Process Design Decisions and Project Economics
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Module - 4
Design of Separation Processes
Lecture - 17
General Introduction
(Types of Separation Processes and Criteria for Selection of the Processes)

Welcome, will start the 4 module of our course that is Design of Separation Processes. In the previous 2 modules, that is the input output structure of the flow sheet and the recycle structure of the flow sheet. We saw as how we can fix the total mass and energy balance of the processes, and also go for the rector design. The next stage in the design of the process is the design of separation processes. The effluent that will come out of the reactor will have several components, several products, bi products, un-reacted reactants, and all of these need to be carefully separated before purification of the main product. So, we shall see as what are the guidelines that govern the design of a separation process.

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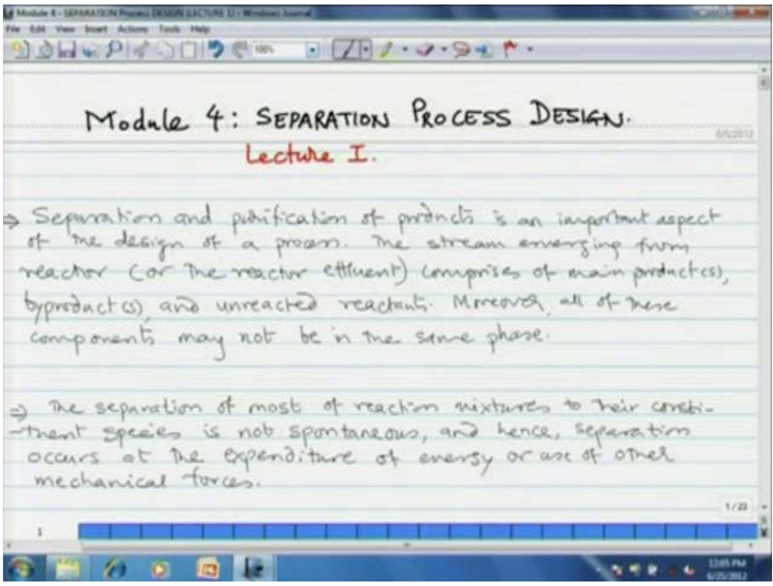

Module 4: SEPARATION PROCESS DESIGN.

Lecture I.

⇒ Separation and purification of products is an important aspect of the design of a process. The stream emerging from reactor (or the reactor effluent) comprises of main product(s), byproduct(s), and unreacted reactants. Moreover, all of these components may not be in the same phase.

⇒ The separation of most of reaction mixtures to their constituent species is not spontaneous, and hence, separation occurs at the expenditure of energy or use of other mechanical forces.

Separation and purification of the products is an important aspect of the design of the process the stream emerging from the reactor comprises of main products that I just said, the separation of most of the reaction mixtures to their constituent species is not spontaneous. And therefore, the separation occurs at the expenditure of energy or use of other mechanical forces.

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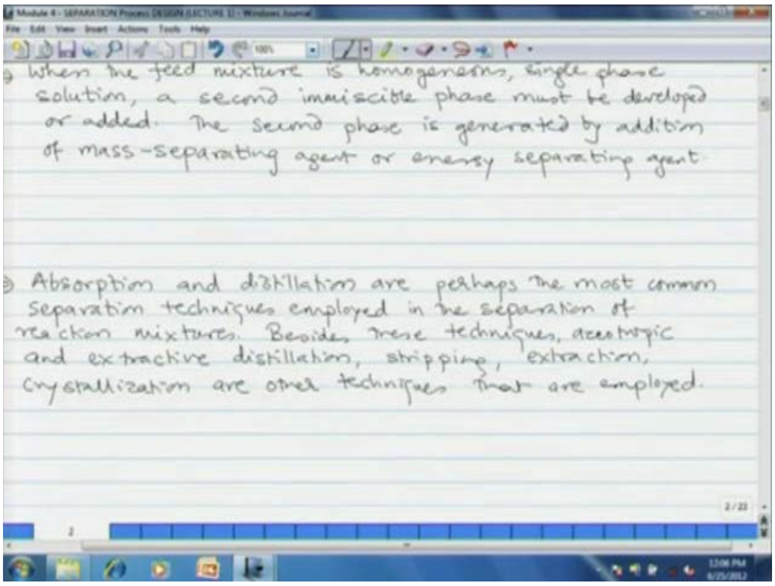

- When the feed mixture is homogeneous, single phase solution, a second immiscible phase must be developed or added. The second phase is generated by addition of mass-separating agent or energy separating agent.
- Absorption and distillation are perhaps the most common separation techniques employed in the separation of reaction mixtures. Besides these techniques, azeotropic and extractive distillation, stripping, extraction, crystallization are other techniques that are employed.

When the feed mixture is homogenous or a single phase solution then a second immiscible phase has to be developed or aided and the second phase is generated by either addition of a mass separating agent or energy separating agent. Now, the most common operations used in chemical industry are absorption and distillation for separation of the processes. In the NPTEL course of mass transfer you have learnt the basic principles of these processes in great depth.

So, I will not go for repetition of it, but we shall see these two processes from economics point of view, that is the additional a feature of this particular a course of NPTEL. In addition to absorption and distillation we also have isotropic or extractive, distillation, stripping, extraction, crystallization, as the techniques for separation of the components of the reactor effluent.

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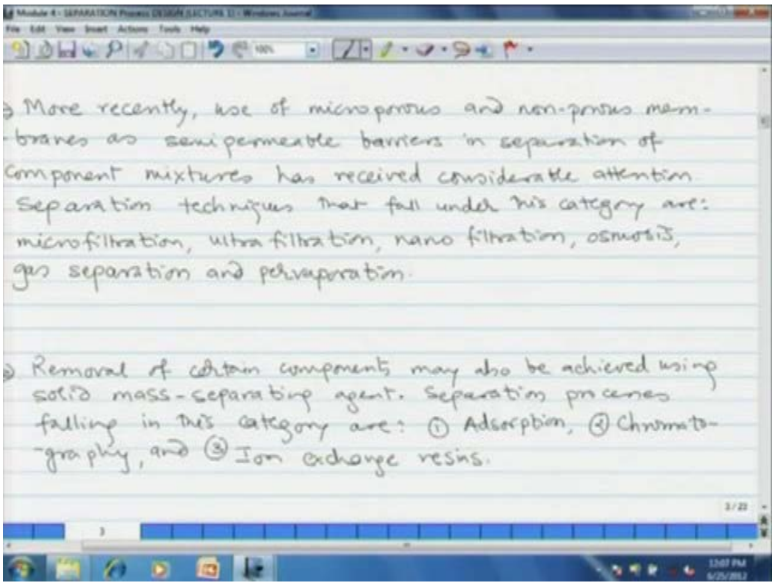

→ More recently, use of microporous and non-porous membranes as semipermeable barriers in separation of component mixtures has received considerable attention. Separation techniques that fall under this category are: microfiltration, ultra filtration, nano filtration, osmosis, gas separation and pervaporation.

→ Removal of certain components may also be achieved using solid mass-separating agent. Separation processes falling in this category are: ① Adsorption, ② Chromatography, and ③ Ion exchange resins.

More recently use of micro porous and non porous membranes as semi permeable barriers in the separation of component mixtures has received considerable attention. The separation techniques that fall under this category are micro filtration, ultra filtration, Nano filtration, osmosis, reverse osmosis gas separation and evaporation. Now, these separation.

Now, these separation processes have merit that these are a less energy intensive as compared to distillation and absorption. In case of absorption the particular component in the gaseous mixture is absorbed in the solvent however, we need further distillation to recover this particular component and recycle of the solvent. So, although absorption itself could be absorption on it is own could be less energy intensive but coupled with the distillation for the recovery of the solute it becomes energy intensive process.

The advantage of these membrane processes is that they are far less energy intensive as compared to absorption and distillation however, there are certain limitations of their own that we shall discuss later on in this lecture. Removal of certain components may also be achieved using solid mass separating agent, now the a separation processes that fall in this category are adsorption chromatography and ion exchange resin.

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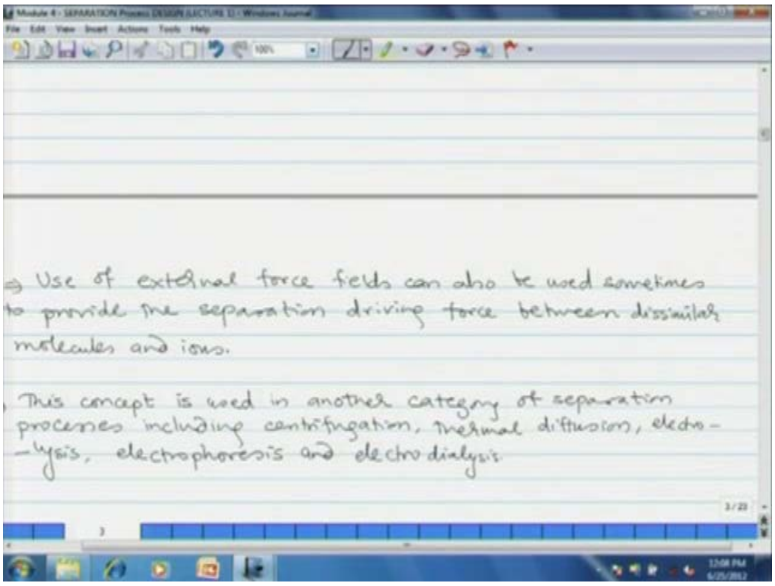

⇒ Use of external force fields can also be used sometimes to provide the separation driving force between dissimilar molecules and ions.

⇒ This concept is used in another category of separation processes including centrifugation, thermal diffusion, electrolysis, electrophoresis and electro dialysis

The use of external force field can also be used sometimes to provide the separation dragging force between dissimilar molecules and ions for example, the usual method of sep separation of solid particles from reaction mixture is that of settling. However, if the particles are very small then their terminal settling velocity is very small and then it takes very long time for separation of this particles as you have already seen a in the course of fluid mechanics and unit operations.

In some cases they even form stable emulsion where the forces on the particles are perfectly balanced and it remains suspended in the solution, in such cases we have to apply additional force it could be either mechanical force. Such as centrifugation or it could be electrical force as in case of electrolysis or electrophoresis or electrostatic precipitation. So, these are some of the additional techniques for separation of the components of a reactor effluent.

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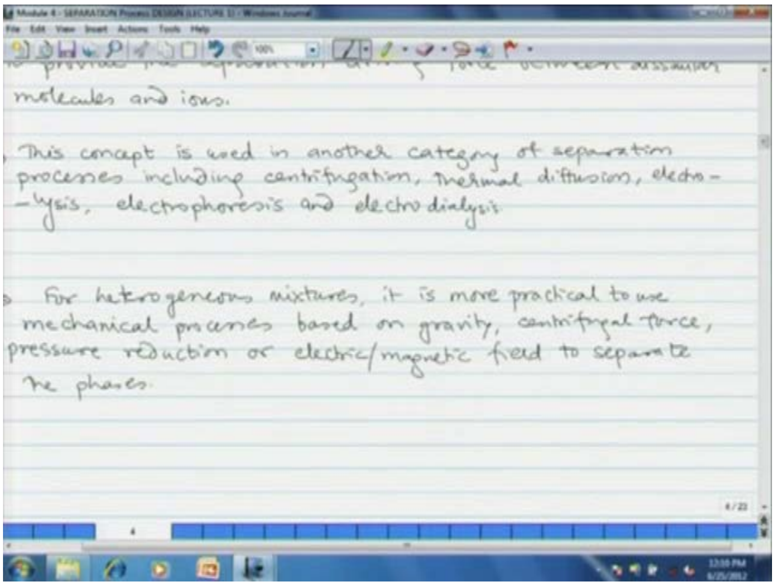
molecules and ions.

This concept is used in another category of separation processes including centrifugation, thermal diffusion, electrolysis, electrophoresis and electro dialysis

For heterogeneous mixtures, it is more practical to use mechanical processes based on gravity, centrifugal force, pressure reduction or electric/magnetic field to separate the phases.

As far as heterogeneous mixtures are concerned, it is more practical to use mechanical processes based on gravity, centrifugal force, pressure reduction or electric magnetic field to separate the phases.

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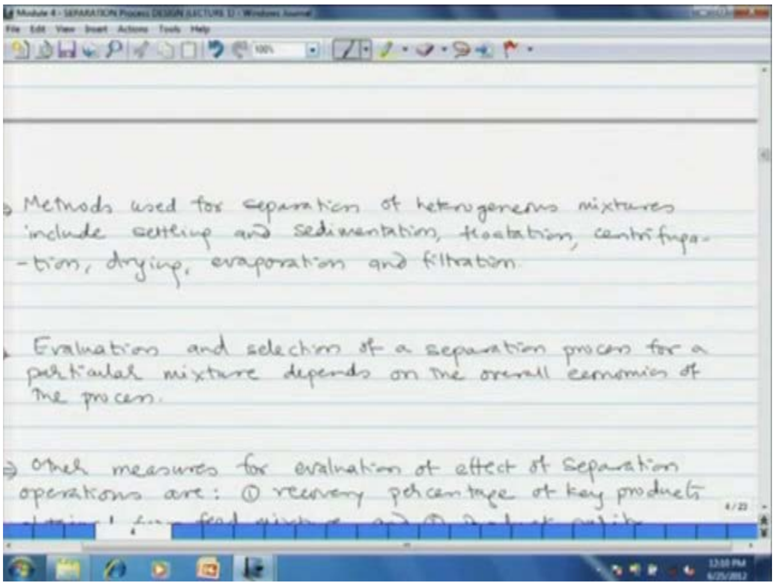
Methods used for separation of heterogeneous mixtures include settling and sedimentation, floatation, centrifugation, drying, evaporation and filtration.

Evaluation and selection of a separation process for a particular mixture depends on the overall economics of the process.

Other measures for evaluation of effect of separation operations are: ① recovery percentage of key products

Methods used for separation of heterogeneous mixtures include settling and sedimentation which I just mentioned however, we need a certain particular size or the density difference between solid and fluid to achieve, effective, settling and sedimentation. Then there is process of floatation where you generate a froth like generate bubbles in the a solution of the solid mixture and solvent, and certain particles from the solid mixture attached to the bubble surface that we are going to, see in greater details later in this core a in this lecture.

Then centrifugation which I just mentioned a use of centrifugal force then drying which is basically for removal of moisture, evaporation and filtration. Now, filtration is another cousin of membrane separation but this filtration is completely mechanical, where the particles are separated by only size exclusion. Evaluation and selection of separation process for a particular mixture depends on the overall economics of the process.

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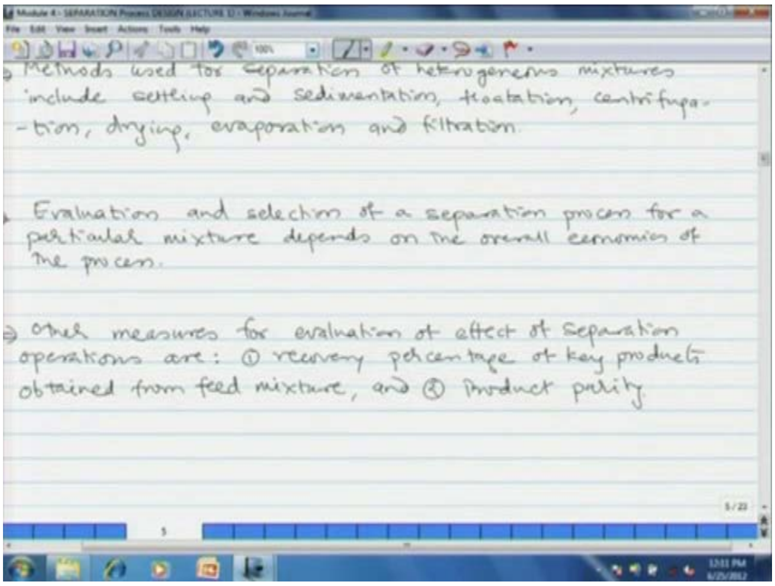

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⇒ Other measures for evaluation of effect of separation operations are: ① recovery percentage of key products obtained from feed mixture, and ② Product purity

We shall see a the criteria for selection of the processes a very shortly however, economics comes into picture there could be several alternatives for separation of the same reaction mixture. However, we have to assess based on the throughput of the molecule that is to be processed the concentration that is there in the reactant effluent of different components based on several factors we have to select the best possible process, that will give most effective as well as economic operation this is in terms of both fixed and operating cost.

The other majors for evaluation of the effect of separation, operation are recovery percentage of the key products obtained from the feed mixture and. Secondly, the product purity. Same purity may not be obtained from different processes. So, depending on the cost of the component that is going to be recovered against purity we have to select certain process. In some cases we may go for energy intensive process for separation for the recovery of particular product. Because it gives high purity and the cost that purified product draws is a worth the a or which upsets the cost of a separation.

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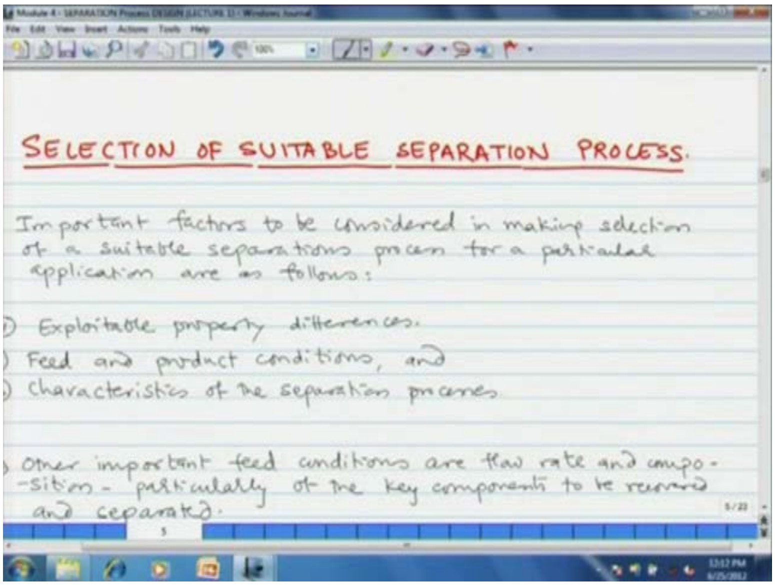

SELECTION OF SUITABLE SEPARATION PROCESS.

Important factors to be considered in making selection of a suitable separations process for a particular application are as follows:

) Exploitable property differences.
) Feed and product conditions, and
) Characteristics of the separation processes

Other important feed conditions are flow rate and composition - particularly of the key components to be recovered and separated.

We shall see the selection of a suitable separation process, what criteria we have to use for the selection of the process to separate the same reactor effluent. The important factors to be considered in making selection of the suitable separation process for a particular application are first the exploitable property difference whether it is, physical property or chemical property then the feed and product condition which I just mentioned that what is the volume of the feed, what is the phase of the feed and what is what are the number of products. That are available in that are present in the reactor effluent. Then the characteristics of the separation processes other important feed conditions are the flow rate and composition that I just mentioned and particularly the key components that are to be recovered and separated.

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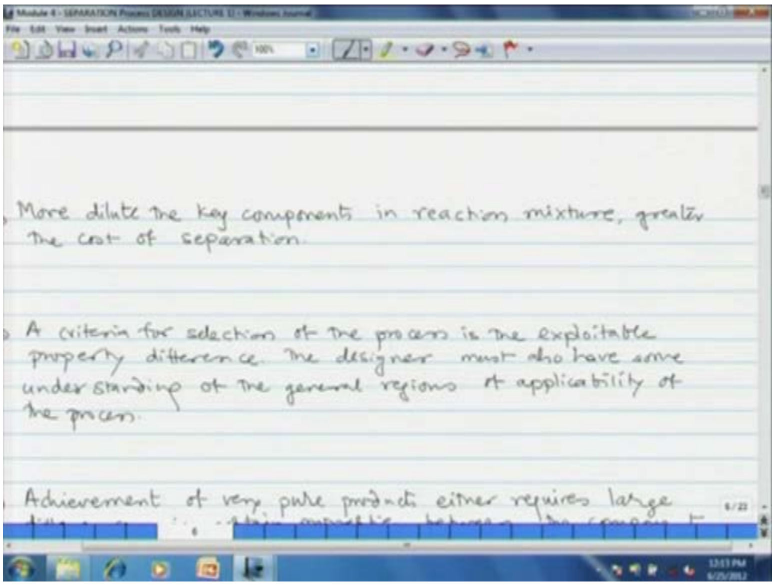
- More dilute the key components in reaction mixture, greater the cost of separation.
- A criteria for selection of the process is the exploitable property difference. The designer must also have some understanding of the general regions of applicability of the process.
- Achievement of very pure products either requires large

Now, one obvious feature of the whole separation process is that how concentrated is the reaction mixture to be separated. The more dilute the key components in the reaction mixture the greater the cost of separation, that is obvious, because it you have to treat a very large amount of volume for recovery of very small amount of product. So, the cost of operation increases with the volume that is been treated however, the cost of product even with 99.999 percent recovery is much lesser.

Then the another criteria for selection of process is the exploitable property difference. The designer must also have some understanding of the general regions of applicability of the process, achievement of very pure products either requires large differences in certain properties between, components to be separated or requires large number of stages for separation. You have already seen these things in the course of mass transfer in this NPTEL. We you must have seen that as the number of stages increases, in a distillation column the product purity increases.

However, to achieve there is again a trade of between the reflux ratio and the number of stages, if you have greater reflux then you have lesser number of stages but the amount of product removal is less also the volume that is under treatment at a certain time increases,. So, the column size diameter increases, but the number of plates decreases,. So, there is a trade off that we have to see.

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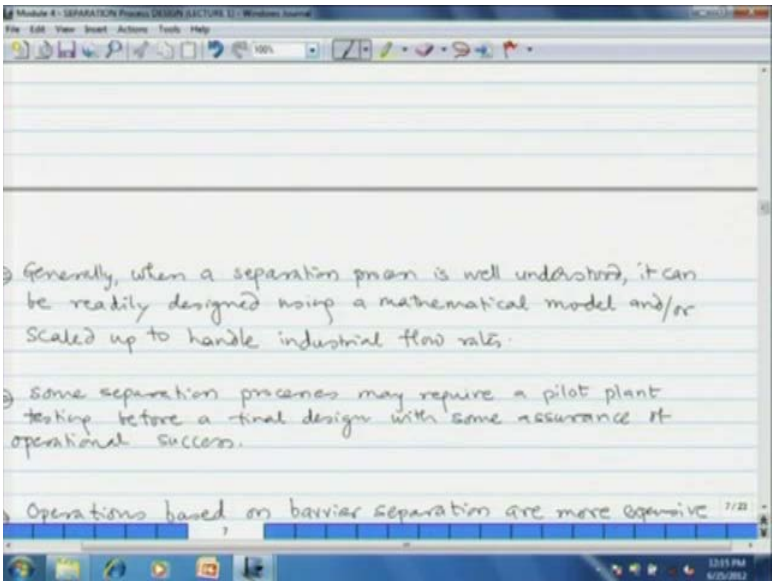

- Generally, when a separation process is well understood, it can be readily designed using a mathematical model and/or scaled up to handle industrial flow rates.
- Some separation processes may require a pilot plant testing before a final design with some assurance of operational success.
- Operations based on barrier separation are more expensive

Generally, when separation process is well understood it can be readily designed using the mathematical model and scaled up to handle the industrial fluorides. Now, elaborate mathematical models are available for processes which have been which have in operation in chemical industry for last one century that is absorption then liquid extraction, distillation there are certain professional software's which give very elaborate design of this particular processes.

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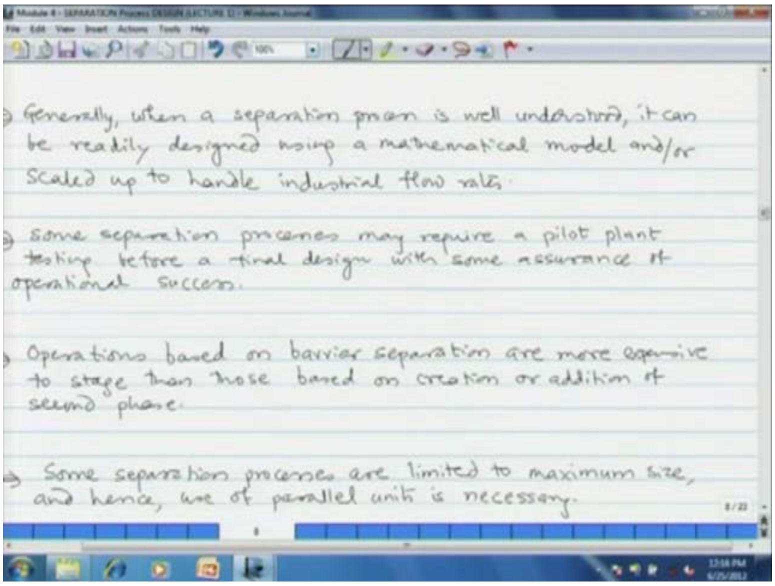

- Generally, when a separation process is well understood, it can be readily designed using a mathematical model and/or scaled up to handle industrial flow rates.
- Some separation processes may require a pilot plant testing before a final design with some assurance of operational success.
- Operations based on barrier separation are more expensive to stage than those based on creation or addition of second phase.
- Some separation processes are limited to maximum size, and hence, use of parallel units is necessary.

So, once the mechanics of the process is understood it is very easy to develop a mathematical model for that particular process and then scale up according to the dissimilation results using the mathematical model. Some separation processes may require a pilot plan testing before final design with some assurance of the operational success, operations based on barrier separation or more expensive to stage than those based on creation on addition of a second phase.

For example, membrane modules are difficult to stage as compared to the distillation or absorption, which is creation of addition of second phase now some separation processes are limited to maximum size. Hence, unit of parallel hence use of parallel units is necessary if your throughput is very large. Then the size of one single unit creating the entire through put becomes and in practically large, in that case we have to go for several parallel units. In some cases there is problem of fouling, there is problem of problem of clogging some operational problems. Therefore, it is always better to put some equipment under stand by which means that when one equipment is under repair or maintenance.

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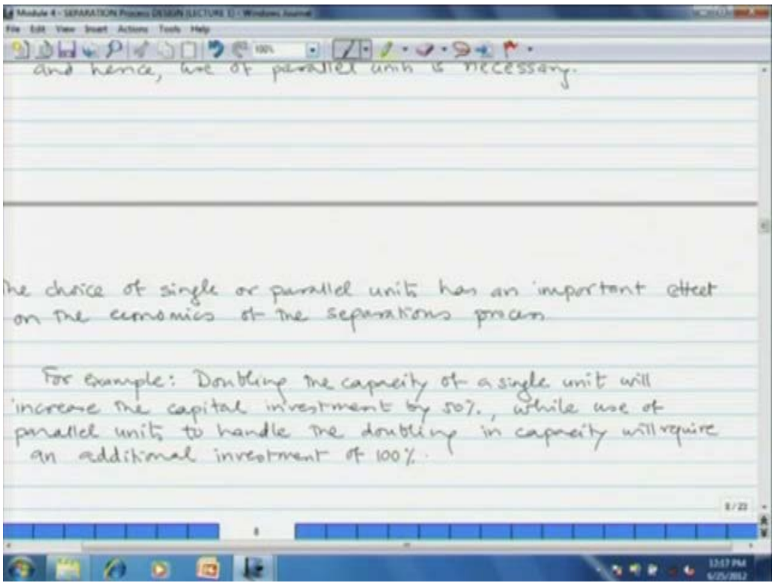

and hence, use of parallel units is necessary.

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For example: Doubling the capacity of a single unit will increase the capital investment by 50%, while use of parallel units to handle the doubling in capacity will require an additional investment of 100%.

The second unit can operate and a take the load of overall treatment, which I choice of single or parallel units has an important effect on the economics of the separation processes. Obviously, there is a tradeoff here, if you reduce, if you split the total feed to be treated into two parts then you will have two equipments of moderate size but the individual cost of operation goes down. So, two distribution columns will the fixed cost will be higher because we are having two units separate units there is always an economy of scale up if you have one big single unit it will definitely cost lesser than two single units of the same total capacity. Doubling of a capacity of a single unit will increase the capital investment by 50 percent while use of parallel units to handle the doubling yield capacity will require an additional investment of 100 percent that is what is a obvious from the discussion that we just had.

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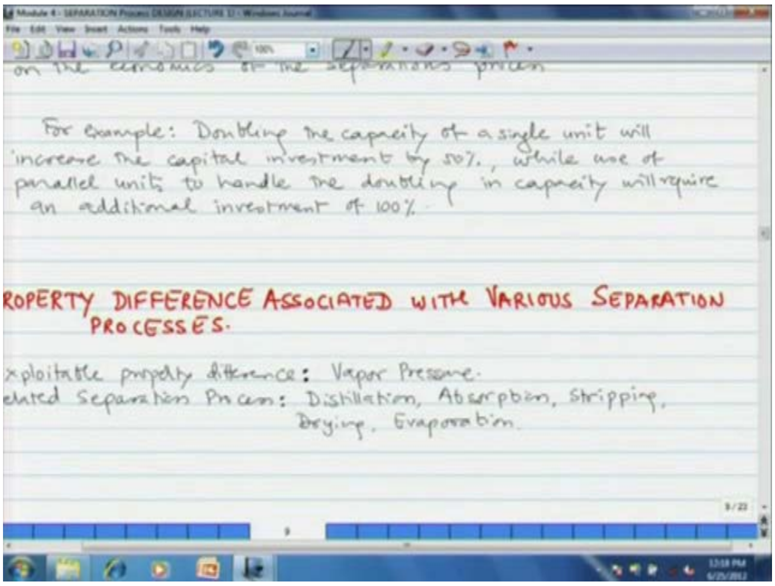

on the economics of the separations process

For example: Doubling the capacity of a single unit will increase the capital investment by 50%, while use of parallel units to handle the doubling in capacity will require an additional investment of 100%

ROPERTY DIFFERENCE ASSOCIATED WITH VARIOUS SEPARATION PROCESSES.

xploitable property difference: Vapor Pressure.
elated Separation Process: Distillation, Absorption, Stripping, Drying, Evaporation.

Then let us see, what are the property differences as associated with various separation processes. As I said that we have to identify the exploitable property difference of different components that are present in the reaction mixture, so as to achieve the separation. One simple separation like for example, separation by sedimentation there the difference is of density. So, density is the property that is exploited. But, let us see what are we have seen. So, many operations of a separation we what operation is suitable under what property difference are available.

Now, the easiest or the more obvious property difference is the vapor pressure, most of the many processes are liquid phase processes therefore, vapor pressure of the liquid is a very handy tool or handy property for design of the separation processes. The separation process which is based on difference of vapor pressures of different components in the reaction mixture are distillation, absorption, stripping, drying and evaporation.

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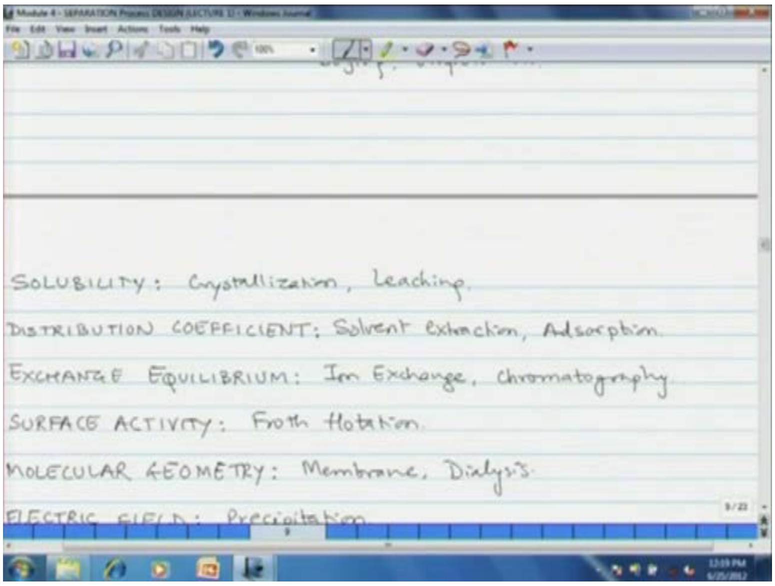

SOLUBILITY: Crystallization, Leaching.

DISTRIBUTION COEFFICIENT: Solvent extraction, Adsorption.

EXCHANGE EQUILIBRIUM: Ion Exchange, Chromatography.

SURFACE ACTIVITY: Froth flotation.

MOLECULAR GEOMETRY: Membrane, Dialysis.

ELECTRIC FIELD: Precipitation

Solubility is another property and crystallization or leaching processes use make a they make use of this property for the design, differences in solubility of different components in a particular solvent. So, that is a basis for design of a crystallization or leaching processes, then the distribution coefficient. When a particular component when a particular reaction mixture is contacted with the solvent, then not all of the components that are initially present in the reaction mixture have equal affinity or solubility in the solvent which is contacted.

So, some a components gets selectively soluble at or diffuse to the second phase that is added that is a solvent, and this helps us in achieving some separation between the components. So, the distribution coefficient or partitioning coefficient is the basis for design of process of solvent extraction and adsorption. Then the exchange equilibrium this is a property for a ion exchange resin or a chromatography then the surface activity.

That is used for froth floatation that there is some surface activity some attractive force is between certain solid particles and the bubble surface which is of hydrophobic character. So, in froth floatation bubbles are generated and particles from the a when you have a solid mixture then particles of a certain component get attached to the bubble due to the surface activity.

That is the property for design of froth floatation processes, then the molecular geometry that is for membrane and dialysis then the electric field for precipitation then the particle size is a property for filtration, screening, settling and sedimentation processes then particle size and density together are the properties for the design of centrifugation or classification, thickening, decantation of scrubbing processes.

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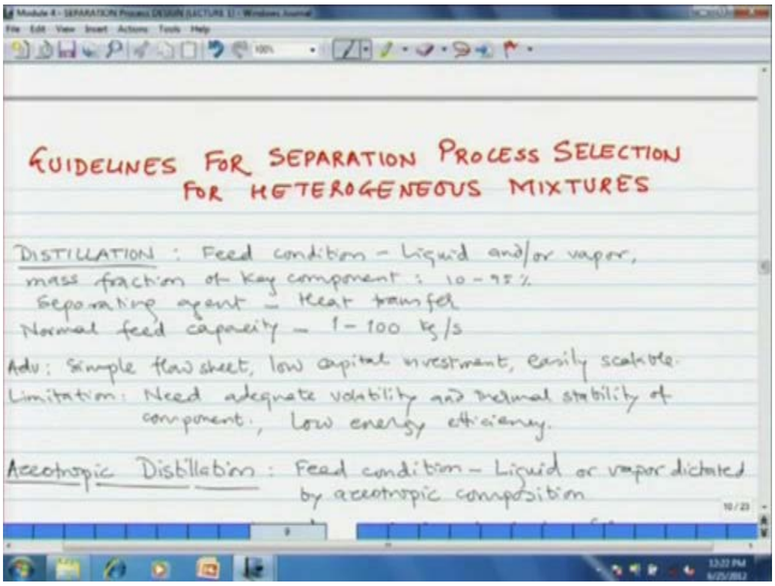

GUIDELINES FOR SEPARATION PROCESS SELECTION
FOR HETEROGENEOUS MIXTURES

DISTILLATION : Feed condition – Liquid and/or vapor,
mass fraction of key component : 10 – 95 %
Separating agent – Heat transfer
Normal feed capacity – 1 – 100 kg/s
Adv : Simple flow sheet, low capital investment, easily scalable.
Limitation : Need adequate volatility and thermal stability of component, Low energy efficiency.

Azeotropic Distillation : Feed condition – Liquid or vapor dictated by azeotropic composition

Let us see certain general guidelines for selection of a separation processes. I would like to mention that these are very broad guidelines, these are not applicable like hard fas for each and every process. But, these guidelines give us a general view or like thumb rules for selection of a separation process. So, let us first take the process of distillation which is one of the most common processes used in chemical industry. Now, in this case the feed condition of the reaction mixture has to be liquid or some mixture of vapor or it could be even be complete vapor.

Then the mass fraction of the key component has to be between 10 to 95 percent it cannot be too. Dilute below 10 percent you have to handle very large amount of liquid as I mentioned at the beginning in the lecture distillation is a energy intensive operation because we have to buy a large amount of liquid. However, people still go for distillation because of the flexibility of product purity that can be obtained from the distillation and also to know how that is already established for distillation column design.

For this two things people would like to go that the design would like to go for a operation of distillation for separation of a reaction mixture. Normal feed capacity is between 1 to 100 kg per second then advantage is simple flow sheet that I just mentioned it is a well-established process lot of data is available. So, design of a very efficient column is easy you can even energy integrate these columns as a we shall see subsequently that if, you have size of columns then you can alter the pressures in these columns.

So, that the vapor from one column can drive the revolver of the second column that can be achieved by adjusting the pressure that is energy integration with in a distillation sequence. So, distillation is a very well established science and that is the major advantage of it then low capital investment easily scalable. You can go for large scale the limitation however, is that of need of adequate volatility and thermal stability of the component if the volatility is very close then you will end up with very large number of plates, very tall column that will give you enough capital investment.

And then if the components are thermally unstable then you will have to control the temperature very carefully another problem with the distillation is when extremes of temperature and pressures are encountered. For example, if the vapors that are in the system like that the at the re boiler a sorry at the condenser the vapors at the condenser. If the condenser temperature lesser than let us say 20 degree centigrade, which is a typical entry temperature of cooling water then you will have to give a special a utility for that condenser.

For example, let us say chilled water, if the condenser temperature is even below let us say below 5 degree centigrade or below a even 0 degree centigrade then you will have to go for refrigeration. So, typically for temperatures lesser than minus 40 degrees or greater than 250 degrees the cost of added refrigeration or heating involved escalates rapidly and then the operating cost also goes up. Same thing holds for re boiler also the, most common utility for re boiler is steam that is either medium pressure or high pressures steam but that is up to 250 degrees.

If the boiling point of your reaction mixture bottoms of the distillation is greater than 250 degrees, then you have to go for some special utility, Such as, for furnace or diaphragm liquid that increases the cost, you can have a pressure variation like for example, if the boiling point of the distillation a column bottoms is greater than 300 degrees then you can apply vacuum to lower it. So, that you can still use steam at the same time you have to see that how the condensation temperature of the vapor also, decreases with pressure it should not go below twenty degrees, so that you will have to use the refrigeration. So, this is all a big trade off, but distill typically if you see the typical temperature range of a distillation column it is between minus 40 to 250 degree centigrade above which the operating cost escalates the total cost of operation.

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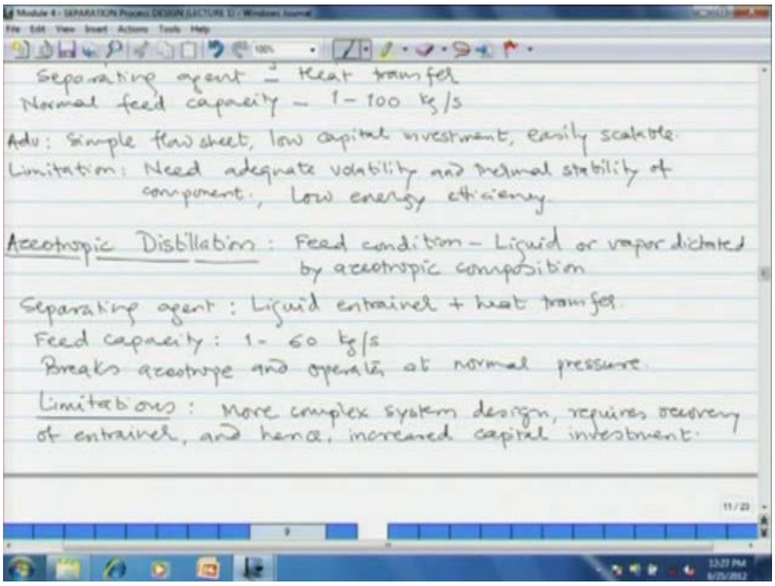

Separating agent – Heat transfer
Normal feed capacity – 1 – 100 kg/s
Adv: Simple flow sheet, low capital investment, easily scalable.
Limitation: Need adequate volatility and thermal stability of component., Low energy efficiency.

Azeotropic Distillation: Feed condition – Liquid or vapor dictated by azeotropic composition

Separating agent: Liquid entrainer + heat transfer.
Feed capacity: 1 – 60 kg/s
Breaks azeotrope and operates at normal pressure.
Limitations: More complex system design, requires recovery of entrainer, and hence, increased capital investment.

Then there is aziotropic distillation by definition is to separate the components which have same volatility. Like for example, ethanol water mixture achieves aziotropic composition after about 87 percent mole percent ethanol. Then at a time the relative volatility becomes one and no more distillation can be applicable. In that case a we have to go for aziotropic distillation, now the field condition for aziotropic distillation has to be either liquid or vapor which is dictated by the aziotropic composition the separating agents is a liquid entrainer or and plus heat transfer.

So, you have to add an entrainer which bounds to which forms the a temporary complex a with one of the components and then a that complex has a volatility greater than 1 with the other component and then you can, achieve the separation and then later on the that particular complex is broken to achieve a to recover the intoner which is recycled. But, addition of the third component increases the cost the aziotropic distillation itself is much complex operation than mere distillation you have to be very careful while designing a column. The degrees of freedom are less, and therefore the cost prizes. The feed capacity is typically 1 to 60 kg per second distillation may also be unsuitable.

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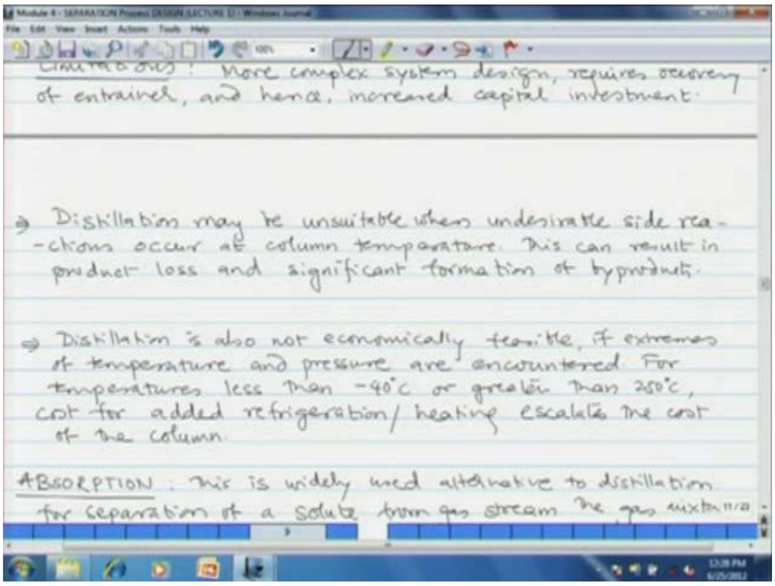

More complex system design, requires recovery of entrainer, and hence, increased capital investment.

⇒ Distillation may be unsuitable when undesirable side reactions occur at column temperature. This can result in product loss and significant formation of byproducts.

⇒ Distillation is also not economically feasible, if extremes of temperature and pressure are encountered. For temperatures less than -40°C or greater than 250°C, cost for added refrigeration/ heating escalates the cost of the column.

ABSORPTION: This is widely used alternative to distillation for separation of a solute from gas stream. The gas mixture

When undesirable side reactions occur at column temperature. We find defect in product loss and significant formation of the bi products. Then the operation of absorption, this is the another widely used operation in a chemical industry and it is also used as an alternative to distillation for separation of solute from gas stream. Because, mixture is contacted with liquid solvent which preferentially absorbs one or more components from gas stream the absorption factor which is l by mg. L is the molar flow rate of liquid, m is the slope of the equilibrium line m equilibrium curve and g is the molar flow rate of gas the absorption factor l by mg for relatively dilute components gas phase determines as how, readily that component is absorbed or when absorption factor is large absorptivity will be increased.

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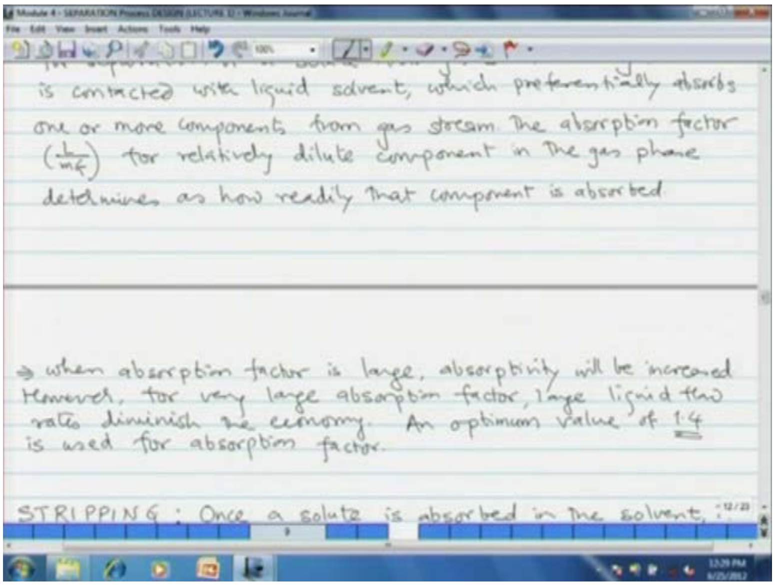

is contacted with liquid solvent, which preferentially absorbs one or more components from gas stream The absorption factor $\left(\frac{L}{mG}\right)$ for relatively dilute component in the gas phase determines as how readily that component is absorbed

⇒ when absorption factor is large, absorptivity will be increased However, for very large absorption factor, large liquid flow rates diminish the economy. An optimum value of 1.4 is used for absorption factor.

STRIPPING : Once a solute is absorbed in the solvent,

However, we will have very large amount of liquid which is rather dilute and then the recovery of the solute from that liquids become un economical. We have a separate module for absorption column design that is the economic decision making during design of an absorption column. So, I do not elaborate this thing further we have a separate module for about 4 to 5 lecture. So, I suggest that you watch that module very carefully to see as how economics and the engineering can be couples for design of a distribution column. The optimum value of absorption factor is 1.4. Now, how it is explained in the module of economic decision making of absorption column. So, I suggest that you watch that module to get answer to that question why 1.4 is the optimum value for absorption factor.

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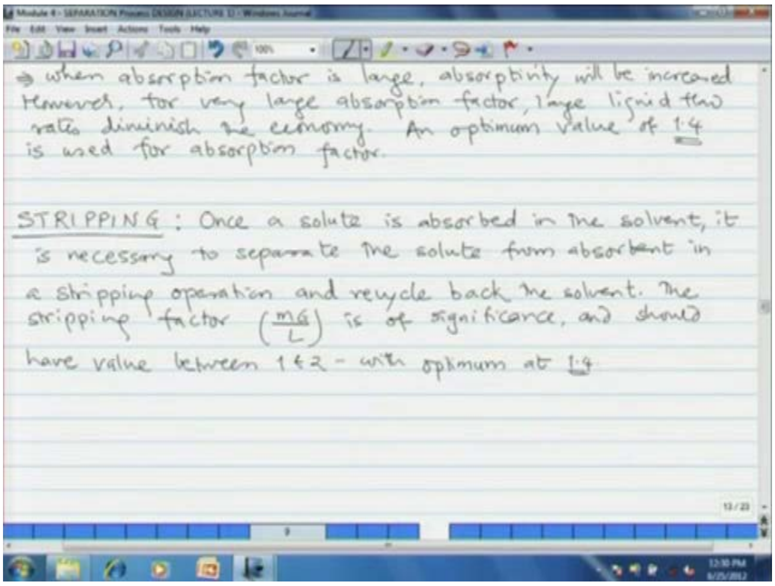

⇒ when absorption factor is large, absorptivity will be increased However, for very large absorption factor, large liquid flow rates diminish the economy. An optimum value of 1.4 is used for absorption factor.

STRIPPING : Once a solute is absorbed in the solvent, it is necessary to separate the solute from absorbent in a stripping operation and recycle back the solvent. The stripping factor $\left(\frac{mG}{L}\right)$ is of significance, and should have value between 1 & 2 – with optimum at 1.4

Then there is process of stripping, stripping is exactly opposite of absorption once the solute is absorbed in the solvent it is necessary to separate the solute from the absorbent in a stripping operation and recycle back the solvent the stripping factor is exactly opposite mg by l which is of significance and well, it should have value between 1 and 2 optimum again 1.4 for dilute mixtures. Stripping could be achieved even by steam that you pass superheated steam through the solve a through the solution that contains solute and solvent.

And thus the steam takes off the relatively volatile solutes then but in that case the solute has to be immiscible with the steam and you condense the whole vapors to get the solute the that forms two phases and then the liquid a the water part can be recycled for the generation of the steam further and the solute can be recovered, then the process of extraction.

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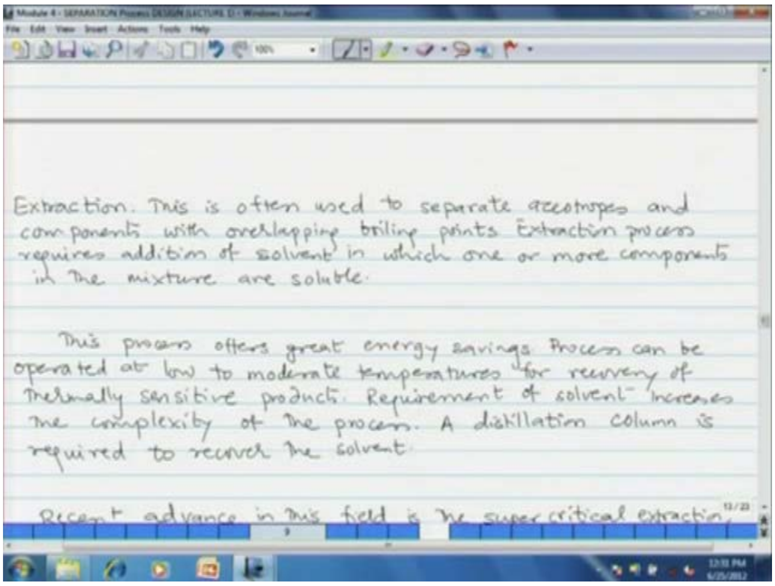

Extraction. This is often used to separate azeotropes and components with overlapping boiling points. Extraction process requires addition of solvent in which one or more components in the mixture are soluble.

This process offers great energy savings. Process can be operated at low to moderate temperatures for recovery of thermally sensitive products. Requirement of solvent increases the complexity of the process. A distillation column is required to recover the solvent.

Recent advance in this field is the super critical extraction

This is often used to separate aziotropes and components with overlapping boiling points, extraction process requires addition of solvent in which one or more components in the mixture are soluble. Again the principles of this operation you are already learning in mass transfer course. So, we will not deal with the basic principles of the process, now again here there is a tradeoff we have to add a solvent then there is there are two things, first is the distribution co-efficient and the selectivity.

You have let us say more than one solutes then you should have a higher partition co-efficient of the desired solute and it should be also your solvent should also be selected that it should pick up selectively one solute leaving apart the others. Now, this advantage of this process is that it offers great energy saving, process can be operated at low to moderate temperatures for recovery of thermally sensitive products then requirement of solvent increases the complexity of the process. You have to very carefully study the liquid -liquid properties it has to be heterogeneous.

That then the many diagrams that you have learnt in mass transfer course help you guide selection of a suitable sample. The recent advance in this field is that of super critical extraction using super critical fluids. Now, super critical fluids have greater solubility or selectivity. So, that they increase the extent of separation a that can be achieved. However, extraction operation itself will not give you the desired product it has to be coupled with a distillation operation.

The output the or extract that is from the a extraction product contain solute and solvent, which has to be separated and the most common operation is distillation. However, that a thus the subsidiary distillation operation if you have to make it as in cost intensive then you have to select a solvent which has a very high relative volatility with respect to the solute. So, that you can achieve separation in very small number of plates the solvent which you are going to should also have less boiling point and it should be less viscous and so on and so forth. So, well these things you have already learnt in mass transfer code what are the desired properties of the solvent for a particular extraction process.

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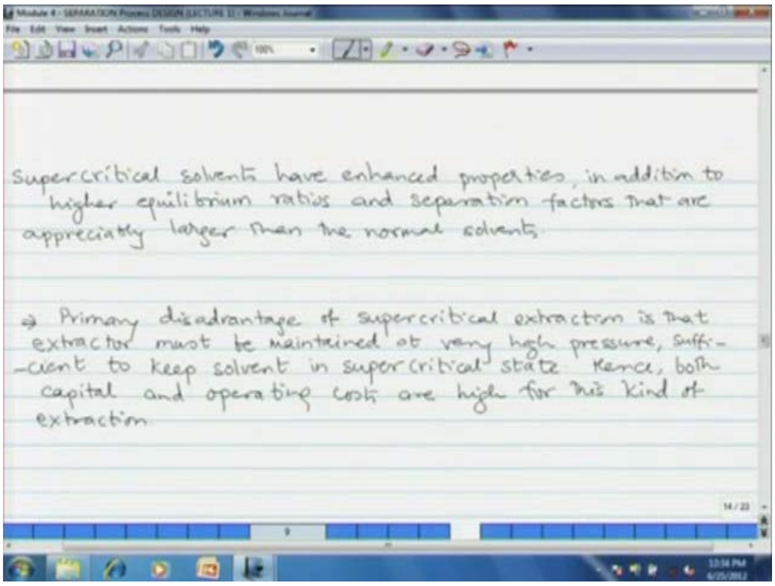

Supercritical solvents have enhanced properties, in addition to higher equilibrium ratios and separation factors that are appreciatly larger than the normal solvents

⇒ Primary disadvantage of supercritical extraction is that extractor must be maintained at very high pressure, sufficient to keep solvent in supercritical state Hence, both capital and operating costs are high for this kind of extraction

Primary dis-advantage of supercritical extraction however, is that the extractor must be maintained at very high pressure sufficient to keep the solvent in the super critical state and therefore, the capital and operating cost are high for this kind of extraction and this point has to be noted when we talk of advantage of super critical solvents.

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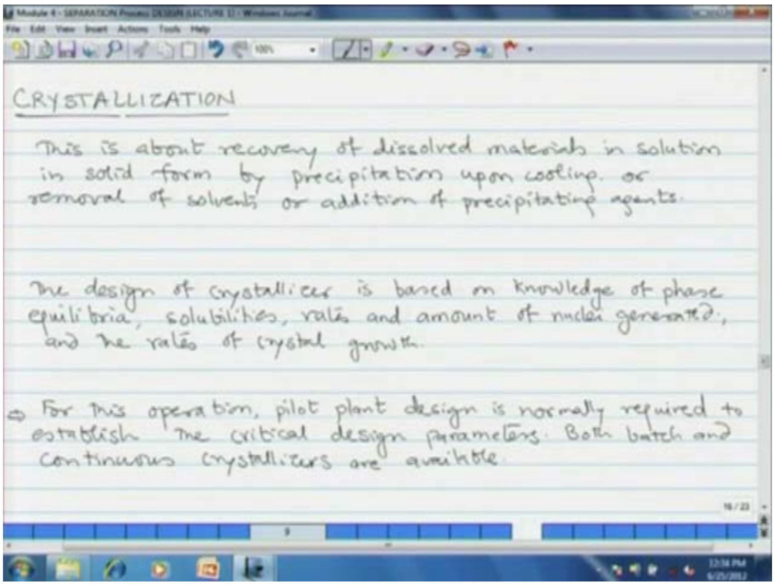

CRYSTALLIZATION

This is about recovery of dissolved materials in solution in solid form by precipitation upon cooling. or removal of solvents or addition of precipitating agents.

The design of crystallizer is based on knowledge of phase equilibria, solubilities, rates and amount of nuclei generated, and the rates of crystal growth.

⇨ For this operation, pilot plant design is normally required to establish the critical design parameters. Both batch and continuous crystallizers are available.

Then comes the operation of crystallization, this is about recovering of the dissolved material in the solution in solid form by precipitation of point cooling or removal of solvent or addition of precipitating agents. The most common operation the most common method of achieving cristilation is you have to dissolve the solids in a suitable solvent at high temperature because solubility usually increases with temperature. And then cool that solution, so that at lower temperatures the solution becomes super saturated and then the precipitation occurs. However, the pure component although impure solid solutions dissolve in the solvent and only the pure component precipitates out, and all the impurities are retained in the solution. The design of the crystaliser is based on the knowledge of phase equilibria solubility a rates and amount of nuclei that are generated and the rate of crystal growth.

You have already learnt the MSMPR model Mixed Suspension Mix Productery model in a topic of crystallization which will be covered in our mass transfer course. MSMPR model uses the delta law that it assumes that the crystal is growing equally from all sides then you have a trade off between delta g and then you get the predominant particle sizes three times the these retention time. So, all these things you have already learnt. But, now we shall see how crystallization is useful for treatment of very large amount of reactor effluent that come out.

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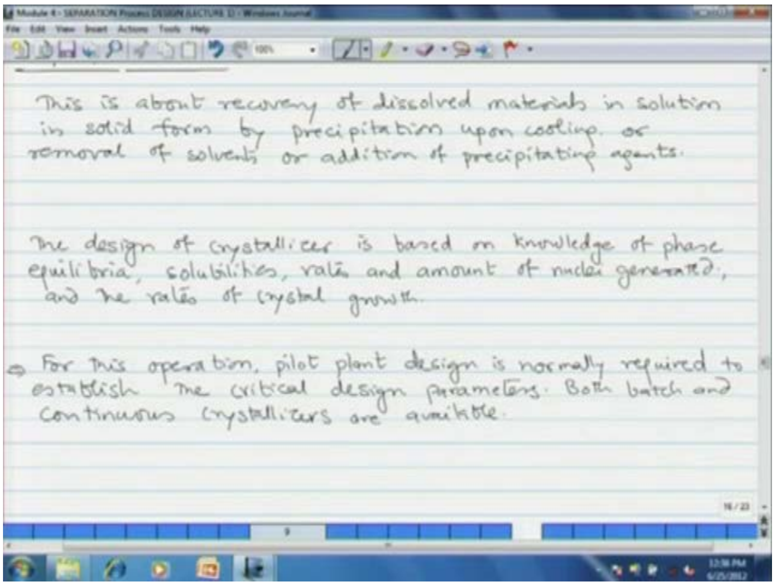

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The design of crystallizer is based on knowledge of phase equilibria, solubilities, rates and amount of nuclei generated, and the rates of crystal growth.

⇒ For this operation, pilot plant design is normally required to establish the critical design parameters. Both batch and continuous crystallizers are available.

For this operation pilot line design is normally required to establish the critical design parameters. Both batch and continuous crystallizers are available earlier before development of the adsorption process for separation of xylenes a crystallization was major operation for separation of xylenes. Like ortho meta para xylene they have very close value point therefore, separation of these xylenes through distillation is highly energy intensive and cost intensive.

However, if you see the solidification temperatures these were quite different and therefore, the xylenes were separated by crystallization and not by boiling. So, that you cool down the temperature at about minus 40 degrees the ortho xylene separates out and then meta and para further can be a separated by lowering of temperature. So, crystallization is a very common operation that is used in industry. Various types of crystals are available and; however, before you choose a particular crystallization for a particular operation islet plant, pilot plant testing is necessary.

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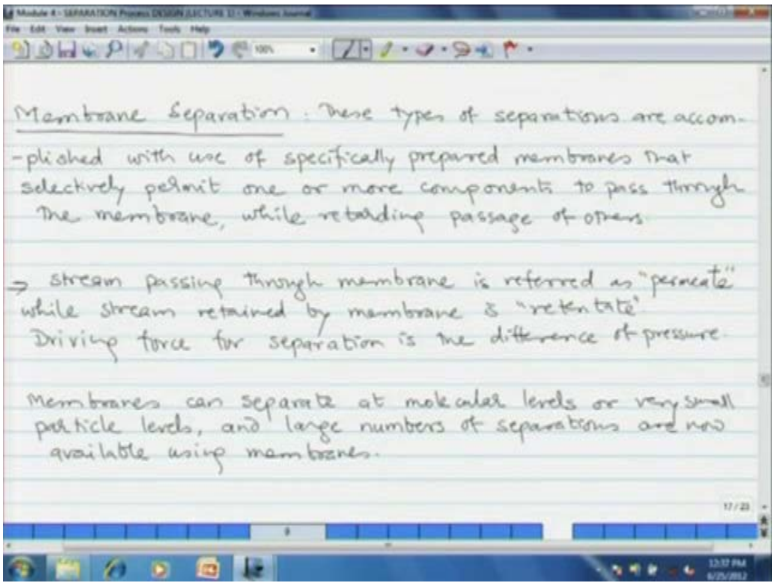

Membrane Separation : These types of separations are accom-
-plished with use of specifically prepared membranes that selectively permit one or more components to pass through the membrane, while retarding passage of others.

⇒ stream passing through membrane is referred as "permeate" while stream retained by membrane is "retentate"
Driving force for separation is the difference of pressure.

Membranes can separate at molecular levels or very small particle levels, and large numbers of separations are now available using membranes.

Then comes the topic of membrane separation which is one of the most popular modern day process for separations of different reaction mixture. As I already told you that membranes overcome a major drawback of conventional operations of absorption and distillation, in that they are very less energy intensive. They only have a barrier in the form of membrane and separation occurs at no high energy cost, except for pervaporation that we shall see later.

These types of separations are accompanied with use of specifically prepared membranes that selectively permit one or more components to pass through the membrane while retarding passage of others. A stream passing through the membrane is referred as permeate while the stream that is retained by the membrane is retentate. Driving force for separation is the difference of pressure, that is applied at the on the two ends or two sides of the membrane. Membrane can separate at molecular level or very small particle level and large numbers of separations are now available using membranes.

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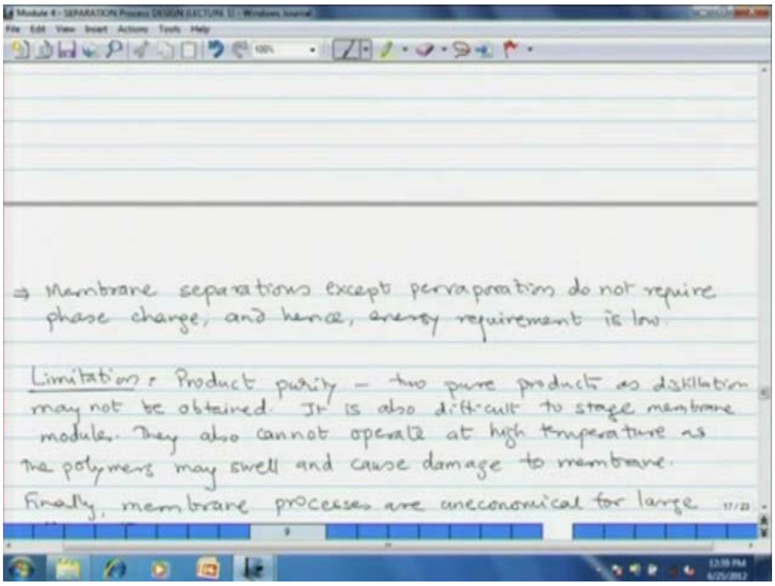

⇒ Membrane separations except pervaporation do not require phase change, and hence, energy requirement is low.

Limitation: Product purity – two pure products as distillation may not be obtained. It is also difficult to stage membrane module. They also cannot operate at high temperature as the polymers may swell and cause damage to membrane. Finally, membrane processes are uneconomical for large

Membrane separation except for pervaporation where a component dissolve at one side of now the membrane and that diffuses and then evaporates on the other side that is what pervaporation permeation evaporation. So, pervaporation that is only a slightly energy intensive operation. Because, the there is a phase change that is occurring across the membrane at one side the component is dissolving in liquid state and on the other side is evaporating. So, that the energy of evaporation is absorbed from the liquid that is on the other side. So, you have to continuously keep the liquid at a certain temperature. Except that operation the membrane separations are less energy intensive. Now, I will give you some common membranes a processes, and the size classification.

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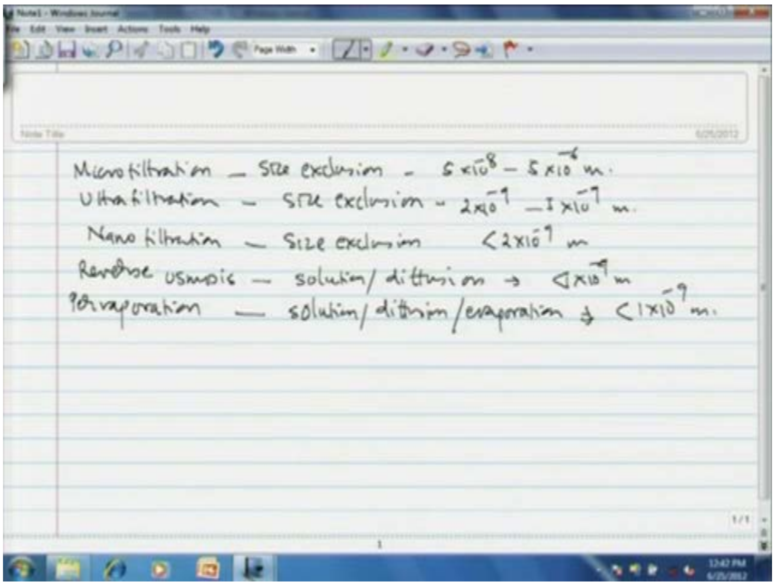

Microfiltration — size exclusion - $5\times10^{-8} - 5\times10^{-6}$ m.

Ultrafiltration — size exclusion - $2\times10^{-9} - 5\times10^{-9}$ m.

Nano filtration — size exclusion $<2\times10^{-9}$ m

Reverse osmosis — solution/ diffusion → $<1\times10^{-9}$ m

Pervaporation — solution/ diffusion/evaporation → $<1\times10^{-9}$ m.

For example, the micro filtration works on size exclusion the separating mechanism is size exclusion and the pore or inter molecular size is something like 2 into 10 to power 8 5 into 10 to power minus 8 or 2 5 into 10 to power minus 6 meters. Then ultra-filtration, this works again on size exclusion and here the pore size is even lesser 2 into 10 to power minus 9 to 5 into 10 to power minus 9 meters, then the Nano filtration this also works on size exclusion that is the pore size is more lesser than 2 into 10 to the power minus 9 meters then reverse osmosis.

Reverse osmosis is considered as a potential process for desalination of sea water. So, as to make it portable, here the mechanism is solution diffusion which means, one of the components selectively gets dissolved in the membrane at one end it diffuses to the other side and then desorbs. So, here the pore size is lesser than 1 into 10 to power minus 9 meter then there is pervaporation here there is solution on one side then diffusion on the other side and then evaporation we have to maintain lesser pressure at the permettant. So, that the components the a component evaporates and here also, you have the pore size less than 1 into 10 to power minus 9 meters. So, that is the typical size classification of the membranes.

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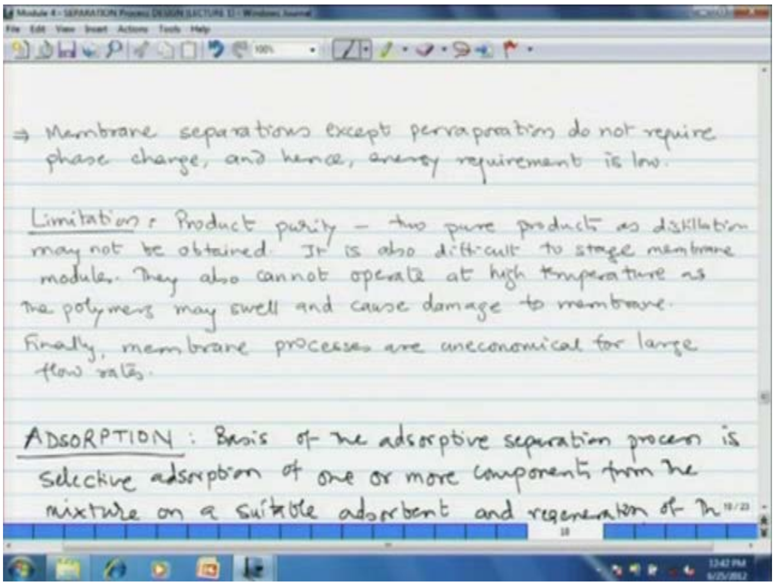

⇒ Membrane separations except pervaporation do not require phase change, and hence, energy requirement is low.

Limitation: Product purity – two pure products as distillation may not be obtained. It is also difficult to stage membrane module. They also cannot operate at high temperature as the polymers may swell and cause damage to membrane. Finally, membrane processes are uneconomical for large flow rates.

ADSORPTION: Basis of the adsorptive separation process is selective adsorption of one or more components from the mixture on a suitable adsorbent and regeneration of th

Although membranes have several positive points to their credit, there are also some limitations. For example, the product purity, two pure products as distillation may not be obtained distillation can distillation column can be designed to get 99 percent or 99.9 or 99.99 percent pure product. But, that kind of design may not be available with the membrane and unless and until it is extremely selective, that is the thing it is also difficult to stage membrane values. They cannot they also cannot operate at high temperature as the polymer may swell and cause damage to the membrane.

Then another point is that of the purity of the liquid that is been treated any solid impurity in that liquid can rupture the membrane and can hamper the complete operation. Membranes have also been used in many electro chemical processes for example, the caustic soda manufacture is now a days only using membrane cells which allow passage of certain ions only the cations, but not anions and they give very high electrical efficiency. But, again the brine that is suitable for our for treatment in case with membrane cells has to be very pure.

Like for example, any solid impurity has to be reduced to PPB level Parts Per Billion. And then the cost of this purification adds to the total operating cost although means it is a trade off you are saving money due to higher electrical efficiency but at the same time we have time we have, to go for a more stringent conditions for your feed quality and that a to meet that stringent condition you have to go for additional separation processes which could be expensive. So, that is all about the membranes, then comes the operation of adsorption it is as old if not as if not as old, but it is as conventional as absorption.

Now, here the basis of adsorptive separation adsorptive process is that, one or more components from the mixture adsorbed on the suitable adsorbent and the regeneration of the loaded adsorbent is possible through either thermal or pressure swing. Now, these are very widely used technique, the in separation of many products. For example, air splitting Pressure Swing Adsorption PSA is one of the very popular processes used in process industry. In pressure swing operation a sorry pressure swing adsorption you use zeolites with very, very controlled pore size.

And then air separation is achieved that you pass oxygen and nitrogen and then apply pressure and then the smaller molecules that diffuses inside the zeolite the pores of zeolite and then that gets adsorbed. And then the relatively a pure other compound nitrogen is left that is taken out, now you have an adsorbent which is loaded with adsorbate that is oxygen and then it is subjected to the pressure swing that is the total pressure is reduced, so as to desorb the adsorbed component.

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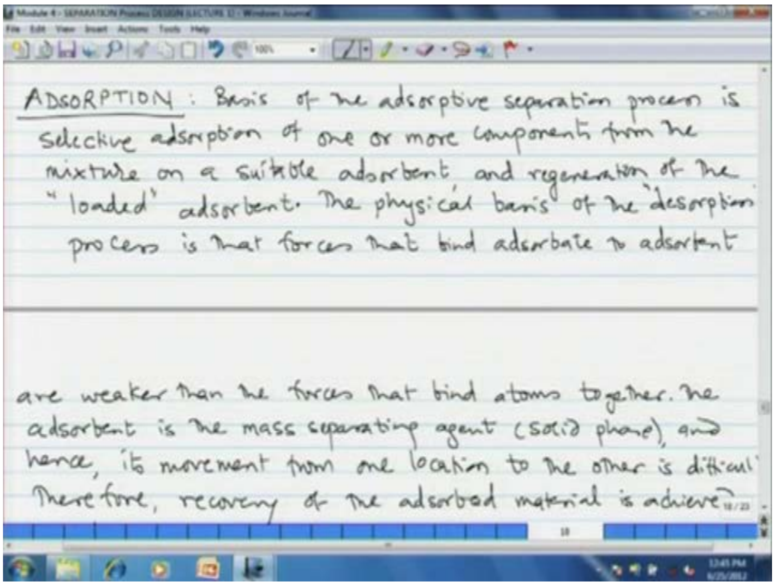

ADSORPTION: Basis of the adsorptive separation process is selective adsorption of one or more components from the mixture on a suitable adsorbent and regeneration of the "loaded" adsorbent. The physical basis of the "desorption" process is that forces that bind adsorbate to adsorbent

are weaker than the forces that bind atoms together. The adsorbent is the mass separating agent (solid phase), and hence, its movement from one location to the other is difficult. Therefore, recovery of the adsorbed material is achieved

Now, the physical basics for desorption process is that the forces that bind adsorbate to the adsorbent are weaker than the forces that bind atoms together. That is the very simple a principle, adsorption is very commonly used the for example, our domestic water purifiers use activated carbon, activated charcoal as adsorbent for separation of organic impurities. We in the north east we have lot of iron in the iron dissolved in water and that can also be separated using a suitable a adsorbent resin. So, adsorption is a very common operation which is used all over industry as well as in several domestic gadgets.

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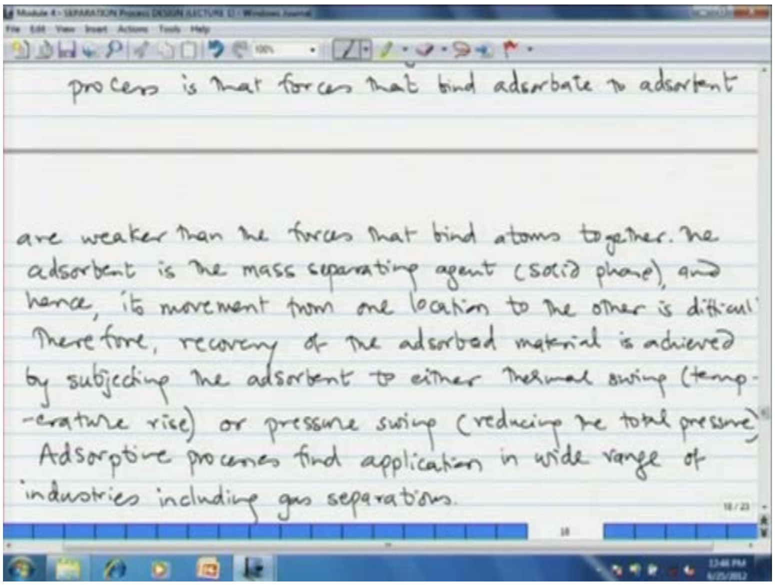

process is that forces that bind adsorbate to adsorbent

are weaker than the forces that bind atoms together. The adsorbent is the mass separating agent (solid phase), and hence, its movement from one location to the other is difficult. Therefore, recovery of the adsorbed material is achieved by subjecting the adsorbent to either thermal swing (temp-erature rise) or pressure swing (reducing the total pressure). Adsorptive processes find application in wide range of industries including gas separations.

The major problem with adsorption was that when you use thermal sea for example, adsorption is bond formation. So, it releases energy and therefore, if you have to desorb the adsorbed product then you have to heat it, so that the product gets desorbed. But, this thermal swing can cause hindering of the particles if you are using the same adsorbent by overall by overall over again, then successive operation can cause reduction in the adsorbent capacity. Secondly the adsorbent is the mass separating agent solid phase.

And hence its movement from one location to other is difficult. Therefore, recovery of the adsorbed material is achieved by subjecting the adsorbent to either thermal swing as I just mentioned or pressure swing in the adsorption at as a bed itself. So, you cannot separate it and then transport transportation is difficult, in some cases like when for separation of methane a methane and ethane from the hydrocarbon gases they use to have a convective loop like air convection for they used to have a counter current a operation with adsorbent passing from top to bottom. And gas hydrocarbon mixture passing from bottom to top and then the after the adsorbent comes down it is lifted back to the top of the tower in air convener air convening like pneumatic convening. So, that the sintering the attrition and sintering effect is lowered.

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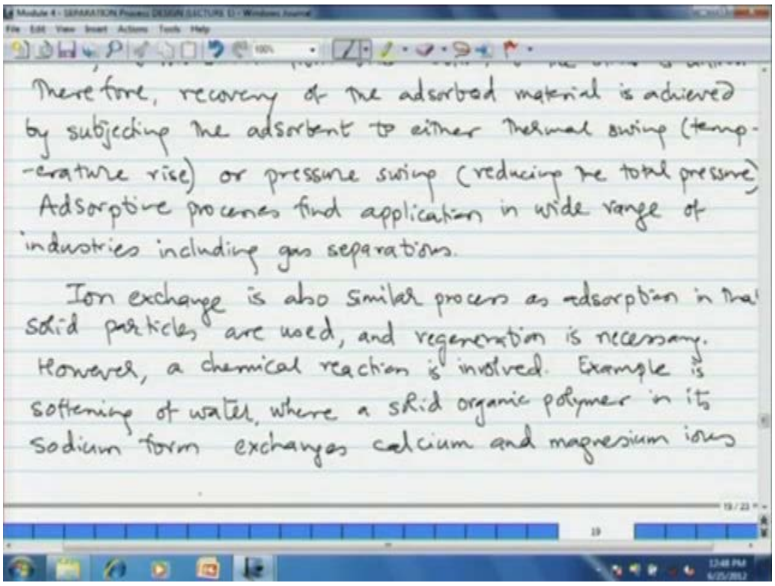
Therefore, recovery of the adsorbed material is achieved by subjecting the adsorbent to either thermal swing (temp-erature rise) or pressure swing (reducing the total pressure)
Adsorptive processes find application in wide range of industries including gas separations.

Ion exchange is also similar process as adsorption in tha solid particles are used, and regeneration is necessary. However, a chemical reaction is involved. Example is softening of water, where a solid organic polymer in its sodium form exchanges calcium and magnesium ions

Ion exchange is a cousin of a adsorption, ion exchange is also similar process as adsorption in that solid particles are used and regeneration is necessary. However, in this case a chemical reaction is involved example of, that is softening of water, when especially the boiler fed water, which is used for generation of steam, where in this case if solid organic polymer emits sodium or h form the hydrogen form, exchanges calcium and magnesium ions which are likely to precipitate which is called hardness of water from water.

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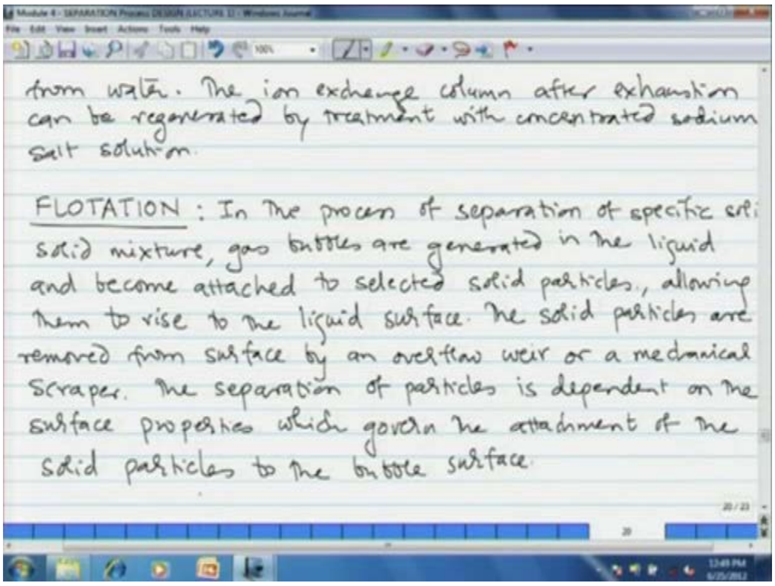
from water. The ion exchange column after exhaustion can be regenerated by treatment with concentrated sodium salt solution.

FLOTATION : In the process of separation of specific sol solid mixture, gas bubbles are generated in the liquid and become attached to selected solid particles, allowing them to rise to the liquid surface. The solid particles are removed from surface by an overflow weir or a mechanical scraper. The separation of particles is dependent on the surface properties which govern the attachment of the solid particles to the bubble surface.

The ion exchange column after exhaustion can be regenerated, but treatment with concentrated sodium salt solution. Now, these ion exchange resins are quite widely used for making boiler hood water the demineralized water as they call these ion exchange resins are also used for preparation of brine for caustic soda industry that I just mentioned. So, ion exchange is also a very common operation for separation of the reaction mixture. And then comes the a process of floatation I have already discussed this briefly before.

It is the basically a the process of separation of specific solid, solid mixture using gas bubbles. The gas bubbles are generated in the liquid and become attached to selected solid particles which are lifted to the top allowing them to rise to the liquid surface then the solid particles are removed from the surface by an overflow wire or a mechanical a scraper. The separation of particles is dependent on the surface properties which govern the attachment of solid particles to the bubble surface.

Now, many times coal is purified using this kind of technique the separation of coal particles from impurities can be achieved by what is known as the froth floatation. There are several factors that govern the efficacy of froth floatation that there are several a surfactants are added the PH has to be controlled very properly, so as to achieve as maximum separation as possible.

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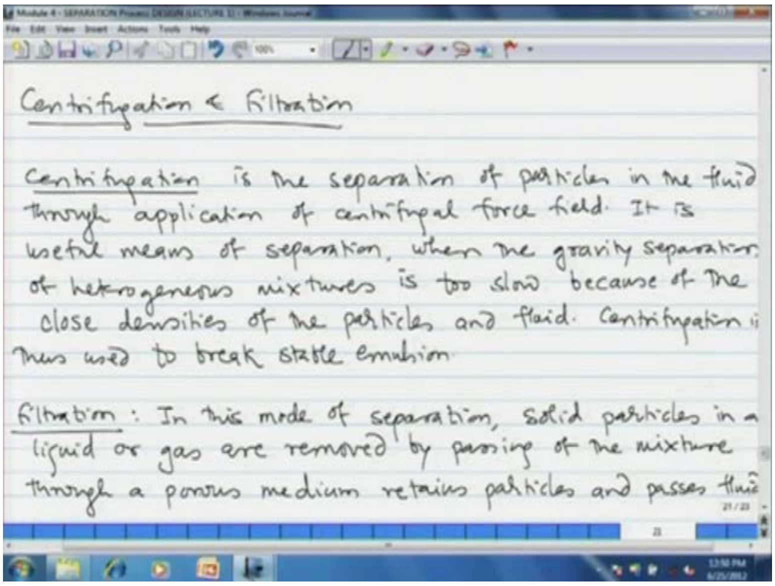

Centrifugation & Filtration

Centrifugation is the separation of particles in the fluid through application of centrifugal force field. It is useful means of separation, when the gravity separation of heterogeneous mixtures is too slow because of the close densities of the particles and fluid. Centrifugation is thus used to break stable emulsion.

Filtration : In this mode of separation, solid particles in a liquid or gas are removed by passing of the mixture through a porous medium retains particles and passes the

And then finally, we shall see the centrifugation and filtration. Centrifugation is for separation of either solid particles or it can also be used for separation of some heterogeneous liquid mixture. Centrifugation is the separation of particles in the fluid through application of centrifugal force field, the liquid is put in a certain vials and or containers and it is rotated at a very high RPM 5000 6000 RPM and then the particles get thrown how because of higher density. This is usually applied when the gravity separation of the heterogeneous mixture is too slow.

Because, of the close densities of the particles of the particles and the fluid. So, centrifugation is thus used to break stable emulsion, then the process of filtration. Filtration again it is a sort of membrane process, but it is not for as small particles as we use for a, membrane these are for like macroscopic particles. In this mode of separation the solid particles in liquid or gas are removed by passing of the mixture through a porous medium that retain solid particles and passes the fluid.

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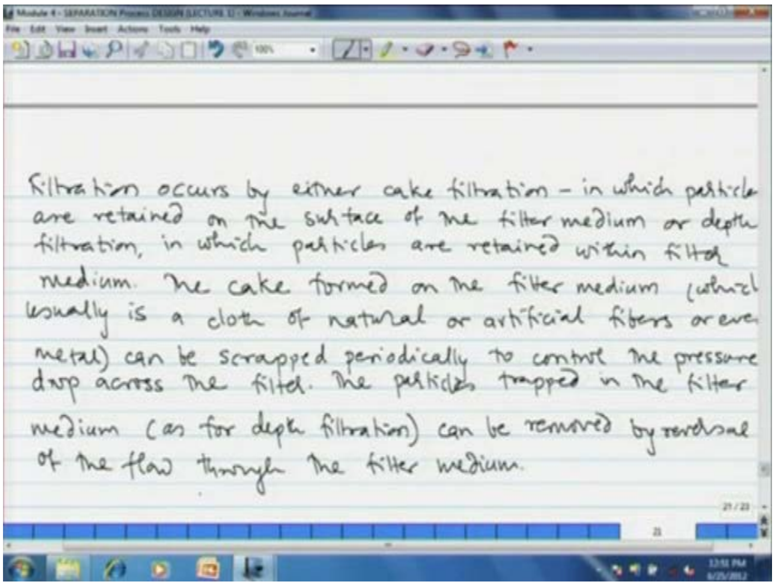

Filtration occurs by either cake filtration – in which particle are retained on the surface of the filter medium or depth filtration, in which particles are retained within filter medium. The cake formed on the filter medium (which usually is a cloth of natural or artificial fibers or even metal) can be scrapped periodically to control the pressure drop across the filter. The particles trapped in the filter medium (as for depth filtration) can be removed by reversal of the flow through the filter medium.

The filtration occurs by either cake filtration in which the particles are retained on the surface of the filter medium or depth filtration. In which the particles are retained within the filter medium, the cake formed on the filter medium as the filtration proceeds, it offers additional resistance to the fluid flow therefore, the pressure drop across the total filter it could be a stacked filter with stacked a pieces of the filter. It could be the filter medium could be a cloth of natural or artificial fiber or it could be even metal and these filter media are basically a enclosed in metal boundaries metal frames.

And then these are stacked one after the another to achieve as high separation as possible. But, as the filtration proceeds the formation of cake increases the pressure drop across he filter, so that has to be carefully monitored. And then as the cake formation increases the amount of the flow rate of the filtrate decreases. So, one has to carefully study that this particular portion will be covered in the course of unit operations the filtration as how you can obtain lots of civility volume collected versus time and there from that plot you can calculate the resistance of the cake it could be ether compressible or incompressible cake.

So, all these things the basic principles you are going to see in other NPTEL courses. Here we shall see only the economic aspects. So, when you are trying to separate these macroscopic particles you have to see that the cake formation does not exceed a certain thickness,. So, you have to continuously scrape the cake from the filtered surface or if it is depth filtration then once the pressure drop exceeds a certain limit then you have to reverse the flow, so that the particles that are trapped inside the filter medium can be removed.

So, these are essentially all the basic separation processes that are used in chemical industry. Depending on what kind of mixture we have reaction, mixture we have to go for selection of a process. Now, taking the same example of HDA process, that we have seen in the previous module hydro de alkylation. That process contains total 5 components the reactor effluent contains 5 components, first is the product benzene, the biproduct diphenyl toluene, unreacted toluene, hydrogen and methane.

Now, when the reactor effluent comes out it is in vapor phase,. So, all components are in vapor phase. So, we have to phase split that is the phase plate becomes the first separation operation, then how is phase split up obtained it is by expansion of the reaction mixture. However, before a the reactor effluent is at high temperature 1800 degrees Fahrenheit I am sorry 1300 hundred degrees Fahrenheit. And then it is at high pressure also about 500 PSI pressure. We first try to recover the heat of that particular stream through different exchangers.

That whole topic will be covered in the module of heat integration of the process and then we go for phase splitting, the high boiling components like benzene toluene diphenyl they condense hydrogen and methane are left out and then the separation of the benzene toluene and diphenyl is achieved through two distillation columns. And then for the gaseous mixture we have to go for purging,. So, that the methane concentration does not get built up beyond a certain limit and then part of the gas is recycled and a part is purged.

So, however; now on a days since these membrane modules are available for gas separation, you can install another separation process that is a gas separation process. So, that selectively methane is purged and hydrogen is recycled, if you use a general recycle purged stream then along with methane some hydrogen also get purged but by application of a membrane module at the pores stream you can selectively filter out hydrogen and recycle to the reactor. In the subsequent lectures we shall see how we can generally design a separation process.

Making a combination of all these unit operations that we have reviewed in this lecture and then we shall study mainly the distillation operation as I told you for absorption we have a separate module for economic decision making, and design of absorption columns. So, that topic is prepared in greater detail in that module other topics are already covered the basic principles in the mass transfer courses, so we shall focus mainly on the distillation operation in the subsequent lectures of this module.

We shall see how distillation column sequence is designed what are the guidelines, what are the quick methods of design of a distillation column, the number of plates, deciding the reflux ratio. So, that will be the content of the coming lectures. In the second lecture that is immediate next to this we shall see the general design of separation process, like what separation processes we have to use and where these should be located in the overall process. And there after the distillation operation will be studied in detail.