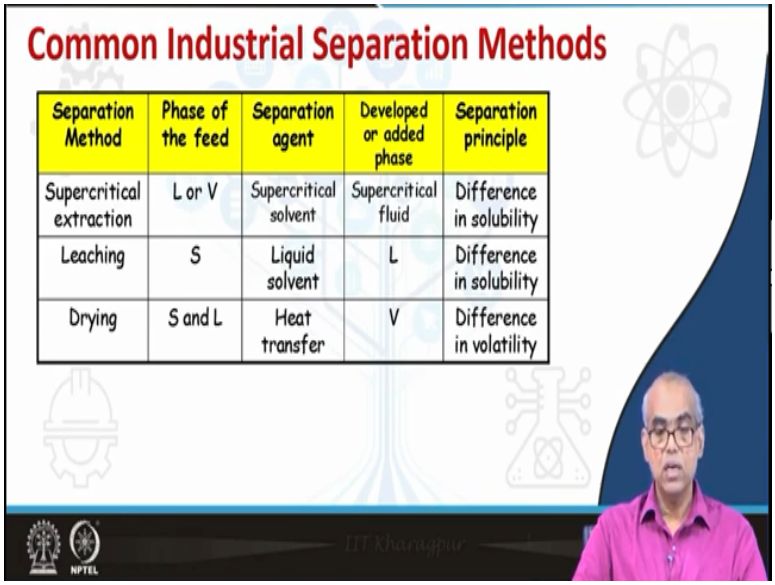

Supercritical extraction makes use of difference in solubility. Leaching also makes use of difference in solubility. And drying makes use of difference in volatility. So these are common industrial separation methods. In the next class, we will talk about what should be the selection criteria for such separation processes that are available. So with this we stop our discussion here.