Process Design Decisions and Project Economics
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Module - 4
Design of Separation Processes
Lecture - 18
Guidelines for Design of Separation Systems

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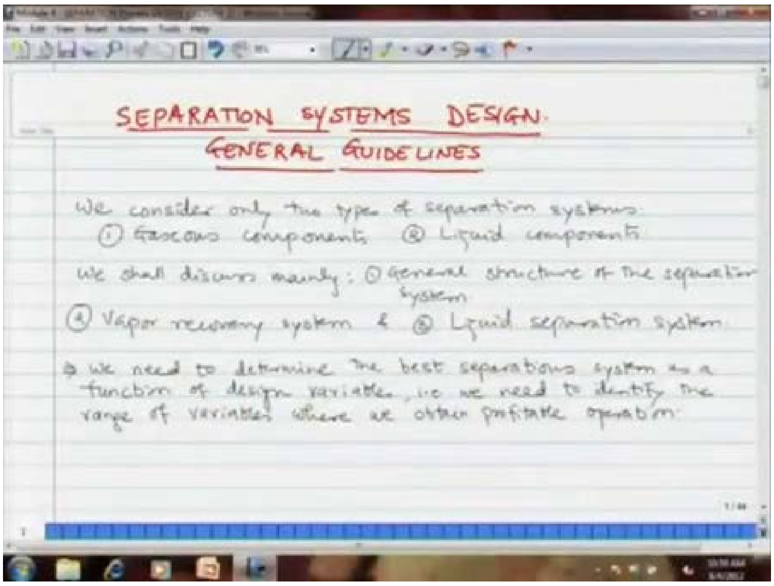

SEPARATION SYSTEMS DESIGN.
GENERAL GUIDELINES

We consider only two types of separation systems.
① Gaseous components ② Liquid components

We shall discuss mainly: ③ General structure of the separation system
④ Vapor recovery system & ⑤ Liquid separation system.

⑥ We need to determine the best separations system as a function of design variables, i.e we need to identify the range of variables where we obtain profitable operation.

Welcome, we are looking at the design of separation systems. In the previous lecture, we got an introduction at as what kind of separation processes exist for different kinds of mixtures that emerge from the reactor or from the process. In this lecture, we take ahead the theme to see the general guidelines of separation systems design, and this will be followed by certain aspects of distillation column design, which is the most widely used a separation process in all types of chemical and process industries.

So, here we consider only two types of separation systems; gaseous components and liquid components. We are not considering solid components, because separation of solids is relatively easier, and we shall discuss mainly the general structure of the separation system that is repo recovery system, and liquid separation system. We need to determine the best separation system as a function of the design variables that is we need to identify, the range of variables were we obtained the profitable operation.

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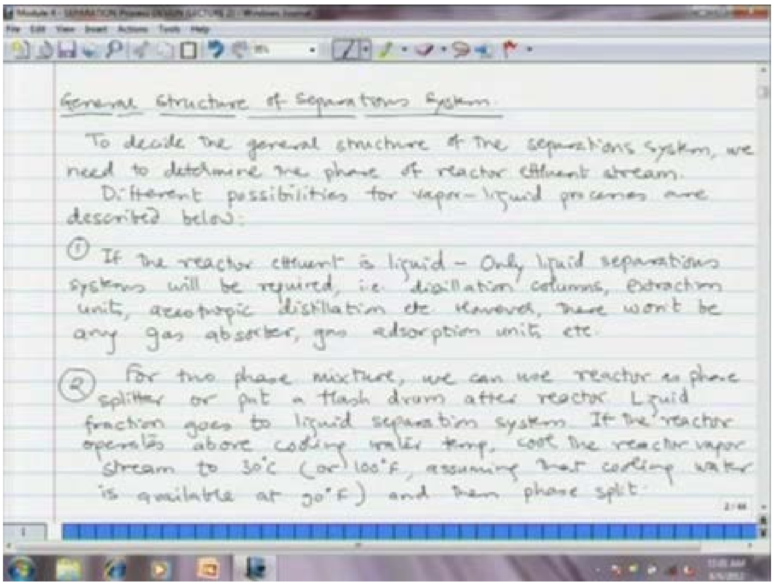

General Structure of Separations System

To decide the general structure of the separations system, we need to determine the phase of reactor effluent stream.

Different possibilities for vapor-liquid processes are described below:

① If the reactor effluent is liquid – Only liquid separations systems will be required, i.e. distillation columns, extraction unit, azeotropic distillation etc. However, there won't be any gas absorber, gas adsorption unit etc.

② For two phase mixture, we can use reactor as phase splitter or put a flash drum after reactor. Liquid fraction goes to liquid separation system. If the reactor operates above cooling water temp., cool the reactor vapor stream to 30°C (or 100°F, assuming that cooling water is available at 90°F) and then phase split.

The first aspect that we need to deal with is the general structure of the separation system, to decide the general structure of the separation system we need to determine the phase of the reactor effluent stream. The process could be completely vapor phase process, or it could be a gas liquid process, or a liquid-liquid hydrogenous process. So, depending on the reaction that is occurring, the temperature pressure the phase of the reactor effluent is decided.

There are different possibilities for vapor liquid processes as below, first is that if the reactor effluent is completely liquid then only liquid separation systems will be required, such as distillation column, extraction units, azeotropic distillation, etcetera. However, there would not be any gas absorber, gas absorption unit etcetera. For two phase mixture we can use the reactor as the as phase splitter, or we can put a flash drum after the reactor, where the phase split occurs.

The liquid factor emerging from the flash drum will go to the liquid separation system. If the reactor operates above cooling tower temperature, then we can use the cooling water to cool the reactor vapor stream from up to 30 degrees or 100 degrees Fahrenheit. Assuming that the cooling water is available at about 90 or about 90 degrees Fahrenheit about 20, 25 degree centigrade and then we have to phase split.

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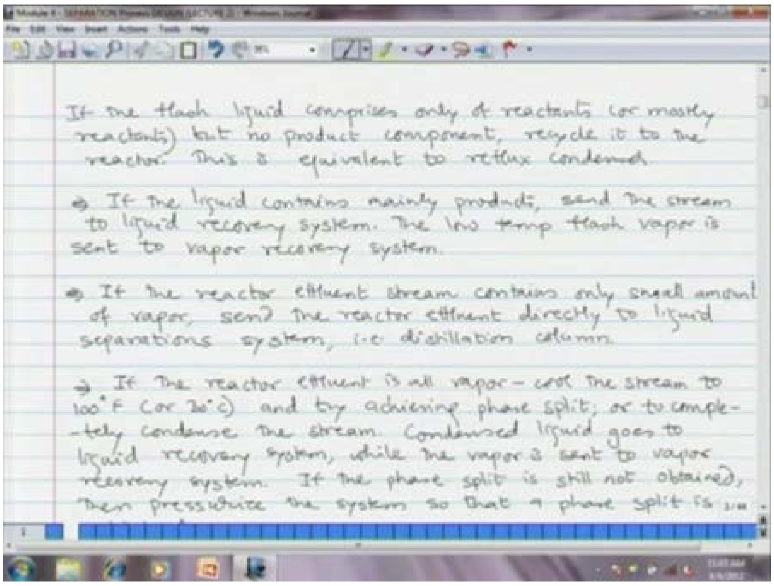

If the flash liquid comprises only of reactants (or mostly reactants) but no product component, recycle it to the reactor. This is equivalent to reflux condenser.

⇒ If the liquid contains mainly products, send the stream to liquid recovery system. The low temp flash vapor is sent to vapor recovery system.

⇒ If the reactor effluent stream contains only small amount of vapor, send the reactor effluent directly to liquid separations system, i.e distillation column.

⇒ If the reactor effluent is all vapor – cool the stream to 100°F (or 38°C) and try achieving phase split; or to completely condense the stream. Condensed liquid goes to liquid recovery system, while the vapor is sent to vapor recovery system. If the phase split is still not obtained, then pressurize the system so that a phase split is

If the flash liquid comprises only of the reactants or mostly reactants, but no product component. Means, our product is mainly in gaseous phase or vapor phase then that stream can be directly recycled to the reactor, this is equivalent to a reflex condenser. If the liquid contains mainly products.

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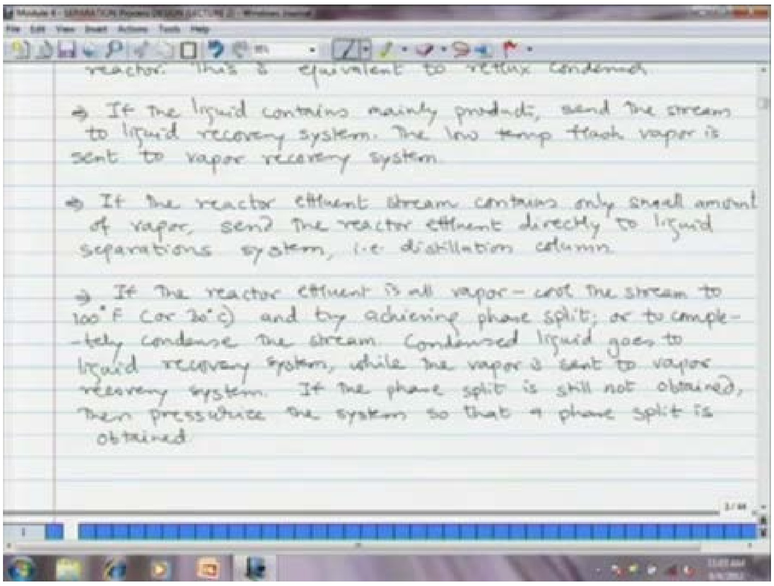

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Then we have to send the stream to liquid recovery system. If low temperature of flash vapor is then sent to vapor recovery stream, and that vapor mainly contains the un-reacted reactant which are finally, recycled to the reactor. If the reactor effluent stream contains only small amount of vapor then we have to send the reactor effluent directly to the liquid separation system that is distillation column. Now, distillation column contain various types of feeds, as you have seen in the course of mass transfer that decides the quality of the feed. If that q value which is used in distillation, if q is equal to 1 then the feed is completely saturated liquid or liquid at its bubble point. If q is less than 1, then it is vapor and so on and so forth.

If the reactor effluent is all vapor then we have to cool the stream to room temperature, and then try achieving the phase split or to completely condense the stream. Condense liquid goes to the liquid recovery system while, the vapor is sent to the vapor recovery system. If the phase split is still not obtained then we have to pressurize the system so that a phase split is obtained. Whenever you have the reactor effluent at high pressure and high temperature, first try to reduce the temperature and then reduce the pressure so that the phase split is easy.

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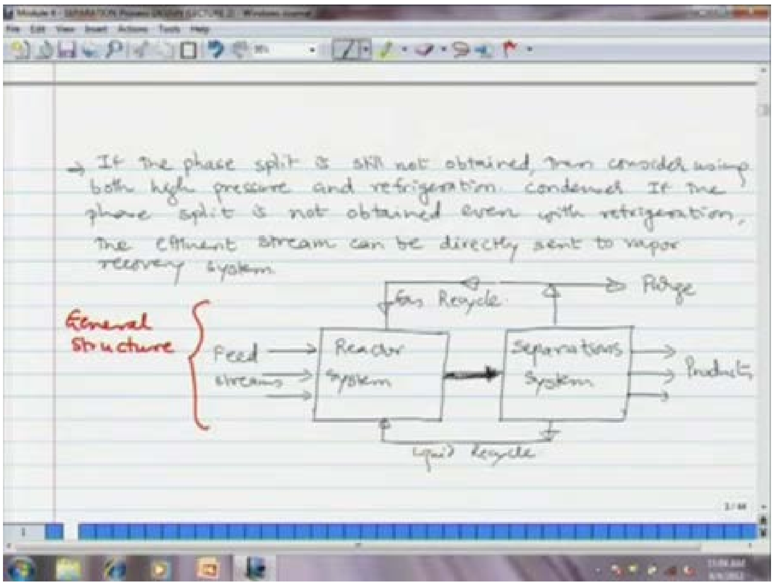

→ If the phase split is still not obtained, then consider using both high pressure and refrigeration condenser. If the phase split is not obtained even with refrigeration, the effluent stream can be directly sent to vapor recovery system

If the phase split is still not obtained then consider using both high pressure and refrigeration, condenser. If the phase split is not obtained even with refrigeration then the effluent stream can be directly sent to the vapor recovery system, to take off only the vapor component that is reactant. And all the gaseous products are then either recycled or they are recycled and purged. So, that the inert or whatever or the in gaseous components that can be deleterious for the process, it may be like catalyst poison etcetera. And the those are the concentration of these components is kept below limit.

So, as you see here on the screen after vapor recovery, you can recycle the gas with a purge stream. So, what we see now is a general structure of the separation system, we have a rector system taking in the feed streams then the reactor effluent comes out it is separated. Now, we have shown only the block diagram we are not we have not shown the details, as what kind of separation system exists, but we can open the second box this box later on. And then we have product streams a liquid cycle stream and gas recycle and purge stream.

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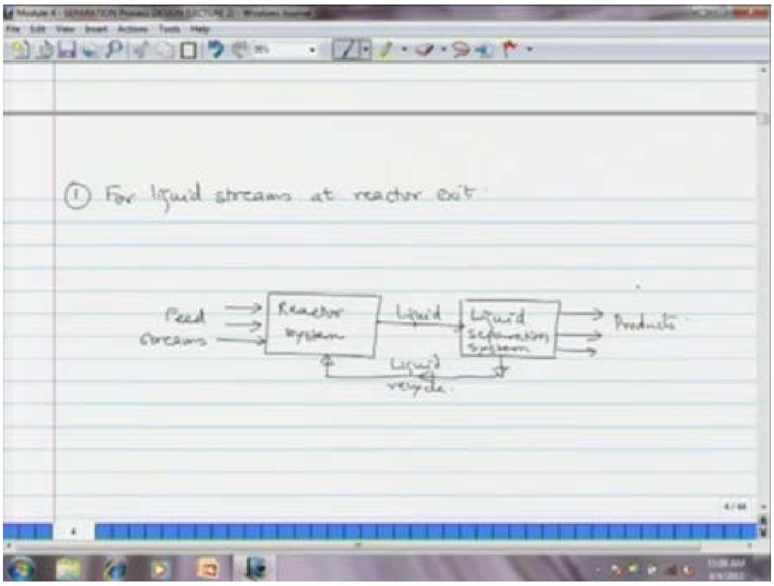

① For liquid streams at reactor exit

For the liquid streams at the reactor exist, we have a reactor system then liquid and then liquid separation system. Liquid separation system could be distillation, it could be extraction and so on and so forth.

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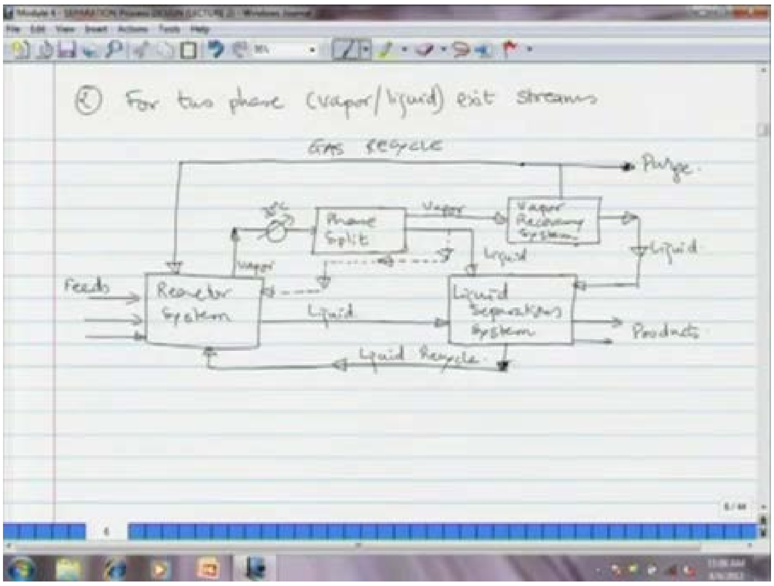

(c) For two phase (vapor/liquid) exit streams

Then if we have a two phase mixture vapor liquid mixture at the reactor exist. Then we have the reactor system, the liquid goes to the liquid separation system as per as vapor is concerned, you have to cool to about 30, 35 degrees, then you have to phase split, phase spilt will generate two streams liquid stream, which is sent to the liquid separation system as shown. And it will generate vapor, which is sent to the vapor recovery system. Vapor recovery system will again generate liquid because you have to recover that vapor that solute vapor in some solvent.

Then the solvent has to be recycled after recovery of solute that separation, or splitting or recovery of solute from solvent is again by distillation, from vapor recovery system you see a stream that is coming to the liquid separation system. The output of vapor recovery system is non condensable gas, which is either is recycled and also purged. And then liquid separation system will generate again a liquid recycle stream. So, this is how we can write a general structure of the vapor liquid separation system. You can also here in dotted line a stream coming from phase split liquid stream to the reactor.

Now, if the liquid contains all un-reacted reactants, then there is no reason for separating them know because in the in one of the previous lectures. In the second module of synthesis of fluid sheet, we have learnt a heuristic that never separated mix the two streams. If the destination of different components is same those components can be sent in the same stream and that is exactly, what this dotted line is...

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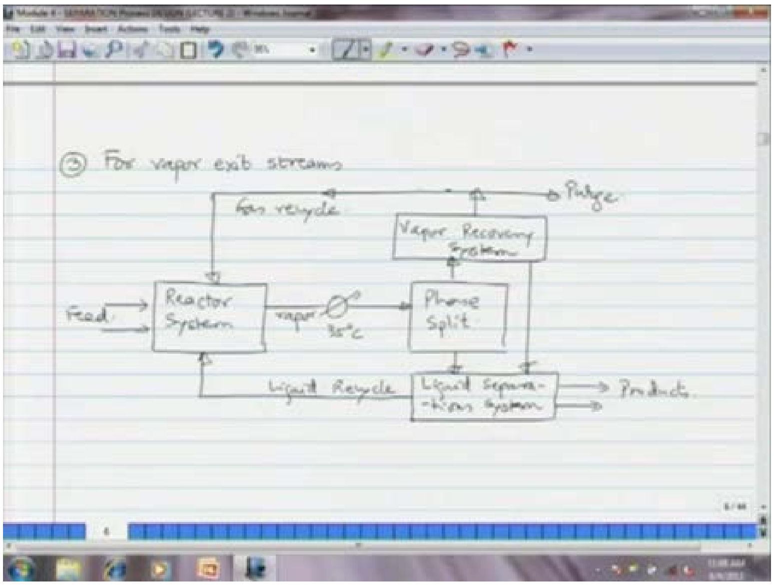

③ For vapor exit streams

The reactor exist stream is completely vapor then you have a system that is shown on the screen. Reactor system takes in feed, then the outlet is vapor stream it is cooled to 35 degrees then it is sent to phase split phase split generates two streams, liquid stream which is sent to liquid separation system. Then the un-reacted reactant the liquid phase is recycled, the products are taken out. Then the vapor stream is sent to the vapor recovery system, vapor recovery takes recovers all the solute vapor the non condensable gas is recycled and purged. Again you a stream coming from vapor recovery system into the liquid separation system, for the recovery of solute, from the solvent and recycle of solvent to the vapor recovery system.

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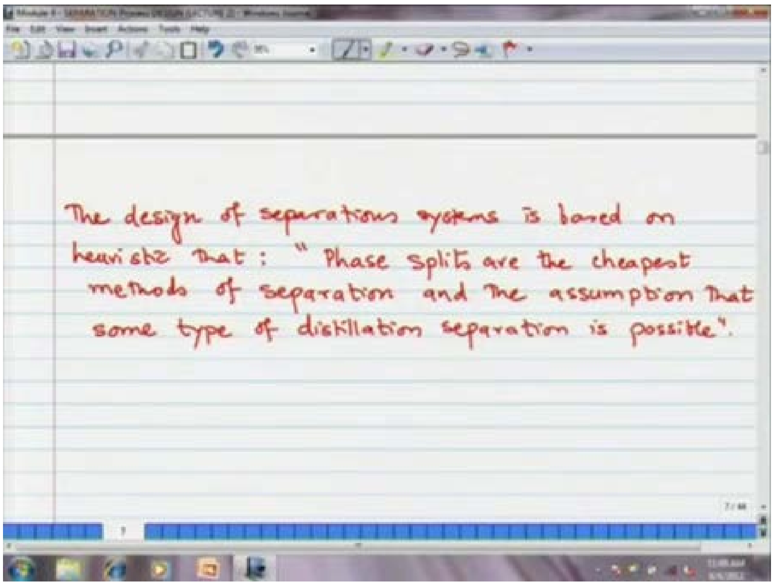
The design of separations systems is based on heuristic that : " Phase splits are the cheapest methods of separation and the assumption that some type of distillation separation is possible".

This design of separation systems is based on the heuristic that phase splits are the cheapest method of separation, and assumption that some type of distillation separation is possible. So, all of these systems that we saw are based on these heuristics or thumb rules.

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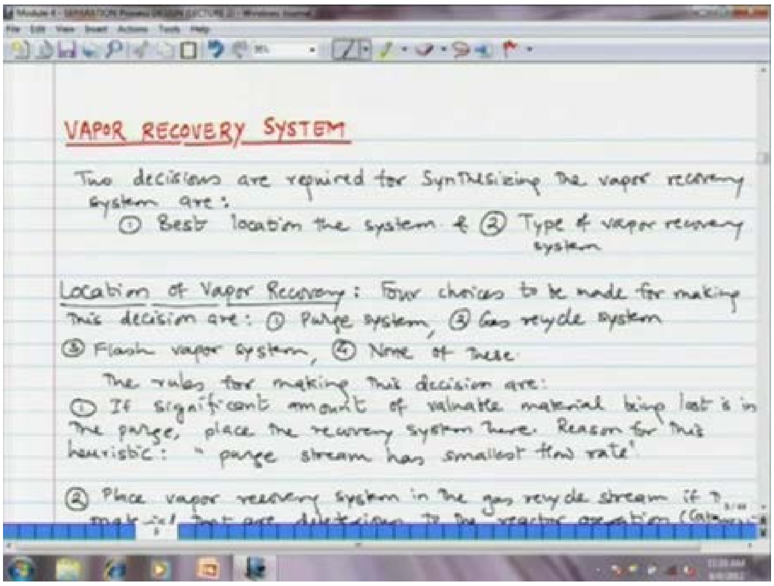
VAPOR RECOVERY SYSTEM

Two decisions are required for Synthesizing the vapor recovery system are:
① Best location the system & ② Type of vapor recovery system

Location of Vapor Recovery: Four choices to be made for making this decision are: ① Purge system, ② Gas recycle system, ③ Flash vapor system, ④ None of these.

The rules for making this decision are:
① If significant amount of valuable material being lost is in the purge, place the recovery system here. Reason for this heuristic: " purge stream has smallest flow rate"

② Place vapor recovery system in the gas recycle stream if materials that are deleterious to the reactor operation (Cat

Let us see what kind of vapor recovery system, we can design and what are the aspects that govern this design. The two decisions that are required for synthesizing the vapor recovery are, first the best location of the system and secondly the type of vapor recovery system. The location of vapor recovery, we have four choices the choices are first the purge stream.

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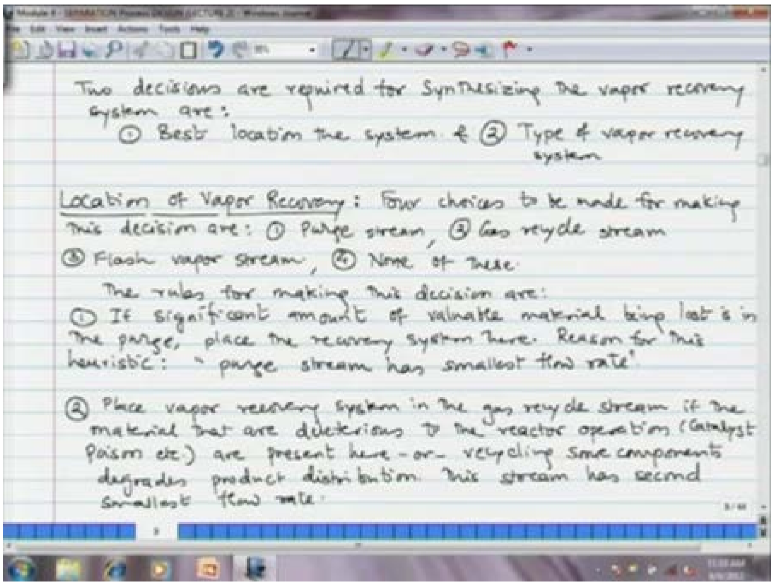

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The rules for making this decision are:
① If significant amount of valuable material being lost is in the purge, place the recovery system here. Reason for this heuristic: "purge stream has smallest flow rate"

② Place vapor recovery system in the gas recycle stream if the material that are deleterious to the reactor operation (Catalyst Poison etc.) are present here -or- recycling some components degrades product distribution. This stream has second smallest flow rate.

The gas recycle stream, then the flash vapor stream and somewhere else none of these. So, this is these four choices we have for location of the vapor recovery system. Now, depending on the type of process, and type of reactor effluent that we have the choice will be decided. Now, let us see what governs the choice or the rules for making this decision. If significant amount of valuable material is being lost is in the purge stream, then place the vapor recovery system in the purge stream.

Another reason for this heuristic or thumb rule is that the purge stream has smallest, total flow rate. Remember that the vapor recovery system has to recover vapor, the solute vapor from non condensable gas, but when you are making the design of an absorber, you have to take into account the total gas that is there, which solute plus the carrier gas. So, if the total through put is high then the size of the absorber will be high, again more amount of solvent will be required and then you will have to treat that solvent and distillation columns. So, the whole process becomes highly under intensive.

So, which stream out of emerging out of the flash drum has least flow rate that is obviously, the purge steam because the stream which emerges out of the flash drum, or phase splitter is divided in two parts one goes to gas recycle, another goes to purge and purge stream is always smaller. So, when you have place your vapor recovery system in the purge stream, then the sides will be smaller, because the purge stream has smallest vapor flow rate.

Then secondly place vapor recovery system in the gas recycle stream, if the material that are deleterious to the reactor operation are present, or recycling some components degrades the product distribution. As I said the stream which is emerging from the flash drum is split into parts, purge and recycle, a recycle goes to the reactor. However, the gas recycle stream is compressed before recycling to the reactor.

So, during that compression some vapor condenses. Now, if that stream contains some deleterious compounds like as I said earlier, some catalyst poisons or some components which are going to trigger side reactions, which will create loss of your valuable reactant. In that case you to prevent the prevent recycle of these components back to the reactor, you have to place the vapor recovery system in that stream. Now, that stream has the second smallest vapor flow rate.

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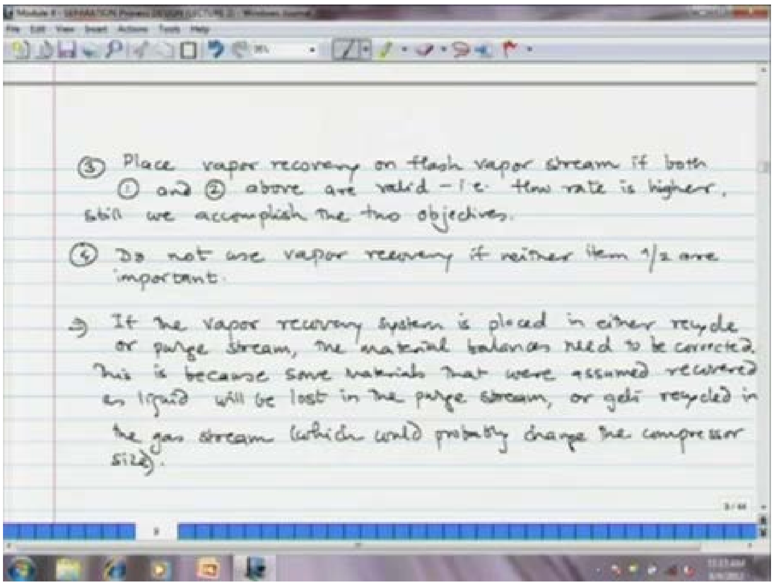

③ Place vapor recovery on flash vapor stream if both ① and ② above are valid – i.e. flow rate is higher, still we accomplish the two objectives.

④ Do not use vapor recovery if neither item 1/2 are important.

⇒ If the vapor recovery system is placed in either recycle or purge stream, the material balances need to be corrected. This is because some materials that were assumed recovered as liquid will be lost in the purge stream, or gets recycled in the gas stream (which would probably change the compressor size).

Then thirdly place the vapor recovery on the flash vapor stream, if both one and two are valid. The stream that emerges from the flash drum has the highest total flow rate it contains total non condensable gas plus total vapor. So, the total flow is highest in that stream. However, if both one and two apply if, let us say you have to recover valuable material at the same time you have to prevent the recycling of deleterious components, then you have to place the gas absorber immediately after the flash drum.

Now, in that case gas absorber operation will be expensive because for the reasons I mentioned earlier. However, you have no choice because if you do not remove the components earlier, you are going to suffer greater loss. And then finally, do not use the vapor recovery, if neither item one and two are important. If the vapor recovery system is placed in either recycle or purge stream, then the material balance need to be corrected this is cause of material that was assumed to be recovered as liquid will be lost in the purge stream or it might get recycled in the gas stream, which could probably change the compressor size. So, this the material balances need to be corrected, whenever we place the vapor recovery system.

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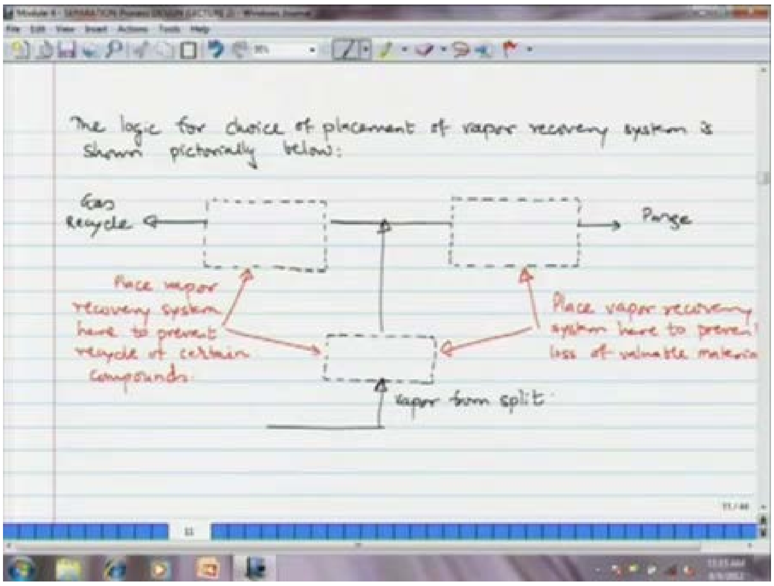

The logic for choice of placement of vapor recovery system is shown pictorially below:

Place vapor recovery system here to prevent recycle of certain compounds.

Place vapor recovery system here to preven loss of valuable materia

Here in this diagram I have shown schematic the of the logic for a choice of placement of vapor recovery system. We have three possible locations after the vapor from the phase split in the purge stream, and in the gas recycle stream. So, place the vapor recovery system here I mean at in either recycle stream, or after the flash drum to prevent recycle of certain compounds. Place the vapor recovery stream in the purge stream or from the vapor from split to prevent loss of valuable material.

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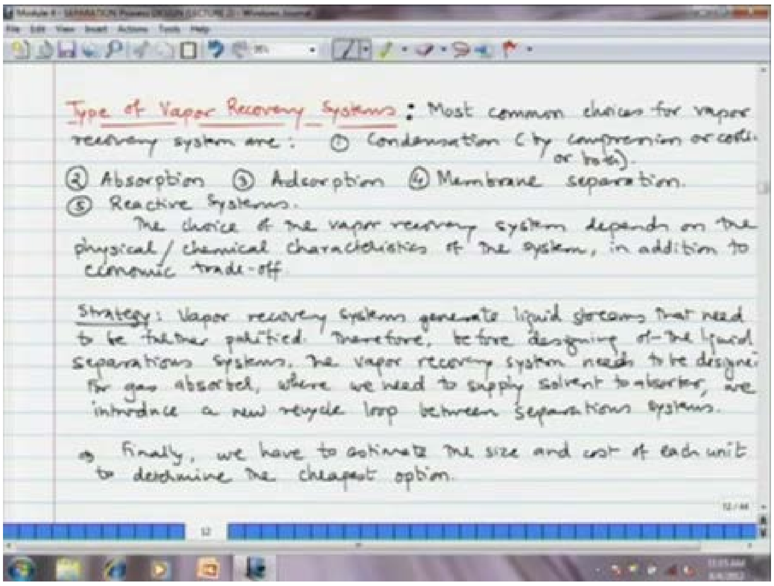

Type of Vapor Recovery Systems: Most common choices for vapor recovery system are: ① Condensation (by compression or cooli. or both). ② Absorption ③ Adsorption ④ Membrane separation. ⑤ Reactive Systems.

The choice of the vapor recovery system depends on the physical / chemical characteristics of the system, in addition to economic trade-off.

Strategy: Vapor recovery systems generate liquid streams that need to be further purified. Therefore, before designing of the liquid separations systems, the vapor recovery system needs to be designe. For gas absorbed, where we need to supply solvent to absorber, we introduce a new recycle loop between separations systems.

⇒ Finally, we have to estimate the size and cost of each unit to determine the cheapest option.

After we decide the location of the vapor recovery system, we have to decide at what kind of vapor recovery system we are going to use. The most common choices for vapor recovery system are first condensation by compression, or cooling, or both. Then second is absorption third is adsorption, fourth is membrane separation, and fifth is reactive systems. Now, the basic principles of all of these processes have been covered in the NPTEL course of course of mass transfer.

So, here we shall not go into the basic aspects or basic principles of these processes, but we shall see the practicable practical applicability of these processes under different circumstances. The choice of vapor recovery system depends on the physical and chemical characteristics of the system in addition to the economic trade off. As I have mentioned in previous lecture, we are going to have a separate module on economic decision making.

Then in that module, we shall take the design of gas absorber as the case study, but here we shall get some basic introduction on this topic. The strategy is that vapor recovery system generates liquid stream that needs to be further purified. Therefore, before designing of the liquid separation system, the vapor recovery system needs to be designed.

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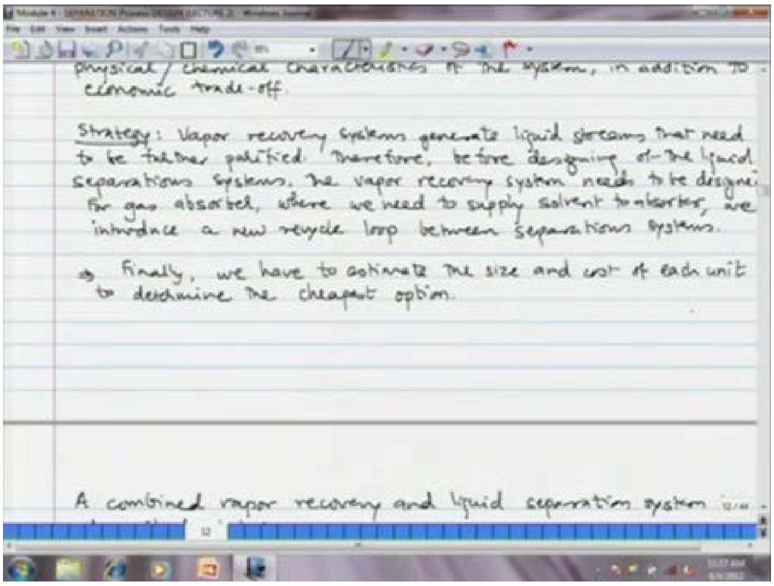

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A combined vapor recovery and liquid separation system is

That will calculate the additional load on liquid separation stream, in addition to the reactant reactor effluent that is coming out, the products. For gas absorber where we need to supply solvent to absorber, we introduce a new recycle loop between the separation systems. Finally, we have to estimate the size and cost of each unit to determine the cheapest operation.

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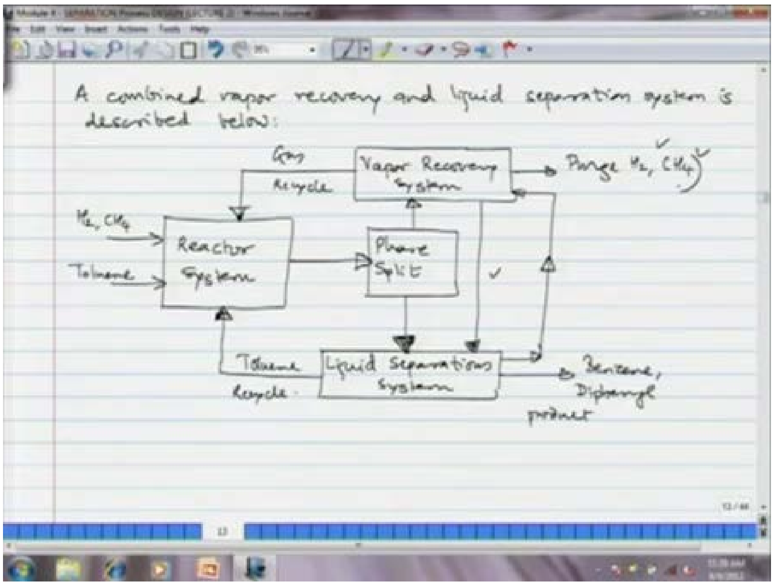

A combined vapor recovery and liquid separation system is described below:

We have taken in all our previous module or lectures, the hydro de-alkylation process as the case study and for hydro de-alkylation process, the combined vapor and liquid vapor recovery and liquid separation system is described below. Here the reactor, the reaction is in vapor phase I have already told you, the temperatures and pressures of the reaction toluene reacts with hydrogen to generate benzene, and methane that reactor operates at about 500 p s i and about 1200 degrees Fahrenheit or 895 temperature.

So, the reactor effluent is mixture of benzene that is main product, di phenyl that is side product un-reacted toluene, hydrogen and methane. And then it is at high pressure, high temperature first we recover all the heat from that stream, then we reduce the temperature thereafter in the phase drum, phase split drum or flash drum we reduce the pressure to have the split between the gas and liquid phase. Toluene, benzene, and di phenyl they condense hydrogen and methane they emerge as the gaseous stream, but they still have vapors of benzene, and toluene in it that is treated that particular exist stream gas exist.

Stream is treated in vapor recovery system, and then the benzene and toluene are recovered. You can see here a stream going from vapor recovery system to liquid separation system, this has the solvent plus benzene plus toluene. And then the gas non condensable gas that comes out vapor recovery system is split, you can see here the purge stream containing hydrogen and methane.

Now, methane is a inert compound here so the concentration of methane has to be kept below certain limit. So, we use a purge stream and rest of the stream this is the gas recycle, the liquid stream generates. The liquid separation system generates streams of benzene and di phenyl, which has taken or which are taken out as product and then the toluene stream is recycled. So, this is the general separation systems design for the hydro de-alkylation process.

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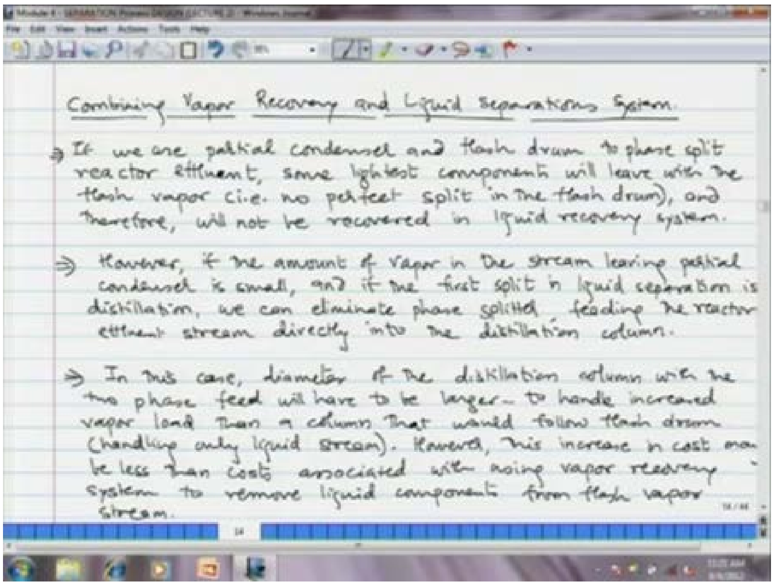

Combining Vapor Recovery and Liquid Separations System.

⇒ If we use partial condenser and flash drum to phase split reactor effluent, some lightest components will leave with the flash vapor (i.e. no perfect split in the flash drum), and therefore, will not be recovered in liquid recovery system.

⇒ However, if the amount of vapor in the stream leaving partial condenser is small, and if the first split in liquid separation is distillation, we can eliminate phase splitter, feeding the reactor effluent stream directly into the distillation column.

⇒ In this case, diameter of the distillation column with the two phase feed will have to be larger – to handle increased vapor load than a column that would follow flash drum (handling only liquid stream). However, this increase in cost may be less than costs associated with using vapor recovery system to remove liquid components from flash vapor stream.

Now, let us see how we can combine vapor recovery and liquid separation system, if we use partial condenser and flash drum to phase split the reactor effluent, some lightest components will leave with the flash vapor. That is no perfect split in the flash drum and therefore, will not be recovered in the liquid recovery system. However, if amount of vapor in this in the stream living partial condenser is small, then and if the first split in the liquid separation is distillation.

We can eliminate the phase splitter and feeding the reactor effluent stream directly into the distillation column. This is what is possible, because the feed to the distillation column need not be completely liquid, it may contain some vapor it might increase the diameter of the distillation column because the vapor which is coming in the in the feed also has to be accommodated, but that will that will take off the phase splitter. So, you reduce one unit from the process which will bring down the fixed cost.

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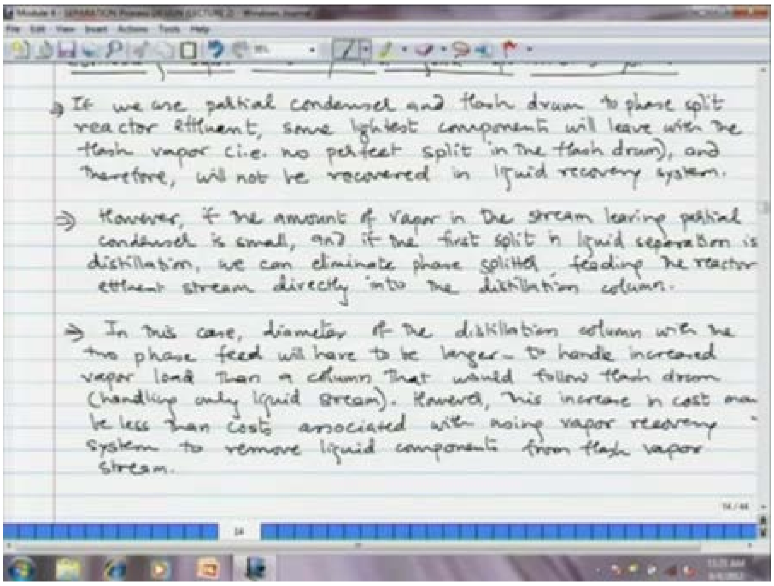

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In this case the diameter of the column with the two phase feed will be higher, as I just told to handle the increase vapor load, but at the same time the cost associated with vapor recovery system to remove liquid components from flash paper will be reduced.

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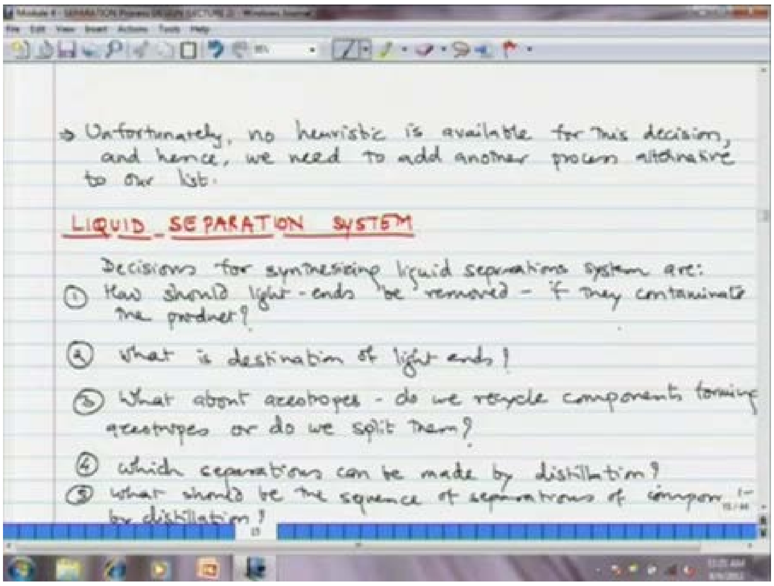

⇒ Unfortunately, no heuristic is available for this decision, and hence, we need to add another process alternative to our list.

LIQUID SEPARATION SYSTEM

Decisions for synthesizing liquid separations system are:

① How should light-ends be removed – if they contaminate the product?

② What is destination of light ends?

③ What about azeotropes – do we recycle components forming azeotropes or do we split them?

④ Which separations can be made by distillation?

⑤ What should be the sequence of separations of compon by distillation?

Now, there are no heuristics or guidelines available for making such decisions. So, these decisions vary from case to case and we need to look at the process carefully, before making such decisions. So, this is how you can combine the vapor and liquid separation system.

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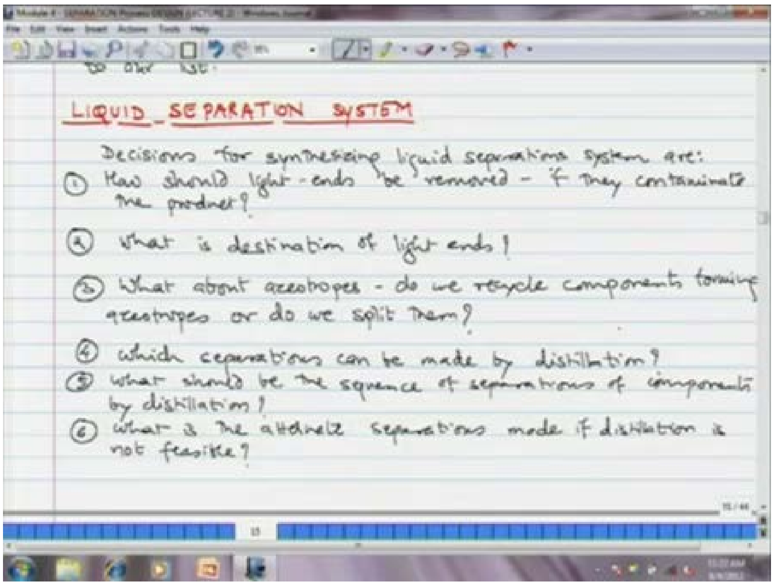

LIQUID SEPARATION SYSTEM

Decisions for synthesizing liquid separations system are:

① How should light-ends be removed - if they contaminate the product?

② What is destination of light ends?

③ What about azeotropes - do we recycle components forming azeotropes or do we split them?

④ which separations can be made by distillation?

⑤ what should be the sequence of separations of components by distillation?

⑥ what is the alternate separations mode if distillation is not feasible?

Let us see the design aspects of liquid separation system, what decisions we need to make the I am, I have listed these decisions on the screen. The first decision is that how should the light ends be removed if they contaminate the product. Now, the liquid stream coming in the distillation column will contain some light ends means. For example, if you consider a petroleum refinery then these light ends are usually c 1 to c 4 components. Usually methane, ethane, ethylene, propane, propylene, butane, butane, butadiene’s these are the light components. Now, these have to be removed before you start your mean distillation operation.

Then so the first question is how should the light ends be removed, secondly what should be the destination of the light ends, what should we do with these light ends. Then thirdly what about the azeotropes because it is very lightly that there will be either binary or ternary or even quaternary azeotropes. So, to separate the azeotropic components mean to have a another supplementary separation system to distillation. Then how do we recycle components forming azeotrope or should we split them. Now, in some cases if both like if the complete azeotrope is going back to the reactor, then there is no need to split it.

However, in some cases for example, like ethanol water system to obtain 99.999 percent ethanol, pure ethanol so as to blend it with petroleum you have to separate water using evaporation, which is a like a process supplement to distillation. Ethanol and water form azeotropic mixture at about 82 percent of ethanol. So, purification of ethanol beyond that percentage is not possible so you have to go for alternate removal of water remaining water. So, splitting of the azeotrope is a major decision and it is highly cost intention, which separation first fourth decision, which separations can be made by distillation you would like to have as many separations per distillation as possible, for two reasons although distillation is a energy intensive process.

Firstly, the distillations columns can be integrated with the process and secondly they are easy to design and operate, even if they are energy intensive you can combine the re-boiler with some other process stream which has higher heat content, or surplus heat and drive that re-boiler. So, that way the distillation columns are easy to heat integrate with the process that is one thing. Secondly the design is easier because once you have it is a operation which has more than hundred years of history. So, you would it will be very easy to design and operate and optimize, the distillation column as per your requirement.

Then what should be the sequence of separation of components by distillation, if you have a multi component mixture then how you should separate them? Then what is the alternative separation to distillation, if distillation is not feasible. So, these are some of the important questions that you will face, while designing the liquid separation system and you have to make decisions on it.

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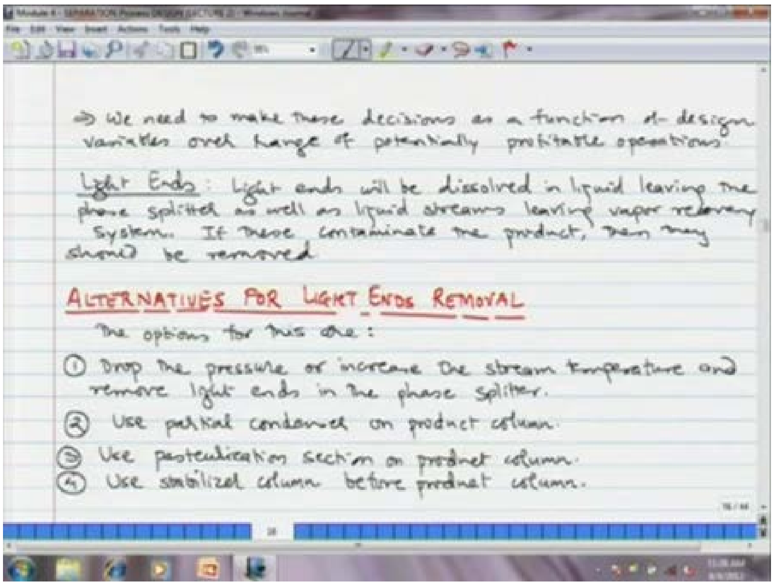

⇒ We need to make these decisions as a function of design variables over range of potentially profitable operations

Light Ends: Light ends will be dissolved in liquid leaving the phase splitter as well as liquid streams leaving vapor recovery system. If these contaminate the product, then they should be removed

ALTERNATIVES FOR LIGHT ENDS REMOVAL

The options for this one:

① Drop the pressure or increase the stream temperature and remove light ends in the phase splitter.

② Use partial condenser on product column.

③ Use pasteurization section on product column.

④ Use stabilized column before product column.

We need to make these decisions as a function of the design variables over the range of potentially profitable operation. Now, let us see the first thing light ends, light ends will be dissolved in the liquid leaving the phase splitter as well as the liquid streams leaving the vapor recovery system, if these contaminate the product then they should be removed. Now, what are the alternatives to distillation for removal of light ends, first option is obvious drop the pressure in the flash drum itself or increase the stream temperature and remove the light ends in the phase splitter.

The solubility of these light ends are essentially the gases as I told you their solubility decreases with temperature. So, if you increase the temperature or if you reduce the pressure then also this desorption occurs and then you can remove the light ends, or you can use a partial condenser in the product column of the dissipation column. So, that only the heavier components like c 5, c 6 onwards they condense, but not these light ends. Then use a pasteurization section on the product column with a side stream, or use a stabilizer column before the product column. Stabilizer column is simply boiling of the mixture liquid mixture so as to dissolve all the gases.

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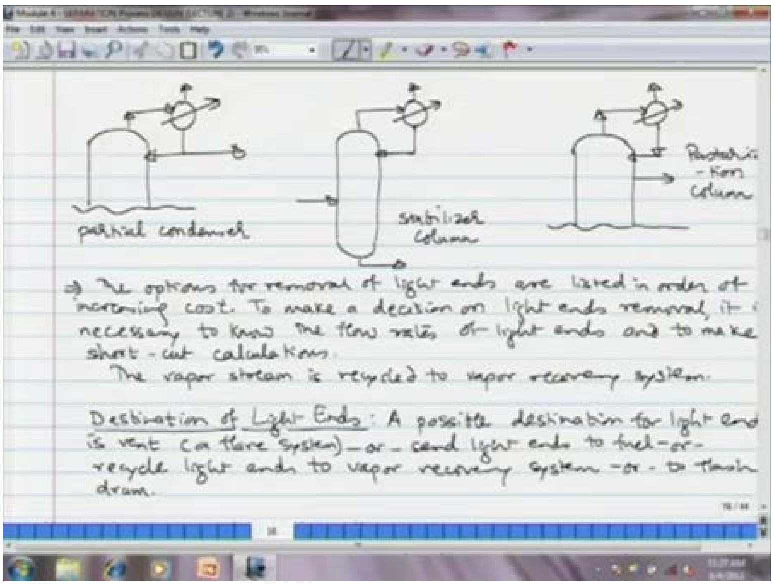

⇒ The options for removal of light ends are listed in order of increasing cost. To make a decision on light ends removal, it
necessary to know the flow rates of light ends and to make short-cut calculations.

The vapor stream is recycled to vapor recovery system.

Destination of Light Ends: A possible destination for light end
is vent (or flare system) - or - send light ends to fuel - or - recycle light ends to vapor recovery system - or - to flash drum.

So, what I have shown here are the schematics of these three types of removal of light ends, a partial condenser then a stabilizer column and a pasteurization column with a side stream. The options for removal of light ends are listed in order of increasing cost. So, partial condenser is the cheapest then comes the stabilizer column, then comes the pasteurization column. To make a decision on the light ends removal it is necessary to know the flow rates of the light ends and to make the short cut calculations. The vapor stream is recycled to the vapor recovery system so that is what it is.

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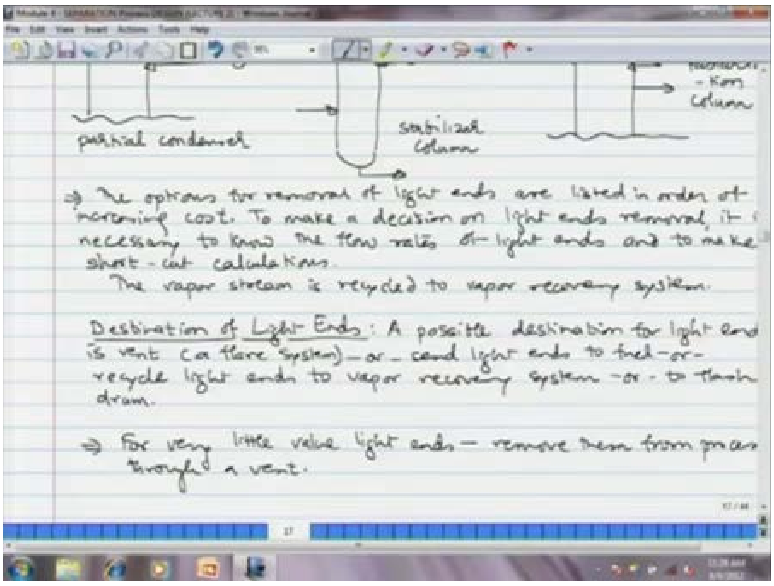

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Destination of Light Ends: A possible destination for light ends is vent (or flare system) – or – send light ends to fuel – or – recycle light ends to vapor recovery system – or – to flash drum.

⇒ For very little value light ends – remove them from process through a vent.

So, in order to have the decision on the removal of these light ends, you have to make certain calculations. First of all you have to know what is the flow rate of the light ends, and then you have to do some short cut calculations. First we decide that then the next question that we face is the destination, destination of light ends a possible destination of light ends is just a flare system, burn them before emitting like convert them to carbon oxide and water and then this is the gases flared, but because of these green house emissions.

Green house gas emissions the flare systems are now, being questioned like in many countries in especially, in gulf countries the flare of these gases is taxed because it is adding C O 2, a green house gas to the atmosphere, the alternate use is that use them as fuel in the process, send the light ends to the fuel or recycle light ends to vapor recovery system or to the flash drum. So, these are some of the options like for example, methane if the light ends are saturated molecules like methane, ethane, propane. Like propane and mixture of propane and butane used as the L P G. So, that is one of the use of these light ends.

Now, if the as the un-saturation of the molecule increases the reactivity increases. So, molecules like ethylene, propylene are being are increasingly being used as the major feed stock for petrochemicals. So, these are recovered and these fetch more market prices. Butadiene, butane is also used as a reactant and butadiene is a monomer for butadiene a rubber styrene butadiene rubber. So, these are some of the possible destinations or uses of the light ends. For very little value of light ends remove them from the process through a vent and then use them basically, as the fuel in the process.

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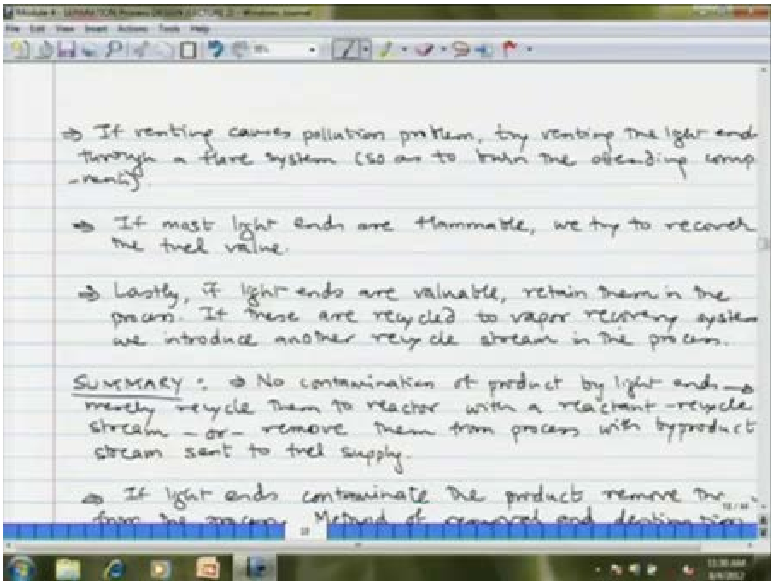

⇒ If venting causes pollution problem, try venting the light end through a flare system (so as to burn the offending components).

⇒ If most light ends are flammable, we try to recover the fuel value.

⇒ Lastly, if light ends are valuable, retain them in the process. If these are recycled to vapor recovery system we introduce another recycle stream in the process.

SUMMARY : ⇒ No contamination of product by light ends → merely recycle them to reactor with a reactant-recycle stream – or – remove them from process with byproduct stream sent to fuel supply.

⇒ If light ends contaminate the product remove them from the process. Method of removal and destination

If venting causes pollution problem then try venting the light ends through a flare system, so as to burn the offending components.

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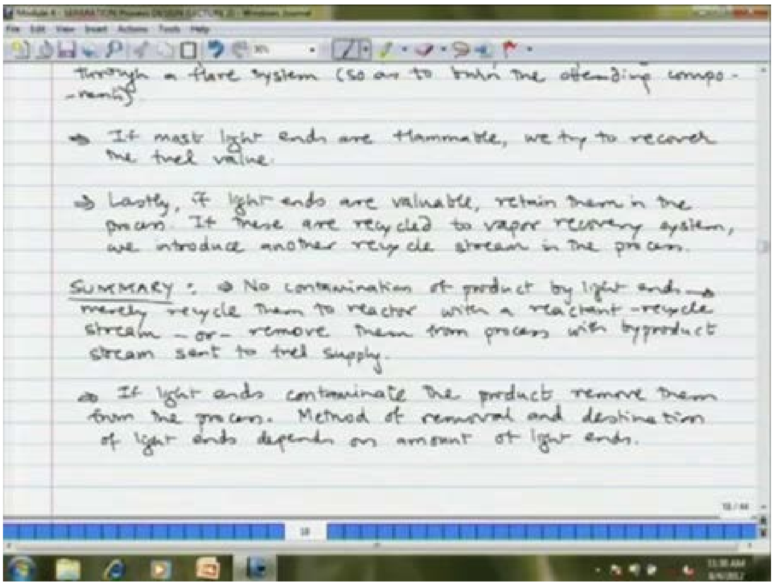
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⇒ If light ends contaminate the product remove them from the process. Method of removal and destination of light ends depends on amount of light ends.

If the light ends are flammable then try to recover the fuel value by using them as a fuel in your furnace, or other heaters. And lastly if the light ends are valuable them retain them in the process like some of the light valuable light ends I just mentioned, the unsaturated molecules. If these are recycled to the vapor recycle system, we introduce another recycle stream in the process, but remember that this recovery of the light valuable light ends come at a cost. So, summarizing no contamination of product by light ends, merely recycle them to the reactor with a reactant recycle stream or remove them from the process with the byproduct stream, and send to fuel supply.

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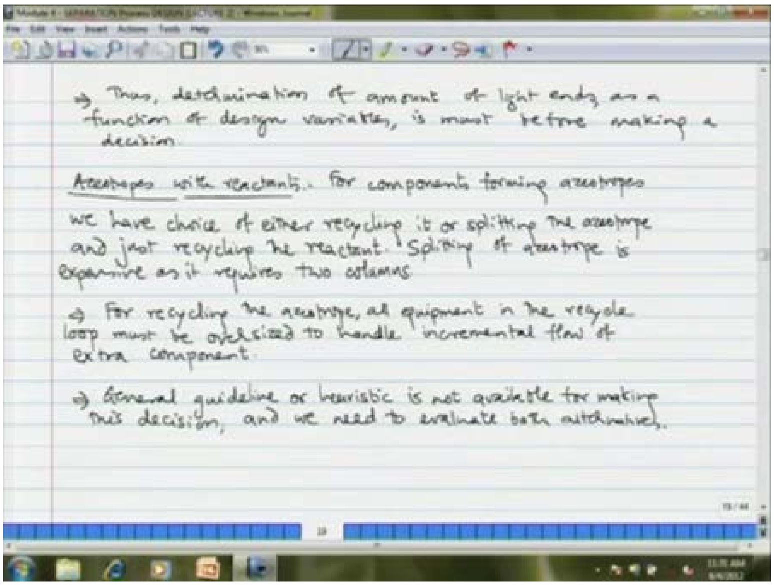
⇒ Thus, determination of amount of light ends as a function of design variables, is must before making a decision.

Azeotropes with reactants: For components forming azeotropes we have choice of either recycling it or splitting the azeotrope and just recycling the reactant. Splitting of azeotrope is expensive as it requires two columns.

⇒ For recycling the azeotrope, all equipment in the recycle loop must be oversized to handle incremental flow of extra component.

⇒ General guideline or heuristic is not available for making this decision, and we need to evaluate both alternatives.

A method of removal and of destination of light ends depends on the amount of light ends that I already mentioned. So, the determination of amount of light ends as a function of design variables is must before making a final decision, this point we note. Azeotropes with reactants if a component forms azeotrope, we have choice of either recycling it or splitting it, if the component forms azeotrope with reactant then we have trouble because if we have to recycle the un-reacted reactant to the reactor, the azeotrope component also gets recycled.

Then we have to split the azeotrope, if the azeotrope forming component is deleterious is going to trigger side reactions, or is going to have some adverse impact on the process, we have to split the azeotrope to recycle just the reactant. Azeotrope distillation as I already mentioned is an expensive operation because to break the azeotrope, you have to many times you have to use a third component called as a entrainer. Then you require two columns, first column to make the azeotropic distillation and second component is to recover that entrainer and recycle.

For recycling the azeotrope all equipment in the recycle loop must be over sized to handle, the incremental flow of extra component that cost you are going to incur, if you want to decide or you want to recycle the azeotrope. The general guidelines and heuristics are not available for making this decision, and the final decision over this depends on the exact process and varies from case to case.

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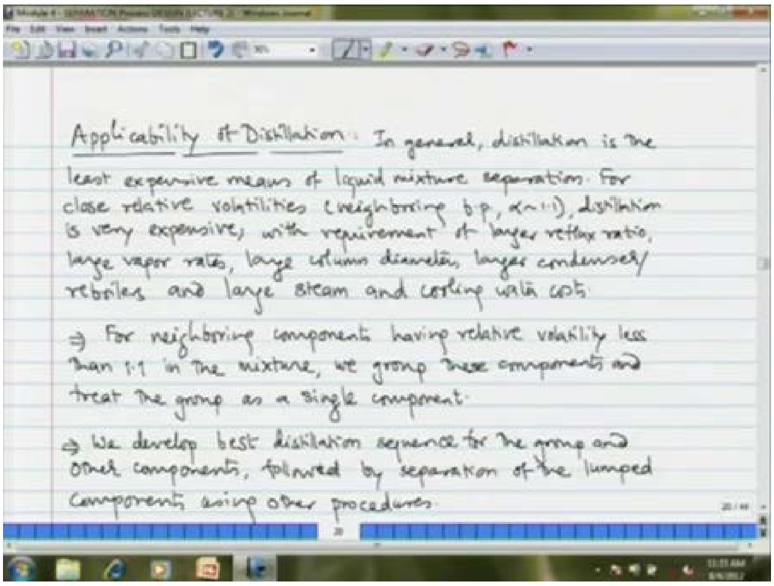

Applicability of Distillation: In general, distillation is the least expensive means of liquid mixture separation. For close relative volatilities (neighboring b.p, $\alpha \sim 1.1$), distillation is very expensive, with requirement of larger reflux ratio, large vapor rates, large column diameter, larger condenser/reboiler and large steam and cooling water costs.

⇒ For neighboring components having relative volatility less than 1.1 in the mixture, we group these components and treat the group as a single component.

⇒ We develop best distillation sequence for the group and other components, followed by separation of the lumped components using other procedures.

The next question is that how long distillation will be applicable, like as I said distillation would be the most preferred method of separation of liquid, but will distillation be possible each and every time. In general distillation is energy intensive, but if you heat integrate the process then the cost grows. So, it is less expensive means of liquid mixture separation compared to other processes. For example, in liquid-liquid extraction you need to recover the solvent. So, you need distillation column there if you want to go for a membrane process then membrane itself is a highly expensive component.

So, if you compare although a distillation is energy intensive because you have to use the re-boiler and condenser, if you compare distillation with these other things distillation still stands a good chance. So, that is what distillation is a comparatively least expensive means of liquid mixtures aggression. For close relative volatilities neighboring boiling point or alpha equal to 1.1, distillation can become expensive because of the high large number of plates that are required, or you have to use high reflex ratio large vapor rates, large column diameter, and which comes as a consequence, large condenser and re-boiler with high heat duties. And basically, heat duties means large stream and cooling water cost.

So, that is what means distillation has that limitation, if as the alpha becomes closer to 1 like 1.1, 1.2 then you have this problem that very large number of plates would be required, for separation of the mixtures. Typical rule here is that for neighboring components having relative volatility less than 1.1 in the mixture, group the components and treat the group as a single component. So, this is a sortuber rule or you can say heuristic.

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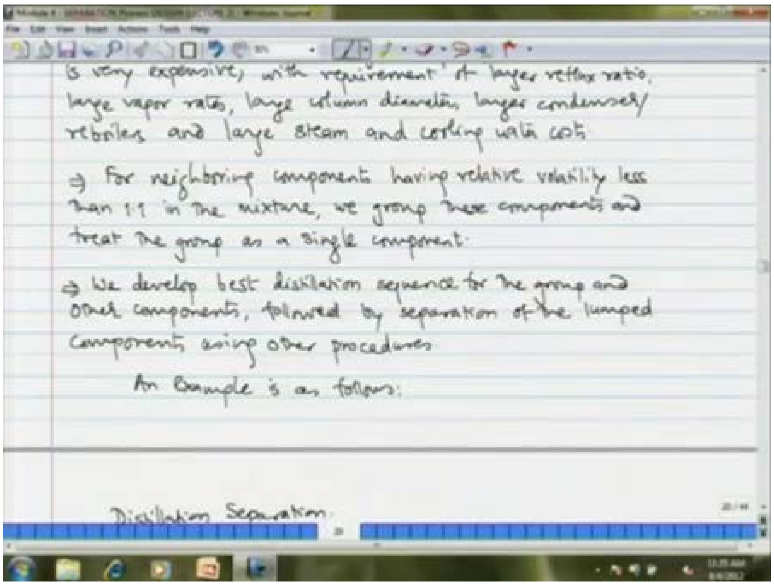

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⇒ We develop best distillation sequence for the group and other components, followed by separation of the lumped components using other procedures.

An Example is as follows:

Distillation Separation

It may not be applicable every time, but this is what usually the designers or engineers do if the, if the relative volatility is less than 1.1. Then you treat that mixture of components as a single entity, try to remove all other things. And once everything is removed then you try to split those that mixture. We have to develop the best distillation sequence for the group and other components followed by separation of the lumped components, using other procedures.

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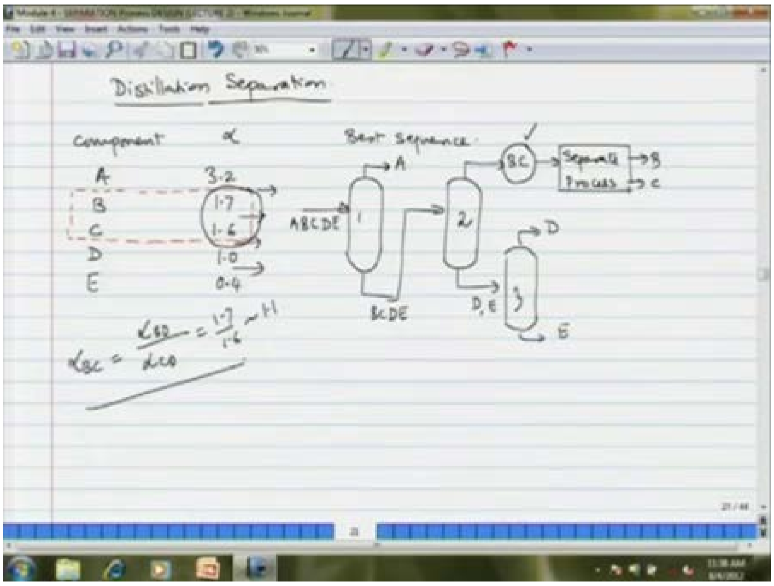

Distillation Separation

Component	α
A	3.2
B	1.7
C	1.6
D	1.0
E	0.4

$$\alpha_{BC} = \frac{\alpha_{BD}}{\alpha_{CD}} = \frac{1.7}{1.6} \sim 1.1$$

Best Sequence

So, what I have shown here is an example distillation separation we consider 5 component mixture A, B, C, D, E and alpha is given with respect to D. Now, D is a heavy key element, which is the important element among all the heavy components. So, alpha value is taken 1 with respect to D. So, E being a heavier component more boiling point, it has a alpha lesser than 1. Now, with respect to D, C has alpha of 1.6, B has alpha of 1.7, A has alpha of 3.2. Now, if you calculate the intermediate volatilities, you can see that you have the you have a very low volatility, a relative volatility between B and C because alpha B, C is essentially alpha B, D divided by alpha C, D.

Then 1.7 divided by 1.6 is very close to 1.1. ‘So, here you have the problem. Now, what I have shown as side is the best sequence for the separation of such kind of mixture, in the first column all the five components going A, B, C, D, E. A is taken off and then B, C, D, E flow to the next column, in this column the top product is a mixture of B and C. As I said that when components have very close boiling points or very alpha very close to 1, treat those two components or three whatever as a single entity.

So, we take off B, C together at the top of column 2. And then the remaining two components D and D flow to the third column, and then they are split as individual components D and D. Now, as far as this B, C mixture is concerned it is taken off as the top product of the second column, and then it is send to a separate process which could be liquid extraction, which could be let us say membrane separation etcetera. And then the two components B and C are separated, or here there could be another azeotropic distillation using an entrainer.

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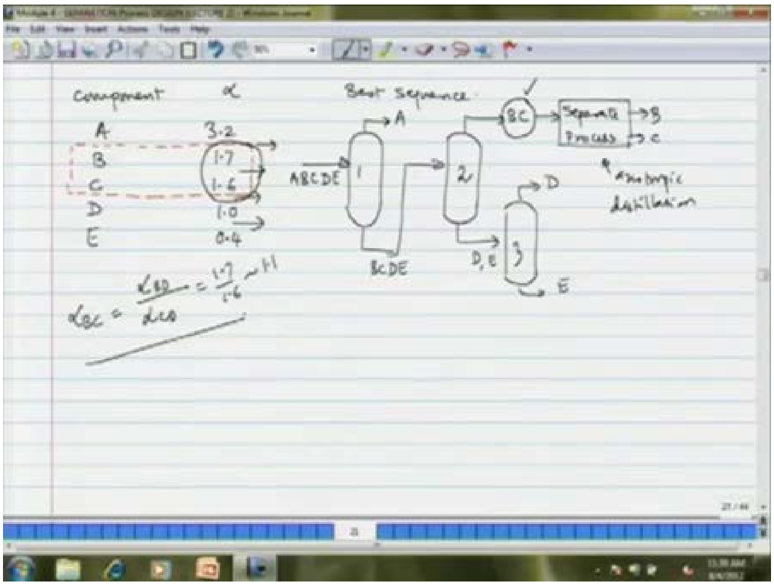

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$$\alpha_{BC} = \frac{\alpha_{BD}}{\alpha_{CD}} = \frac{1.7}{1.6} \sim 1.1$$

So, this is how you would like to treat the azeotropic mixture, if at all you have to split the azeotropes splitting of azeotrope is at most necessary. Then you do it in absence of all other components. So, that we are handling the least total load liquid load and there is no other component, which can complicate your process.

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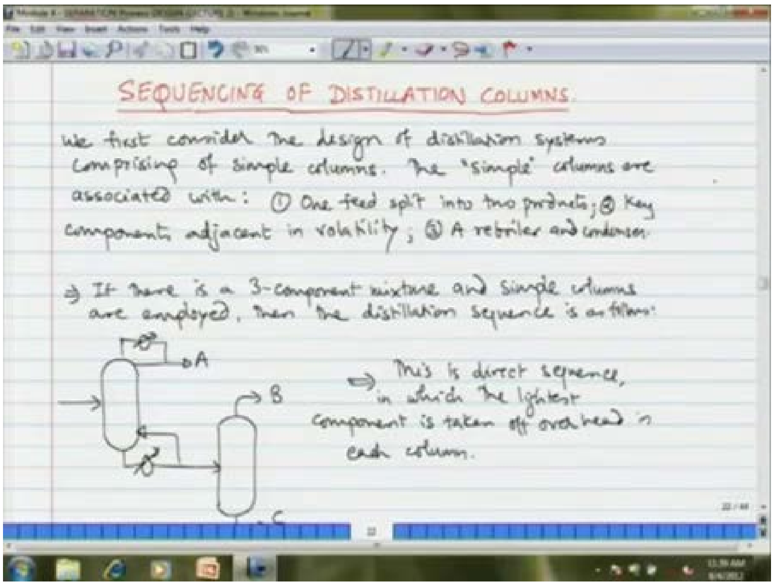

SEQUENCING OF DISTILLATION COLUMNS

We first consider the design of distillation systems comprising of simple columns. The 'simple' columns are associated with: ① One feed split into two products; ② Key components adjacent in volatility; ③ A reboiler and condenser.

⇒ If there is a 3-component mixture and simple columns are employed, then the distillation sequence is as follows:

⇒ This is direct sequence, in which the lightest component is taken off overhead in each column.

Let us see the sequencing of distillation column because this is another major question that you are going to face while designing the separation system. We first consider the design of distillation systems comprising of simple columns, these simple columns are associated with one feed split into two products. Then key components adjacent in volatility and a reboiler and a condenser, which is given independent utilities means there is no heat integration as such. Now, if there is a three component mixture and simple columns are employed then we have two possible sequences for separation.

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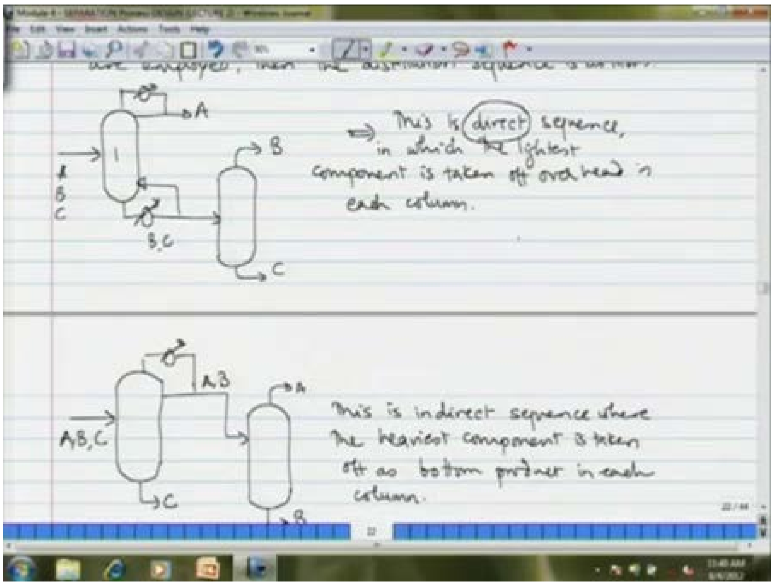

⇒ This is direct sequence, in which the lightest component is taken off overhead in each column.

This is indirect sequence where the heaviest component is taken off as bottom product in each column.

The first sequence is shown here the mixture of A, B, C components flows to the first column, A is taken off as a distillate and B and C go to next column, and then they are split into individual mixtures B and C. This is known as a direct sequence and the characteristic of direct sequence is that the lightest component is taken off over at an each column. So, the mixture is separated sequence of higher volatility that is how like A then B like that.

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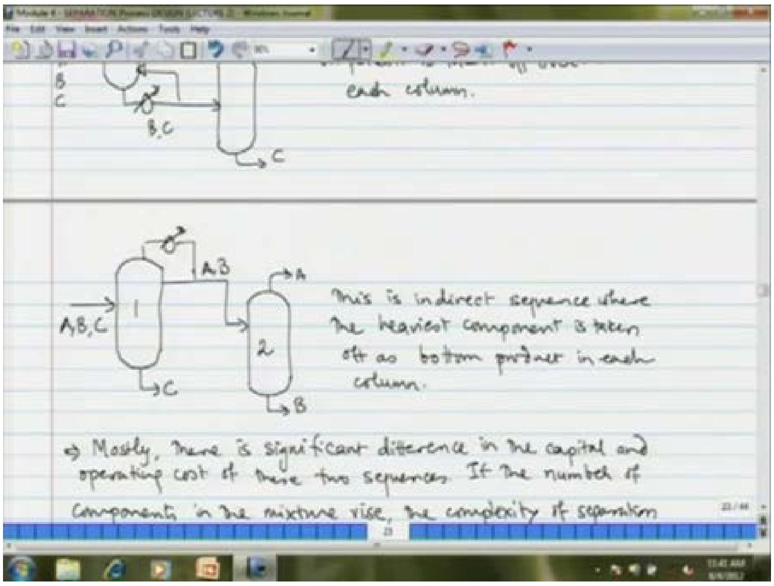

each column.

This is indirect sequence where the heaviest component is taken off as bottom product in each column.

⇒ Mostly, there is significant difference in the capital and operating cost of these two sequences. If the number of components in the mixture rise, the complexity of separation

Another option is that in the first column the mixture A, B, C will flow, but C will be taken off as the bottom product. And then A, B together are taken off as the top product which move to the second column, and then they are split individually. So, this is in not in the direct sequence, direct like proportion of the relative volatility. If A and B are taken off together. So, heaviest component is taken off or removed in the first place. So, this sequence is known as the indirect sequence, where the heaviest component is taken off as the bottom product in each column, this is another option that we have.

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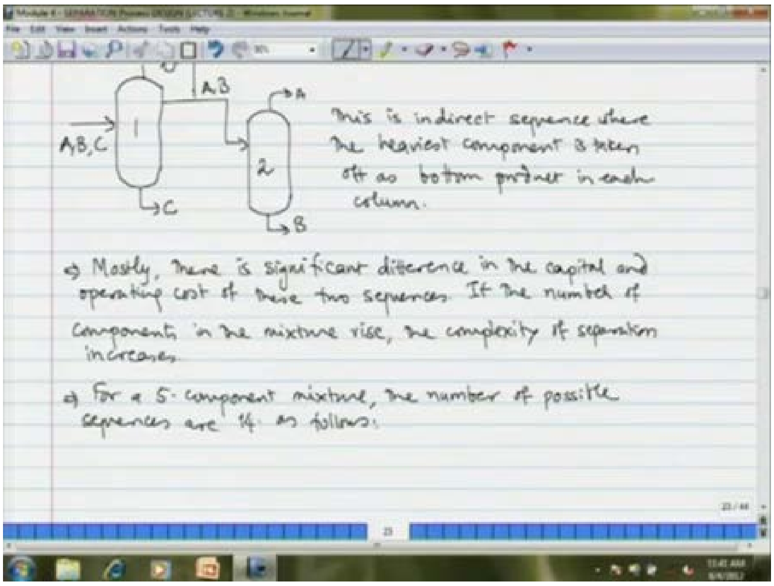

This is indirect sequence where the heaviest component is taken off as bottom product in each column.

⇒ Mostly, there is significant difference in the capital and operating cost of these two sequences. If the number of components in the mixture rise, the complexity of separation increases.

⇒ For a 5-component mixture, the number of possible sequences are 14 as follows:

Although, these sequences look similar they are not same, but they are similar there will be a significant difference in the capital and operating cost of the, of this two sequences. As the number of components in the mixture rise, then the complexity of separation increases. So, for a ternary mixture we had two possible sequences as the number of components increase, then we have more and more possible sequences.

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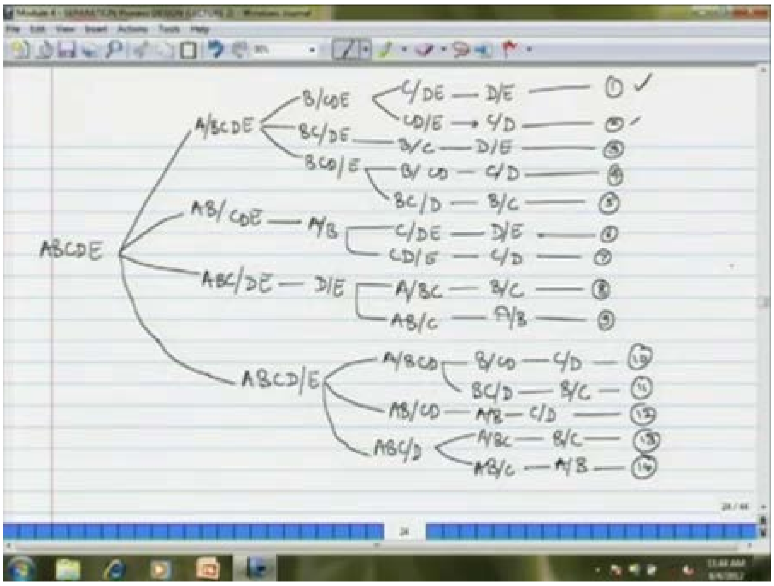

Now, what I have given here is example for a five component mixture like instead of A, B, C. Let us say we have five component mixture A, B, C, D, E then there are 14 possible sequences and this 14 sequences are now on the screen. First A, B, C, D mixture the direct sequence, where the components are remove removed in the order of their the volatility. Like the first sequence A is taken off first, B is taken of second, C is taken off third and then D, E is split after removal of B from A, B, C, D after removal of A and B from the A, B, C, D, E mixture we are left with a ternary system C, D, E.

Now C, D, E can be split in direct and indirect sequence. So, the first one that we just say saw just saw was a direct sequence, there could be an indirect sequence also like C and D taken off as the top product and E taken off as a bottom product in the third column. And then mixture C, D is split individually. Now, another sequence is that after removal of A you are left with a quaternary mixture B, C, D, E then you split as mixture of two components like B, C and D, E. And then you are left with two binary mixtures B, C and D, E.

Then these are split individually, into components into columns B, C and D, E. Then after removal of A you have another choice of splitting the quaternary mixture in indirect sequence, like B, C, D together taken off as the top product, E taken off the heaviest component taken off as the bottom product and then the mixture B, C, D is split again in direct and indirect sequence. So, in this way you can write 14 different sequences, which are shown you can yourself try to write these sequences.

You can take this is an exercise and the answer is already with you and then you try to make your own sequences and compare with the answer which is there on the screen now. Now, as in the number of components further increase the complexity even increases. For example, if you have six component mixture, then you have as many as 42 different sequences.

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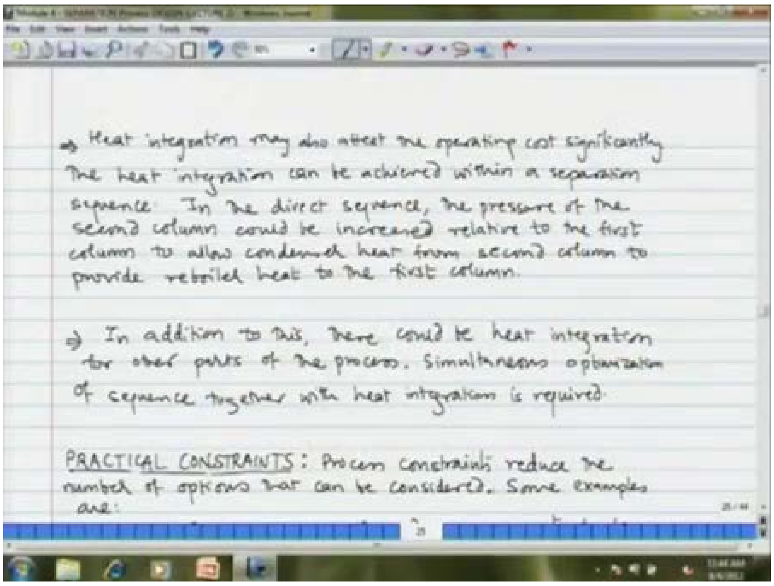

⇒ Heat integration may also affect the operating cost significantly. The heat integration can be achieved within a separation sequence. In the direct sequence, the pressure of the second column could be increased relative to the first column to allow condensed heat from second column to provide reboiled heat to the first column.

⇒ In addition to this, there could be heat integration for other parts of the process. Simultaneous optimization of sequence together with heat integration is required.

PRACTICAL CONSTRAINTS: Process constraints reduce the number of options that can be considered. Some examples are:

Choosing between the sequence is now a major decision mean, as the number of alternatives increase, the difficulty in making decision also increases because you have to evaluate the cost associated with each of the sequence. And there are two cost first is the fixed cost and second is the operating cost. Now, heat integration may also effect the operating cost significantly, heat integration can be achieved within a separation sequence as well. So, the everywhere you are going to require four columns so is it possible to couple these columns.

For example, is it possible that you increase the operating pressure of one column. So, that the condensing temperature of the vapor increases and then you try to condense these vapors in the re-boiler of the second column by reducing the pressure there. So, that you create a sufficient temperature difference in the re-boiler. Suppose, it is a kettle type of re-boiler then you have to have always nuclear boiling, you have to avoid film boiling to have nuclear boiling you have to have delta t at least 40, 50 degree centigrade. So, that temperature difference all these things you have seen in the course of heat transfer in NPTEL.

So, these are my as I said we are not here to learn principles, but only application. So, in one column you increase pressure so that the condensing temperature of the vapor increases. In the second column you reduce pressure so that the boiling point of the liquid decreases and try to achieve the desired delta t to have the heat integration. In addition to this there could be heat integration from other parts of the process, as I said like for example, take the HDA process. The stream which emerges from the reactor is at very high temperature in pressure. So, it has sufficient heat in it to drive the re-boilers of all the columns.

So, that heat integration is also possible you try to condense, you try to couple this the streams with surplus heat with the re-boilers, or let us say if you have cold streams then those can run your condensers and try to achieve heat integration. So, that the operating cost goes down, the simultaneous optimization of sequence together with heat integration is required. So, while choosing the sequence this is another factor that will come into picture.

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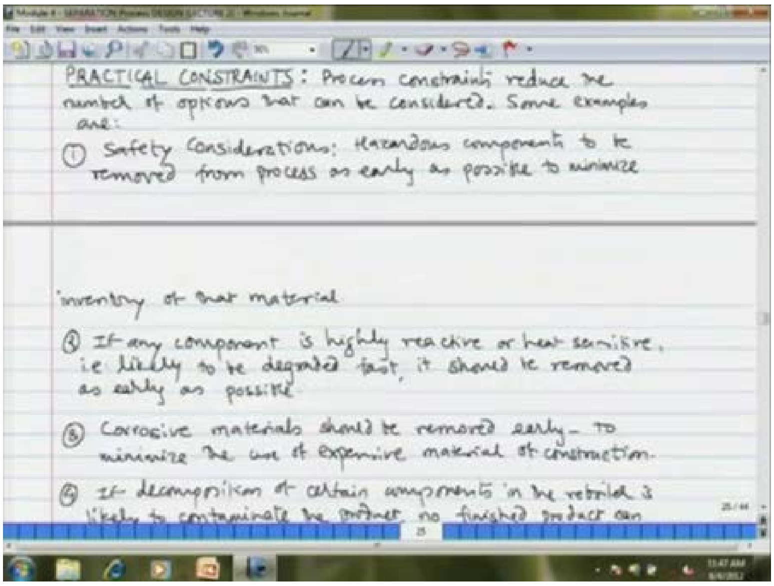

PRACTICAL CONSTRAINTS: Process constraints reduce the number of options that can be considered. Some examples are:

① Safety Considerations: Hazardous components to be removed from process as early as possible to minimize inventory of that material

② If any component is highly reactive or heat sensitive, i.e likely to be degraded fast, it should be removed as early as possible

③ Corrosive materials should be removed early - to minimize the use of expensive material of construction.

④ If decomposition of certain components in the reboiler is likely to contaminate the product, no finished product can

There are some practical constraints, although one sequence may look significantly better over other, there could be practical constraints. Now, what are these constraints first is the safety consideration, hazardous components to be removed from the process as early as possible to minimize the inventory of that material. So, even if one particular sequence has higher, higher energy integration possibility or least cost, if it treats the if it has a hazardous component, which stays in the process for longer time, then you have the problem. So, you may have to go for another sequence which removes this component early. Then if any component is highly reactive or heat sensitive or likely to be degraded fast with heat, then it should be removed as soon as possible.

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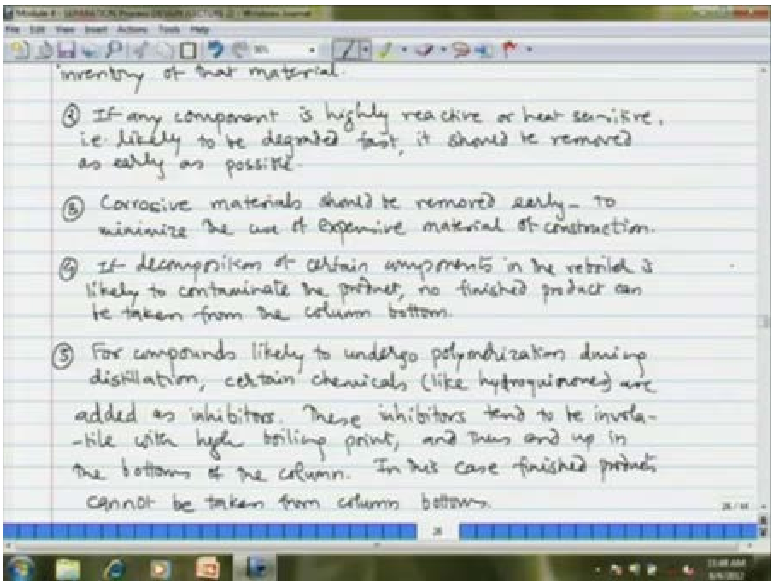

inventory of that material.

② If any component is highly reactive or heat sensitive, i.e. likely to be degraded fast, it should be removed as early as possible.

③ Corrosive materials should be removed early - to minimize the use of expensive material of construction.

④ If decomposition of certain components in the reboiler is likely to contaminate the product, no finished product can be taken from the column bottom.

⑤ For compounds likely to undergo polymerization during distillation, certain chemicals (like hydroquinones) are added as inhibitors. These inhibitors tend to be involatile with high boiling point, and thus end up in the bottoms of the column. In this case finished products cannot be taken from column bottoms.

So, try to get that sequence which takes off that particular material in the first or second column itself. Then third is corrosive material these need to be removed early because like when we talked of sequence, we never talked of the material of construction alike other details. Now, A, B, C, D, E all together may not be may not have same properties like for example, is component E is highly corrosive. Then for the distillation column treating A, B, C, D, E mixture you may require plates and shell made up of stainless steel, S S 302, 304 which is an expensive material.

So, and as long as e remains in that that flow stream everywhere you will have to have that material. So, although the sequence, which is a direct sequence, which removes E in the very last column may look attractive from energy integration point of view, from the mechanical design of the columns is concerned. You have to go sequence which removes E as soon as possible because you have to if you keep E in the process for long time you have to design all columns made up of expensive material that increases the fix cost. So, somewhere tradeoff is important you that you try to achieve tradeoff between fixed cost and operating cost, so that is it.

Then if the decomposition of certain components in the re-boiler is likely to happen, or it is likely to contaminate the process, then no finish product can be taken off from the column bottom. So, we have to remove these components as soon as possible for compounds that are likely to undergo polymerization during distillation, you have to take special care. In this case certain chemicals are added like hydroquinone is one of the chemicals, which are inhibitors which inhibit the polymerization. However, these chemicals are usually, high boiling components and these inhibiters end up in the bottom of the column and they contaminate the product there.

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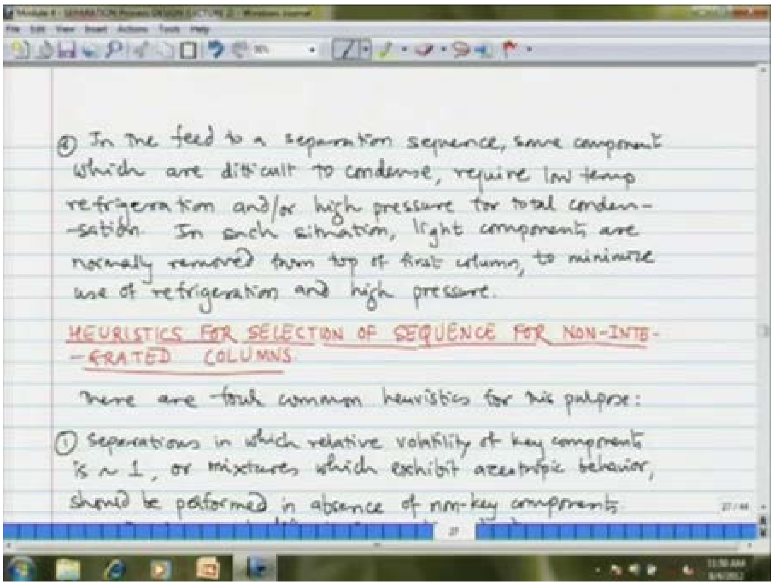

④ In the feed to a separation sequence, some component which are difficult to condense, require low temp refrigeration and/or high pressure for total condensation. In such situation, light components are normally removed from top of first column, to minimize use of refrigeration and high pressure.

HEURISTICS FOR SELECTION OF SEQUENCE FOR NON-INTEGRATED COLUMNS

There are four common heuristics for this purpose:

① Separations in which relative volatility of key components is ~ 1, or mixtures which exhibit azeotropic behavior, should be performed in absence of non-key components

So, in this case the finished product cannot be taken off from the column bottom. Then in the column towards in the in the feed towards separation sequence, some components which are difficult to condense require low temperature, or refrigeration, or high pressure for total condensation. In such situation try to remove them as soon as possible, try to remove them from the top of the first column itself. So, as to minimize the cost of utilities because the longer that to keep them in the process, in every column you may require refrigeration or high pressure, which are expensive utilities. So, in order to limit the operating cost, you have to remove them as soon as possible.

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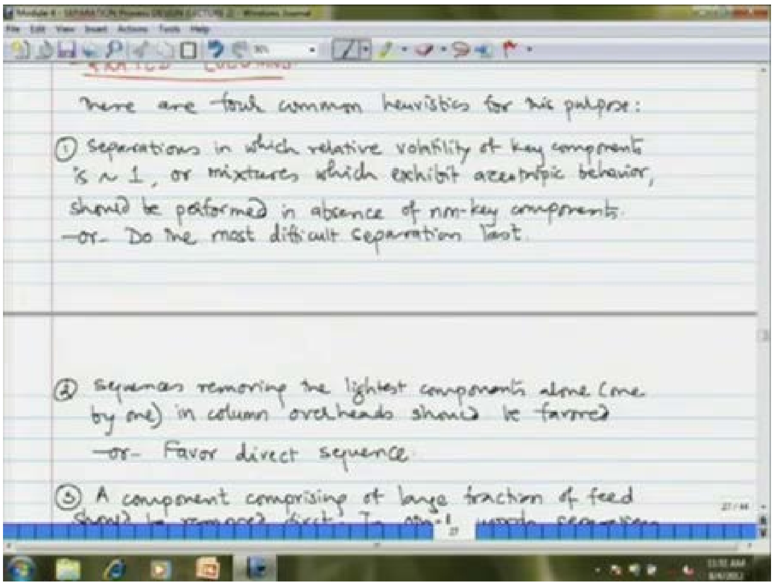

There are four common heuristics for this purpose:

① Separations in which relative volatility of key components is ~ 1, or mixtures which exhibit azeotropic behavior, should be performed in absence of non-key components.
-or- Do the most difficult separation last.

② Sequences removing the lightest components alone (one by one) in column overheads should be favored
-or- Favor direct sequence

③ A component comprising of large fraction of feed

There are some basic heuristics that I will mention here, we shall see these things in greater detail in the next lecture, but I am just mentioning these heuristics for selection of column sequence, for nonintegrated columns, which means we are not considering the heat integration at this moment. So, there are 4 common heuristics, first separation in which the relative volatility of key components is close to 1, or the mixture, which exhibit azeotropic behavior should be performed in the absence of non key components, which I just mentioned that whenever, you are splitting this close boiling mixture try to have as little contamination from other things as possible. That means, you keep them and as a last separation, or do the most difficult separation last. This is first heuristic.

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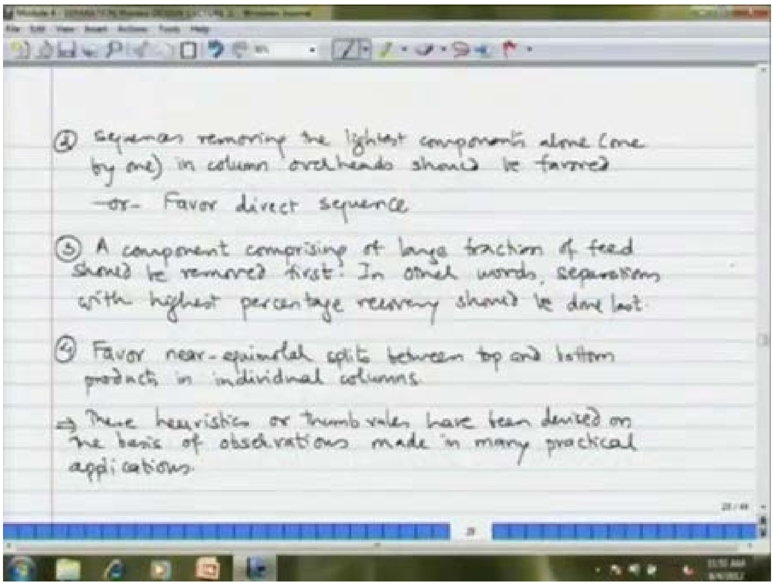

② Sequences removing the lightest components alone (one by one) in column overheads should be favored

—or— Favor direct sequence

③ A component comprising of large fraction of feed should be removed first. In other words, separations with highest percentage recovery should be done last.

④ Favor near-equimolar split between top and bottom products in individual columns.

⇒ These heuristics or thumb rules have been devised on the basis of observations made in many practical applications.

The second heuristic is that sequence, sequences removing the lightest component alone one by one in a column overhead should be favored, or the direct sequence should be favored. The components removed in the order of the relative volatility, highest relative volatility first and decreasing relative volatility. Then thirdly a component comprising of large fraction of the feed should be removed first, this is to limit the total volume that you are handling at any time that because every time you have to pump, this much of liquid that increases the pumping cost. Then you have to boil that liquid that increases the energy cost.

So, in other words separation with the highest percentage recovery should be done last. And then fourthly you have to favor near equimolar splits between top and bottom product, in individuals columns this helps in energy integration to have nearly equimolar split between top and bottom product individual columns, but remember that these are only heuristics these are not laws or rules, these are only heuristics these are thumb rules which have been devised on the basis of observations, made in many practical applications. So, these have come out of experience.

Case to case basis these may vary like all of these heuristics may not be applicable in each case. So, depending on the process that we have, depending on the flow rates and selectivity's many, many factors you have to decide the columns sequence. So, in the next lecture, we shall see an example of deciding the column sequence another thing that you will see is that all these heuristics are qualitative, they do not do any quantitative answer. So, we shall see as how these heuristics could be applied in the best possible way for separation of any mixture. If these heuristics are not applicable, then what other guidelines could be used for deciding the column sequence.