

Membrane Technology
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Lecture-33

Basic principle of MD, mechanism, process parameters, membranes, applications

Good morning students today's lecture 33 module 11 and in today's lecture will discuss about one of the interesting membrane separation process that is called membrane distillation. We will try to learn what is membrane distillation its principles the mechanism. What are the different process parameters that affect its performance then will see about various types of membrane that are being used for Membrane distillation the properties and two or three different applications.

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Thermally driven membrane process

- Most membrane transport processes are isothermal processes with either concentration, pressure or electrical potential difference as the driving force.
- When a membrane separates two phases held at different temperatures, heat will flow from the high-temperature side to the low-temperature side.
- This transport of heat can be expressed by a simple phenomenological equation, i.e. Fourier's law, where the heat flow is related to the corresponding driving force, the temperature difference.
- The heat flux is given by

$$J_h = -\lambda \frac{dT}{dx} \quad (1)$$

λ is the thermal conductivity or heat conductivity.

- Integration of eq. 1 across the membrane at steady-state flow gives,

$$J_h = \frac{\lambda}{l} (T_0 - T_l) \quad (2)$$



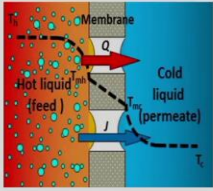
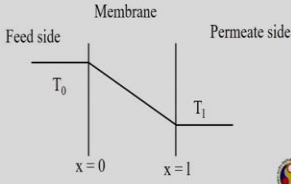
Now, you know its membrane distillation is a thermally driven membrane process. So most membrane transport processes are usually isothermal with other concentration pressure electrical potential difference is the driving force. That is what you have been discussing since our lecture started this course that when a membrane separates two phases held at different temperatures, heat will flow from the high temperature to the low temperature side.

This transport of heat is expressed by simple phenomenological equation that is nothing but for Fourier's law. Now for Fourier's law says that heat flow is related to the corresponding driving

pore and the temperature difference. So your λ is the thermal conductivity or heat conductivity to integrate this equation will get flux equals to $\lambda \Delta T / l$, so l being the thickness of the Membrane.

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- In addition to the heat flow, a mass flow also occurs, a process called thermo-osmosis or thermo-diffusion.
- No phase transitions occur in these processes.
- Another thermally driven membrane process is membrane distillation.
- Here, a porous membrane separates two liquids which do not wet it.
- If the liquids differ in temperature, the resulting vapour pressure difference causes vapour molecules to permeate from the high-temperature (high vapour pressure) side to the low-temperature (low vapour pressure) side.

The diagram illustrates the membrane distillation process. On the left, a 'Hot liquid (feed)' at temperature T_0 is in contact with a 'Membrane'. On the right, a 'Cold liquid (permeate)' at temperature T_1 is in contact with the membrane. Heat Q flows from the hot side to the cold side through the membrane. Mass J flows from the hot side to the cold side through the membrane. The temperature profile graph shows a linear decrease from T_0 at $x=0$ to T_1 at $x=l$.

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In addition to heat flow a mass flow also occurs the process called thermo-osmosis or thermal diffusion. Now phase transitions occur in this processes now and other important applications of this membrane separation process is membrane distillation. So what happen in membrane distillation is here porous membrane separates two liquids which do not wet it even the liquids different temperature the resulting vapour pressure difference causes vapour molecules to permeate from high temperature to low temperature that melts high vapour pressure side to the lower vapour pressure side is being seen in this particular schematics.

You can see here in the fields high temperature is T_0 and permeate side is T_1 . So here we can write a T_0 is greater than equals to T_1 .

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Membrane Distillation

- Membrane distillation is a process in which two liquids or solutions at different temperatures are separated by a porous membrane.
- The liquids or solutions must not wet the membrane otherwise the pores will be filled immediately as a result of capillary forces.
- This implies that non-wettable porous hydrophobic membranes must be used in the case of aqueous solutions.
- When the phases contain pure water and there is no temperature difference, the system is in equilibrium and no transport occurs.
- If the temperature of one of the two phases is higher than that of the other, a temperature difference exists across the membrane, resulting in a vapour pressure difference.

So, in this membrane distillation actually, the two liquids are solutions at different temperatures are separated by a porous membrane other liquids a solution must not wet the membrane otherwise the pores will be filled immediately as a result of capillary forces. It will affect the separation unit, then a pressure operated membrane separation process without that it cannot be achieved. So this also tells us that non wettable porous hydrophobic membranes must be used in the case of aqueous solution when I am dealing with aqueous solution preparation.

When the phases content pure water there is no temperature difference, the system is in equilibrium and no transport occurs. If the temperature of one of the two phases is higher than that of the other at temperature difference exist across the membrane resulting in a vapour pressure difference. Now that vapour pressure difference is eventually the difference in vapour pressure is eventually that the driving pore basically.

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- Vapour molecules will transport through the pores of the membrane from the high vapour pressure side to the low vapour pressure side.
- Transport occurs in a sequence of *three steps*:
 - ❑ Evaporation on the high-temperature side
 - ❑ Transport of vapour molecules through the pores of the hydrophobic porous membrane
 - ❑ Condensation on the low-temperature side

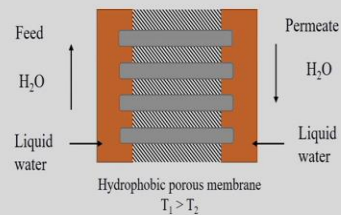


Figure. Schematic representation of membrane distillation

So the vapour molecules transport to the pores of the membrane from high vapour pressure to low pressure side. Usually the transport occurs in a sequence of three steps another thing we have to understand here the membrane is just separating the two phases. The membrane itself is not doing the separation. Ok. So you can say that is the barrier between two phases and the temperature difference between the two of phases which is being separated by physical by the membrane itself and the Separation is occurring.

Transport occurs in a sequence of three steps, first is evaporation on the high temperature side. Then transport of molecules to the pore of the hydrophobic porous membrane then third is condensation on the temperature side as it comes to the temperature side then condensation occurs. So, this is the schematic where T_1 is greater than T_2 so of course here and this is T_1 here and this is T_2 here.

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- Membrane distillation is one of the membrane processes in which the membrane is not directly involved in separation.
- The only function of the membrane is to *act as a barrier between the two phases*.
- *Selectivity* is completely determined by the *vapour-liquid equilibrium* involved. This means that the component with the highest partial pressure will show the highest permeation rate.
- In the case of an ethanol/water mixture where the membrane is not wetted at low ethanol concentrations, both components will be transported through the membrane but the permeation rate of ethanol will always be relatively higher.
- With salt solutions, for example NaCl in water, only water has a vapour pressure, i.e. the vapour pressure of NaCl can be neglected, which means that only water will permeate through the membrane and consequently very high selectivities are obtained.

So, membrane distillation is one of the process in which the membrane is not directly involved in the separation this is what I am just telling you. The only function of the membrane is to act as a barrier between the two phases. So, selectivity is completely determined by the vapour liquid equilibrium. This is very important just like distillation this means that the component with the highest partial pressure will show the highest permission rate.

So let us take an example of ethanol water mixture has the membrane is not wetted at low ethanol concentrations both components will be transported through the membrane that means both ethanol and water will be transported to the membrane because other membrane is not wetted at very low eternal concentrations. But the permeation rate of ethanol will always be relatively higher because of higher pressure ok or we can say her partial pressure.

So, with salt solution for another example, where the sodium chloride is dissolved in water only water has a vapour pressure, since water has a vapour pressure the vapour pressure of sodium chloride uses; present in very low amount can be neglected which means that only water will permeate through the membrane and consequently very high selectivity after. So, it actually depends upon what type of system you are dealing with and what is its application based on that you can obviously a design your membrane distillation and why about membrane distillation, all membrane separation process have been telling you are the courses that we can always Taylor make a membrane to suit a particular application.

So is the case of microfiltration ultrafiltration, reverse osmosis, nanofiltration anything and obviously membrane distillation also.

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- The transport of volatile components through the membrane can be described by phenomenological equations in which the flux is proportional to the driving force, i.e. the temperature difference across the membrane.
- The temperature difference results in a vapour pressure difference (temperature and vapour pressure are related according to the Antoine equation).
- The flux may be described by the phenomenological equation: $J = B \Delta P_i$
Here, flux is related to two parameters, the membrane-based parameter B and the system-based parameter ΔP .
- The proportionality factor B is determined by membrane parameters such as the material (hydrophobic/hydrophilic), pore structure, porosity and membrane thickness.



The transport of volatile components through the membrane can be described by phenomenological equations in which the flux is proportional to the driving force. That is the temperature difference across the member. The temperature difference results in a vapour pressure different that is temperature and pressure related Antoine equation this case we discussed earlier. I am not repeating the equations again. So the flux may be described by phenomenological equation J equals to $B \Delta P_i$.

A flux is related to two parameters. The first one is B , which is the membrane based parameter the second is ΔP which is a system based parameters the pressure difference of the driving force. Proportionality factor B is determined by the membrane parameters such as material. What is the membrane material whether it is hydrophobic and hydrophilic the structure of the pore, porosity in the thickness of the membrane.

As usual for other membrane separation this also holds good for membrane distillation. The thickness of the membrane should be as thin as possible.

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- The main structural parameters are the porosity, which must be as high as possible and the membrane thickness.
- The pore size distribution must be narrow, particularly on the larger pore side because the largest pores will be wetted first.
- In contrast, the system-based parameter ΔP is mainly determined by the temperature difference ΔT .
- Other parameters of interest are the hydrodynamic conditions (flow velocity) and module design, because they determine the effect of temperature polarisation and hence influence the driving force.

The main structural parameters are the porosity which must be as high as possible and the membrane thickness and the pores size distribution must be zero particularly on the larger pore side, which is the largest pore will be better first. This wetting of pore and all hydrophobic we discussed when we are discussing this one bubble point method to character is micro filtration membrane. I hope you recall those in contrast the system based parameter Delta P is mainly determined by the temperature difference that delta T.

Other parameters of interest of the hydrodynamic conditions that flow velocity even the module design also because that determine the effect of the temperature polarization and hence influence the driving force you talk about module design in which are basically the Length and width of the module and how many; if you are using hollow fibre systems, all these things also plays important role.

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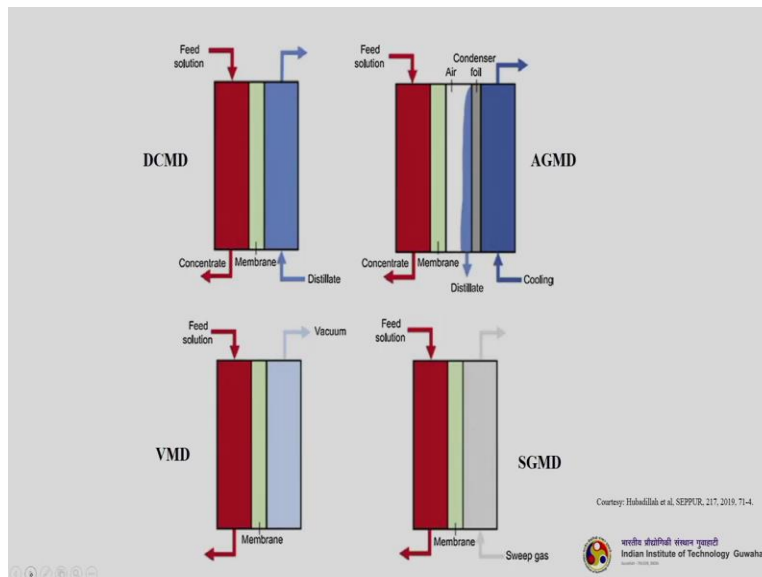
Membrane Distillation Configurations

MD processes can be classified into four basic configurations.

1. Direct contact membrane distillation (DCMD)
2. Vacuum membrane distillation (VMD)
3. Air gap membrane distillation (AGMD)
4. Sweep gas membrane distillation (SGMD)

So there are 4 different types of membrane distillation configuration. The first one is called direct contact membrane distillation DCMD in this system at the feed phase is in direct contact with one of two side of the membrane. Vacuum membrane distillation, then air gap membrane distillation and sweep gas membrane distillation. So there are basically 4 designs are four ways in which we can design our membrane distillation unit, depending upon the application requirements.

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So, let us see the schematic diagram ok. See the first one the DCMD the direct contact membrane distillation. The feed solution is in contact directly with the contact with the membrane here. And your passing distillate here and you are getting here permeate, whatever it

is here, whatever is getting separated will be returned on the surface of the membrane. Ok due to the large size of the molecules are smaller particles obviously pass through.

The second is we can call it BMD. So it is vacuum membrane distillation, so here again you see it is something like pervaporation, you know in which pervaporation membrane system we will try to recall the permeate side we used to make the low pressure by either using sweep gas or using a vacuum. Ok. So here it is using a vacuum system. Ok to maintain the low pressure present here. So what are you doing is trying to maintain ok as low as possible partial pressure.

This is another method. Third one is something little different which we have never discussed is air gap membrane distillation. So here there is a air gap between 1 phase of the one side of the of the membrane ok. So basically you can see in this particular figure the downstream side there is a air gap then the cooling or condenser side is coming into picture actually, basically. So, this is this particular area is the in which it contains air basically.

And the last one is sweep gas membrane distillation where again it is similar to this BMD. OK where we are trying to have the low vapour pressure using a sweep gas. So here we maintain the low pressure here. So the rate of transport of the components those were having her personal pressure will eventually pass through the permeate side. Ok, so you can say the faster, with faster rate whose partial pressure is high and the upstream side.

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Direct contact membrane distillation (DCMD)

Merits

- ❑ The easiest and simplest configuration to realize practically
- ❑ Flux is more stable than VMD for the feeds with fouling tendency
- ❑ High gained output ratio
- ❑ It might be the most appropriate configuration for removal of volatile components

Demerits

- ❑ Flux obtained is relatively lower than vacuum configurations under the identical operating conditions
- ❑ Thermal polarization is highest among all the configurations
- ❑ Flux is relatively more sensitive to feed concentration
- ❑ The permeate quality is sensitive to membrane wetting, suitable mainly for aqueous solutions

So these are the 4 different types of configuration let us see and try to understand the different merits and demerits of this 4 different configuration. So if you look at the DCM the direct contact membrane distillation here this is one of the easiest one and the simplest configuration to make it practically. Here in this case flux is more stable than BMD id you compare with the vacuum distillation membrane distillation.

So for the feed with following tendency the feeds the aqueous stream if they are containing some metals which are very highly susceptible to fouling then in this case if you use the DCMD flux will be more stable than the vacuum system. So gained output ratio it may be the most appropriate configuration for the removal of volatile component. Apart from that the demerits are flux obtained is suitably lower and vacuum configuration under identical operating conditions and being used.

Thermal polarization is highest among all the configuration, flux is relatively more sensitive to feed concentration. This is also very important this and the fouling tendency this merit and demerits they actually are the two parts of the same coin, so the permeate quality sensitive to membrane wetting suitably mainly for the aqueous equation.

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Vacuum membrane distillation (VMD)

Merits

- ☐ High flux
- ☐ It can be used for recovery of aroma compounds and related substances
- ☐ The permeate quality is stable despite of some wetting
- ☐ No possibility of wetting from distillate side
- ☐ Thermal polarization is very low

Demerits

- ☐ Higher probability of pore wetting
- ☐ Higher fouling
- ☐ Minimum selectivity of volatile components
- ☐ Require vacuum pump and external condenser



Next is vacuum membrane distillation BMD. So the merits is that it updates very high flux. It can be used for recovery of aroma compounds and related substances. The permeate quality is stable despite of some wetting no possibility of wetting from distillate side and thermal polarization is very, very low. So it is as demerits also that had probability of pore wetting. That is the reason is that since you are maintaining vacuum that will try to push the liquid through the pores.

So probability of pore wetting becomes more compared to other systems or other configurations fouling will be high again due to the vacuum system. Minimal selectivity of volatile components and they require vacuum pump and external condenser little more energy requirement is there here and little more cost also.

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Air gap membrane distillation (AGMD)

Merits

- ☐ Relatively high flux
- ☐ Low thermal losses
- ☐ No wetting on permeate side
- ☐ Less fouling tendency

Demerits

- ☐ Air gap provides an additional resistance to vapors
- ☐ Difficult module designing
- ☐ Difficult to model due to the involvement of too many variables
- ☐ Lowest gained output ratio

So, the air gap membrane distillation is a very different kind of system in which as I just showed you that air is there is just between after the membrane to the downstream side of the permeate side. So, it maintains very high relative high flux compared to other systems and low thermal losses no wetting on the permeate side. Since I am not drawing forcefully the permeate and less fouling tendency.

So demerits, is that air gap provides and additional resistance to vapors that is always there but see depending upon what type of vapour it is what components you are transferring depending on that we can say whether the air gap is providing a proper resistance or not. However there is a resistance and the function of resistance or the magnitude of resistance may vary depending upon the components. So, difficult module design designing and difficult to model due to an involvement too many variables and lowest gained output ratio.

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Sweep gas membrane distillation (SGMD)

Merits

- ☐ Thermal polarization is lower
- ☐ No wetting from permeate side
- ☐ Permeate quality independent of membrane wetting

Demerits

- ☐ Additional complexity due to the extra equipment involved
- ☐ Heat recovery is difficult
- ☐ Low flux
- ☐ Pretreatment of sweep gas might be needed

The next one is the sweep gas membrane distillation that is SGMD. So here the thermal polarization is lower. So, no wetting from permeate side also. So what happened permeate quality independent of membrane wetting and demerits are additional complexity due to extra equipment involved because anyway you have to pass through gas so you have to away sweep gas to tank in which is the sweep gases is being;

Or we can say the tank which contains the sweep gas then second thing is that you need of pump to pump it from the sweet gas to the membrane module. So, heat recovery is difficult here in this case. Fluxes lower compared to other system and pretreatment of sweep gas is needed depending upon what type of sweep gas you are using and is there any reaction that will going to happen if you; what the sweep gas comes in contact with the components that is getting transferred to the permeate side.

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Process parameters

- Membrane distillation is based on the concept that distillation takes place across a porous membrane.
- The main requirement is that the membrane must not be wetted.
- If wetting occurs, the liquid will penetrate spontaneously into the pores of the membrane.
- The wettability is determined by the interaction between the liquid and the polymeric material, with no wetting occurring at low affinity.
- Information about wettability can be obtained by contact angle (θ) measurements, i.e. a drop of liquid is placed upon a nonporous flat (and smooth) surface and the contact angle is measured.
- For low affinity the θ will have a value greater than 90° , whereas with high affinity the value of θ will be less than 90° .
- In the latter case the liquid will wet the surface.



Let us discuss the different types of process parameters which are affecting the membrane distillation system or efficiency we can say. So, MD is based on the concept that distillation takes place across the pores of the membrane. The main requirement is that the membrane must not be wetted. So that is most important point you can say if wetting occurs the liquid penetrates spontaneously into the pores of the membrane that is determined by the interaction between the liquid and the polymeric material with no wetting occurring at low affinity.

Information about wettability can be obtained by contact angle measurement that is a drop of liquid is placed on a non porous flat and smooth surface. The contact angle is measured. So you just measure the contact angle ok, so what is the theta right? So for the low affinity the greater value greater than 90 degree for high affinity the value of theta with less than 90 degree, so, in the latter case the liquid wets the surface.

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Process parameters

- If the material is porous, the liquid will penetrate into the pores when wetting occurs ($0 < 90^\circ$).

This can be described by the Laplace equation:

$$\Delta P = - \frac{2\gamma_1}{r} \cos \theta$$

- If $\theta > 90$ then $\cos \theta < 0$ and $\Delta p > 0$, and only if a finite pressure is applied (according to the Laplace equation) the liquid will penetrate into the membrane.

- The wettability depends on three factors:

- ☐ Pore size (r)
- ☐ Surface tension of the liquid (γ_1)
- ☐ Surface energy of the membrane material (or $\cos \theta$)



So, the material is pores are the liquid in penetrate and into the pores wetting occurs that is theta is less than 90 degree this can be described by the Laplace Equation. This equation we discussed during this one. May be discussed about the characterization of microfiltration membranes using bubble point method or apparatus so $\Delta p = \frac{2\gamma_1}{r} \cos \theta$ since theta is greater than 90 degree. Ok 90 degree then $\cos \theta$ is less than 0 and ΔP greater than 0 and only if a final pressure is applied according to the Laplace Equation the liquid penetrate into the membrane.

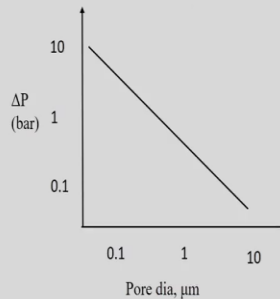
So, wettability depends upon another two or three parameters. The first one is pore size second is surface tension of the liquid and third is surface energy of the membrane material or we can say the $\cos \theta$ in other words you can say $\cos \theta$ directly.

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Process parameters

Pore Size

- The wetting pressure is inversely proportional to the membrane pore size.
- The pressure needed to wet a porous Teflon membrane with water as a function of the pore size.



So, the wetting pressure is inversely proportional to the membrane pore size. The pressure needed to wet a porous Teflon membrane with water is a function of pore size is being plotted here. You can see if you increase the pore diameter here ok. Yeah, Delta P that is coming down the pressure is coming down. Ok. So higher the pore size lesser the pressure that is required.

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Process parameters

Surface Tension

- The second parameter that determines the wettability is the surface tension of the liquid.
- This is related to intermolecular forces such as dispersion forces, polar forces and hydrogen bonding.
- In a hydrocarbon such as hexane, only weak dispersion forces act and consequently the surface tension is low.
- On the other hand, in cases where hydrogen bonding occurs such as in water, the intermolecular forces are very strong and as a result the surface tension is high.
- When a liquid is brought into contact with a (smooth) polymeric surface, various contact angles between the liquid and the polymer are observed depending on the affinity between the liquid and the polymer.

So, surface tension is another parameter. So the second parameter that determines wettability is the surface tension of the liquid. This is related to intermolecular forces such as dispersion forces, polar forces and hydrogen bonding. Anyway this also has been discussed in one of our classes in hydrocarbons such as hexane let us understand what is happening here. So, only weak dispersion

forces act and consequently the surface tension is too low, on the other hand in cases for hydrogen bonding happens such as in water. The intermolecular forces are very strong and as a result the surface tension is high.


So, when a liquid is brought into contact with smooth polymeric surface where is contact angles between the liquid and the polymer observed depending on the affinity between the liquid and the polymers.

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Table. Surface Tension of some liquids at 20 °C

Liquids	Surface Tension (γ_l) (10^3 N/m)
Water	72.8
Methanol	22.6
Ethanol	22.8
Glycerol	63.4
Formamide	58.2
n-hexane	18.4

- If the contact angle is greater than 90° the liquid does not wet the surface.
- When the contact angle is smaller than 90° the liquid wets the surface.
- When $\theta = 0$ the liquid spreads out over the surface.

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This table gives you the values of surface tension of some liquids at 20 degree centigrade. You can see it is water it is 72.8 and 10 power of 3 Newton per metre and methanol is 22.6, Ethanol is more or less same as methanol 22.8 Glycerol is close to that of water it is 63.4, Formamide is 58.2 and n-hexane other very important class of solvent, it is 18.4. If the contact angle is greater than 90 degree the liquid does not wet the surface this we already discussed so when it is smaller than 90 degree liquid wet surface. So and contact angle equals to zero the liquid spreads out over the surface.

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- Wetting is favoured when the solid polymer has a high surface energy.
- To avoid wetting the maximum pore size must be small, the surface tension of the liquid high (for example, water) and the surface energy of the membrane material low such as with polypropylene (PP), polyethylene (PE) polytetrafluoroethylene (PTFE) and poly(vinylidene fluoride) (PVDF).

Table. Surface energies of some polymers

Polymer	Surface Energy (γ_s) (10^3 N/m)
Polytetrafluoroethylene	19.1
Polytrifluoroethylene	23.9
Polyvinylidene fluoride	30.3
PVC	36.7
PE	33.2
PP	30
PS	42

So, wetting is favourite when the solid polymer has a higher surface energy to avoid wetting the maximum pore size must be small the surface tension of the liquid should be high for example water the surface energy of the membrane material should be low such as with polypropylene, polythene, polytetrafluoroethylene and poly vinylidene flouride PVDF. So, this is table again summarizes surface energy of some of the polymer.

So you can see for PTA is 19.1, polytri fluoroethane it is 23.9, PVDF it is 30, PVC 36.7 and so polyprolene is 42. So all this data are given in membrane handbook, as well as you can see from other hand books also.

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Membranes for MD

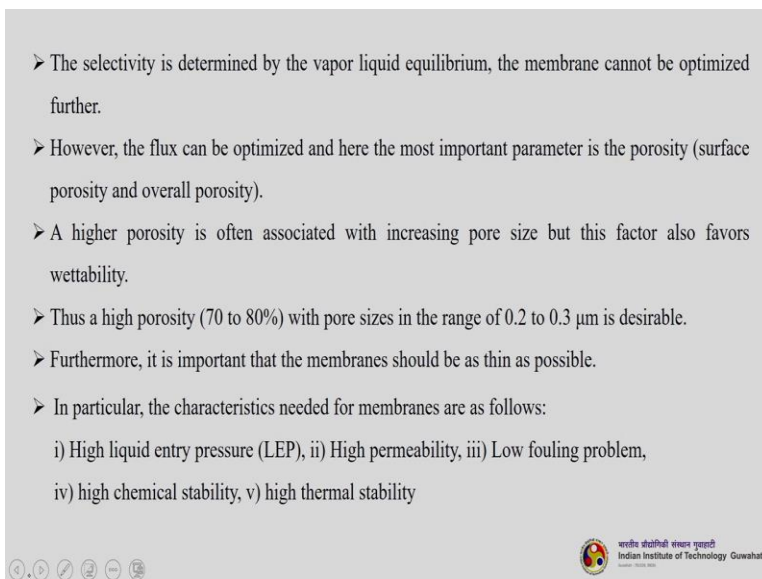
- One of the most crucial aspects of the membrane distillation is to have membranes with well controlled properties.
- The successful outcome of the process is reasonably expected to be depending upon the capability of the membrane to interface two media without dispersing one phase into another and to combine high volumetric mass transfer with high resistance to liquid intrusion in the pores.
- The membranes for membrane contactor application have to be porous, hydrophobic, with good thermal stability and excellent chemical resistance to feed solutions.
- To avoid wetting, the surface energy of the polymer must be as low as possible.
- This means that very hydrophobic materials such as polytetrafluoroethylene, poly(vinylidene fluoride), polyethylene or polypropylene must be used in combination with liquids with high surface tension such as water.

So, let us understand the membranes that are being used and what are the properties that ideal membrane distillation membrane should have? So, one the most crucial aspect of the membrane distillation is to have membrane with well control properties. So, for the successful outcome of the process is reasonable expected to be depending upon the capability of the membrane to interface to media without dispersing one phase to another and two combined high volumetric mass transport ok with high resistance to liquid intrusion into the pores.

So, the membranes for membrane contactor application have to porous, hydrophobic with good thermal stability and excellent chemical resistance to feed solution. But see most of these parameters are the properties are common for all other membrane separation applications tools. To avoid wetting the surface energy of the polymer must be as low as possible. This means that very hydrophobic materials, such as PDAP, PDF, PE or PP must be used in combination with liquids with high surface tension such as water.

So, good hydrophobic membrane and water is the solvent is fine for carrying out membrane distillation process.

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- The selectivity is determined by the vapor liquid equilibrium, the membrane cannot be optimized further.
- However, the flux can be optimized and here the most important parameter is the porosity (surface porosity and overall porosity).
- A higher porosity is often associated with increasing pore size but this factor also favors wettability.
- Thus a high porosity (70 to 80%) with pore sizes in the range of 0.2 to 0.3 μm is desirable.
- Furthermore, it is important that the membranes should be as thin as possible.
- In particular, the characteristics needed for membranes are as follows:
 - i) High liquid entry pressure (LEP), ii) High permeability, iii) Low fouling problem,
 - iv) high chemical stability, v) high thermal stability

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The selectivity is determined by the vapour liquid equilibrium. The membrane cannot be optimised further however the flux can be optimised and here the most important parameter is porosity that is surface porosity and overall porosity. Higher is often associated with increasing

forces, but this factor also favours wettability. So, there is a high porosity 70 to 80% with pore size in the range 0.2 to 0.3 micron is usually desirable. It is further more important that membrane should be as thin as possible, this size this I already told you the beginning of the class and this pores again, I am telling this holds good for all the membrane separation processes also.

So, in particular the characteristic needed for membranous are as follows. High liquid entry pressure which is called as LEP then high permeability, low fouling problem or low fouling tendencies, high chemical stability as well as high thermal stability.

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i) High liquid entry pressure (LEP)

- High liquid entry pressure (LEP), is the minimum hydrostatic pressure that must be applied onto the feed solution before it overcomes the hydrophobic forces of the membrane and penetrates into the membrane pores.
- LEP is a characteristic of each membrane and permits to prevent wetting of the membrane pores.
- High LEP may be achieved using a membrane material with high hydrophobicity and a small maximum pore size.

$$LEP_w = \frac{B\gamma_L \cos \theta}{d_{max}}$$

Here B is a geometric factor determined by pore structure with value equal to 1 for cylindrical pores, γ_L the liquid surface tension and θ is the liquid/solid contact angle.

- However, as the maximum pore size decreases, the mean pore size of the membrane decreases and the permeability of the membrane becomes low.



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So let us discuss one by one so high liquid entry pressure, so high liquid entry pressure is the minimum hydrostatic pressure that must be applied on to the feed solution before it overcomes the hydrophobic forces of the membrane and penetrates in to the membrane pores. So we can say that the minimum pressure that it should have to overcome the hydrophobic process of the membrane. So that a liquid or aqueous media can penetrate into the pore.

So, LEP is a characteristic of each membrane and permits to provide wetting of the membrane pores. High LEP may be achieved using a membrane material hydrophobicity and a small maximum pore size. So, this equation gives LEP how do you calculate LEP so $B \gamma_L \cos \theta$ divided by d_{max} B is the geometric factor determined by the pore structure with value

equal to 1 for cylindrical pore and γ_L is the liquid surface tension θ is the liquid solid contact angle.

So however d_{max} is the maximum pore size so however is the maximum pore size decrease is the mean pore size of the membrane decreases and the permeability of the membrane also becomes very low.

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ii) *High permeability*

- The flux will “increase” with an increase in the membrane pore size and porosity, and with a decrease of the membrane thickness and pore tortuosity.
- In fact, molar flux through a pore is related to the membrane's average pore size and other characteristic parameters by:

$$N \propto \frac{(r^\alpha) \epsilon}{\tau \delta}$$

where ϵ is the membrane porosity, τ is the membrane tortuosity, δ is the membrane thickness, $\langle r^\alpha \rangle$ is the average pore size for Knudsen diffusion (when $\alpha=1$), and $\langle r^2 \rangle$ is the average squared pore size for viscous flux (when $\alpha=2$).

- In other words, to obtain a high permeability, the surface layer that governs the membrane transport must be as thin as possible and its surface porosity as well as pore size must be as large as possible.



The next one is high permeability, flux will increase with an increase in the membrane pore size and porosity and the decrease of the membrane thickness and pore tortuosity. In fact molar flux, through a pore is related to the membrane average pore size. Another characteristic parameters by this equations N proportional to r^α then ϵ divided by τ into δ where ϵ is the membrane porosity, τ is the membrane tortuosity.

Ok the δ is the membrane thickness, r^α is a average pore size for the Knudsen diffusion when α equals to 1 and r^α is average pore size for viscous flow where α equals to 2. In other words to obtain high permeability the surface layer that governs the membrane transport must be as thin as possible and its surface porosity as well as pore size must be as large as possible, as thin membrane as possible ok and the surface porosity as well as pore size must be as largest possible.

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ii) *Low fouling problem*

- Fouling is one of the major problems in the application of porous membranes.
- Fortunately, in the gas–liquid contactor applications, the contactors are less sensitive to fouling since there is no convection flow through the membrane pores.
- However, in industrial applications, gas and liquid streams with large content of suspended particles can cause plugging due to the small hollow fiber diameter.
- Pre-filtration is necessary in such a case.

So, the next one is low fouling problem, so fouling is one of the major problems in the application of porous membrane fortunately in the gas liquid contactor applications the contactors are less sensitive to fouling since there is no convective flow. There is no convective flow through the membrane pores. So however in industrial applications gas and liquid streams with large content of suspended particles can cause blocking due to small hollow fibre diameter.

In case if you are using the hollow fibre then fouling maybe a problem, so pre-filtration is necessary in such cases.

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iv) *High chemical stability*


- The chemical stability of the membrane material has a significant effect on its long-term stability.
- Any reaction between the solvent and membrane material could possibly affect the membrane matrix and surface structure.
- Liquid with high load of acid gases are corrosive in the nature, which make the membrane material less resistance to chemical attack.

Next is high chemical stability, the chemical stability of the membrane material is significant impact on its long term stability. Any reaction between the solvent and the membrane material could possibly affect the membrane Matrix and surface structure. So this is very important. So it should be chemically inert, so liquid with high load of acid gases are corrosive in nature which make the membrane material less resistance chemical attack.

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v) *High thermal stability*

- Under high temperatures, the membrane material may not be able to resist to degradation or decomposition.
- Changing in the nature of membrane depends on the glass transition temperature T_g for amorphous polymers or the melting point T_m for crystalline polymers.
- Over these temperatures, the properties of the polymers change dramatically.
- Polytetrafluoroethylene has a much higher T_g compared to polyethylene and polypropylene.



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The next one is high thermal stability. So under high temperatures the membrane material may not be able to resist degradation or decomposition, changing in the nature of membrane depends on the glass transition temperature T_g for an amorphous polymer and the melting temperature for crystalline Polymers. So, about this temperature is the properties of the polymer changed dramatically polytetrafluoroethylene PTFE has a much higher glass transition temperature compared to polyethylene or polypropylene.


Since we have been discussing about and we understand that the membrane required for membrane distillation should be as much hydrophobic as possible. Ok so then we need to try to understand that. How do you increase or enhance the hydrophobicity of the membranes? So the main objective of the modification is incorporated enhance the hydrophobic character of the membrane surface and surface, of course the use of fluoro-polymers gain popularity for such some modifications.

Thermal stability mechanical strength and lower surface energy at the main attractions for use of this polymers recently in a work using why they introduced CF₄ plasma surface modification on hydrophilic asymmetric PS membrane. The modified membrane showed a contact angle of 120 degree and trans-membrane flux of 45 litres per metre square hour for a 4% sodium chloride solution at a feed inlet temperature of 63 degree centigrade.

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Enhancement of hydrophobicity

- In another work, Zhang et al. used a spray of polydimethylsiloxane (PDMS) and hydrophobic SiO₂ on PVDF membrane to render hydrophobic character.
- A water contact angle of 156° was achieved for 1.5% particles in the spray.
- The modification ensured the operational stability of the process and a permeate of very high quality was achieved during long time operational run.
- Recently, Zhang and Wang modified the surface of polyetherimide hollow fiber membrane with fluorinated silica layer.
- A dramatic increase in hydrophobicity of the membrane was observed due to increased surface roughness and decrease surface energy of the membrane.



So another work is done at all used as properly dimethylsiloxane, which is called as PDMS and hydrophobic silicon dioxide on PVDF membrane to render hydrophobic character. A water contact angles of 156 degrees centigrade was achieved for 1.5% particles in the spray. The modification ensure that operational stability of the process is stable and permeate of a very high quality was achieved during long time operational run.

In another work Zhang and Wang modify the surface of polyetherimide hollow fibre membrane with chlorinated silica layer, a dramatic increase in hydrophobicity of the membrane was observed due to increased surface roughness decrease surface energy of the membrane.

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MD Membranes Fouling

- Due to different transport phenomena, generally rigorous feed characteristics and use of hydrophobic membranes, the nature of fouling in MD is different from other low pressure membrane processes.
- Scale formation at the membrane surface is the most common form of fouling observed in MD process when applied to concentrated salt solutions.
- The mass transfer parameters also affect the MD performance.
- The mass transfer rate across microporous hydrophobic membrane in MD is driven by the temperature gradient across the membrane surfaces.
- The heat losses attributed to the conduction through the membrane and convection associated with the vapor transport reduce the surface temperature at the feed side and increase the corresponding temperature at the permeate side, thus, inducing the thermal polarization at both sides.

So, then let us understand the membrane distillation and membranes fouling. As you know due to different transport phenomena, generally rigorous feed characteristics and use of hydrophobic membranes. The nature of fouling in membrane distillation is different from other low pressure membrane processes. A scale formation at the membrane surface is the most common form of fouling observed in membrane distillation process when applied to concentrated salt solution is the mass transfer parameters.

Also affect the Distillation performance the mass transfer rate across microporous hydrophobic membrane in membrane distillation is driven by the temperature gradient across the membrane surfaces. The heat process attributed to the conduction to the membrane and convection associated with the help of transport reduce the surface temperature at the feet side and increase the corresponding temperature at the permeate side thus increasing the thermal polarization at both sides.

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- The scale formation at the membrane surface has been observed in the studies addressing the MD applied to solutions containing salts.
- Ca and Mg were identified as the main scale forming salts.
- The scale formation in MD was pointed out as one of the major responsible factors for wetting, flux reduction and damage to the membrane structure.
- The formation of porous deposits decreases the flux by lowering the heat transferred to the membrane surface while the non-porous deposits increase the resistance to the mass transfer.
- The effect of wetting is not only restricted to possible reduction in flux and degradation of permeate quality but also a severe fouling inside the pores caused by the precipitated/adsorbed materials.

Other scale formation at the membrane surface has been observed in the studies and addressing in the membrane distillation applied to solutions containing salt. Calcium and magnesium identify calcium and magnesium calcium and magnesium are identified as the main scale forming salts are the scale formation in membrane distillation was pointed out is one of the major responsible factors for wetting flux reduction as well as damage to the membrane.

The formation of porous deposit decreases the flux by lowering the heat transferred to the membrane surface will the non porous deposits increase the resistance to the mass transfer. The effect of wetting is not only restricted to possible reduction influx and degradation of permeate quality, but also a severe falling inside the pore caused by the precipitated observed materials.

So let us; will show you have actually design all real life pictures. So you can see these are scanning electron microscope images all this 1, 2, 3, 4 and this is the real picture of how the scaling is taking place here you can and you can see here also there in microscopic image. These are the scaling here. This is the scale that is taken place. Ok. And these are the material that is forming scales. Ok, maybe calcium and magnesium.

This is how actually the scale formation is happening during membrane distillation and its side channels and these are all Hollow fibre systems basically.

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Table. Different types of fouling observed in MD studies.

Feed type	Membrane used	Type of fouling
Wastewater from heparin production	PP capillary membranes	Wetting, deposition, scaling, biofouling
NaCl solution	Accurel PP S6/2 membrane	Wetting, surface scaling
Synthetic wastewater	PVDF flat sheet MILLIPORE	Wetting, thick layer of biofouling
Bilge water, saline water from meat processing industry, tap water	Accurel S6/2 PP	Deposit layer formation at the surface, bio fouling, surface and internal crystallization
Skim milk and whey solution	PTFE flat sheet membranes with woven PP support	Layer of deposits at the membrane surface
Municipal water and flu gas condensate	PTFE flat sheet	Scale formation at the membrane surface

Courtesy: Droti et al. Desalination, 356, 2013, 56-64.



So, different types of fouling observed in the membrane distillation studies. The first column is field type, second is membrane use and third is type of fouling. Let us understand the wastewater from heparin production. Ok so here you use a polypropylene capillary membrane. So, the type of fouling is due to wetting the deposition scaling and biofouling desalination of sodium chloride solution. So accurel polypropylene S6 by two membrane wetting and scaling are responsible for fouling.

For synthetic wastewater PBDA flats sheet millipore membrane is being used wetting and thick layer of the biofouling are the types of fouling. In case of bilge water again the same as accurel S6 by 2 PP membrane. So, deposit layer formation at the surface by fouling surface and internal crystallization all these are responsible for fouling. Skim milk and their solutions polytetrafluoroethylene flat sheet membrane with you when polypropylene support is being used.

Layer of deposits are the membranes are found Municipal water tank and flu gas condensate. So it is a PTFE flat sheet is being used scale formation at the membrane surface.

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MD Applications

➤ The applications can be classified as to whether:

- i) permeate is the desired product or ii) retentate is the desired product.

The production of pure water

In most applications the permeate is the product of interest. A high quality permeate can be obtained with membrane distillation, as for example

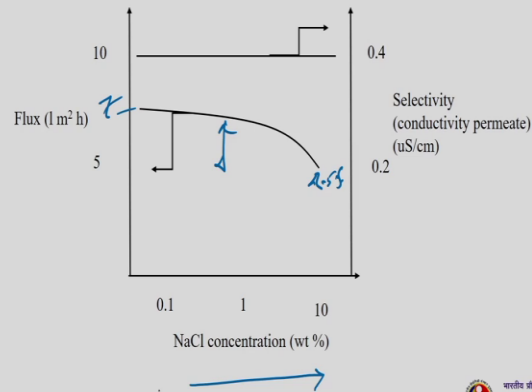
- water for the semiconductor industry
 - boiler feed water for power plants
 - desalination of seawater
- The quality of the permeate remains high even at high feed concentrations.

So now let us understand and the different applications of membrane distillation process. So other applications can be classified into 2 types basically depending upon whether desiring product you are drawing in the permeate side or the desired products in the retentate side. In most application permeate is the product of interest unlike other membrane processes. So in most of the membrane distillation systems usually the product of interest comes through permeate side. So, the first one let us understand the production of pure water.

So a high quality can be obtained with membrane distillation so for example, we are talking about water for semiconductor industry, you need ultra pure water boiler feed water for power plants and desalination of seawater. The quality of permeates remain high feed concentration for all this applications.

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➤ A porous polypropylene membrane was used where the flux and selectivity was plotted as a function of the sodium chloride concentration.



So, just look at this is a typical example where porous polypropylene membrane is used as a flux and selectivity was plotted as a function of the sodium chloride solution. You see when you increase the sodium chloride solution from 0.1 to 1, 10% you see there is a decline in a flux. Ok, so almost you can say this is seven here it is coming to let us say for 4.5% this is a magnitude. Decline of flux right when you are increasing the sodium chloride concentration from 0.1 to 10% whereas the selectivity ok which is important 0.2, 0.4 you can see this is flat here.

The selectivity is almost remains same when you increase sodium chloride concentration that means selectivity is not depending upon the sodium chloride concentration during membrane distillation.

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- With increasing salt concentration, the flux shows some decline, because of a decrease in vapor pressure.
- On the other hand, the quality of the permeate is independent of the feed concentration.
- Whereas in seawater desalination, reverse osmosis is strongly affected by the osmotic pressure of the (highly) concentrated feed solutions.
- Membrane distillation can handle even higher salt concentrations without a substantial decrease in membrane performance.
- The removal of volatile organic components (VOCs), such as chlorinated hydrocarbons or aromatics, from an aqueous solution is another application.
- These volatile contaminants are often present in very low concentrations in surface water or industrial effluent.

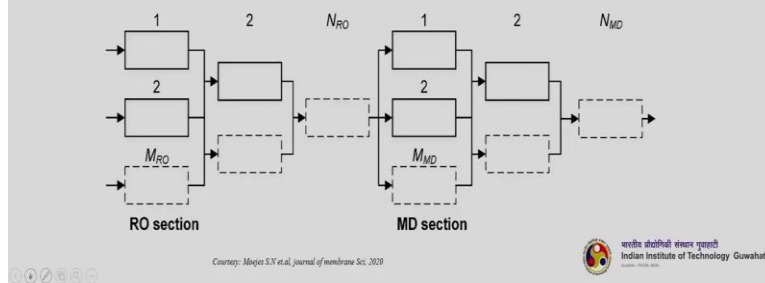
With increasing salt concentration the fluxes some decline because of a decrease in vapour pressure. So, on the other hand the quality of the permit is independent of the field concentration. Now, where is in seawater desalination reverse osmosis is strongly affected by the static pressure of the highly concentrated field solutions. Membrane distillation can handle even higher salt concentrations without a substantial decrease in membrane and performance.

Then when you talk about removal of volatile organic compounds this is one of the applications of membrane distillation or even you know in pervaporation is being used for removing evaporation so such as chlorinated hydrocarbons aromatic from an aqueous solution. Ok these volatile contaminants are often present in very low concentrations in surface water or in industrial effluents that by making others traditional systems of separation almost inefficient for removing the VOC's so that you have to rely upon some sort of membrane based separation process, whether it is a PP or MD.

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Air gap membrane distillation for milk concentration

- Application of MD for concentrating of other food products.
- Here milk was considered as feed.
- Membrane technology with reverse-osmosis and membrane distillation was used.
- In MD, a porous hydrophobic membrane separates the feed and permeate phases and allows only water vapor to diffuse through the membrane.



Another example is air gap membrane distillation for milk concentration. So here membrane of distillation is applied for concentrating food products milk was considered as the feed. Membrane Technology with reverse osmosis membrane distillation is what was used in cascade system type of things. You see first Ro section thereby followed by a membrane distillation system what is happening? There is anything as much as it can then finally membrane separation is doing the final enrichment.

MD a porous hydrophobic membrane separates the field and purposes and allows only water vapour to diffuse to the membrane.

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- Concentration of milk starts with RO to the maximal possible concentration of milk (18% solids).
- RO is followed by membrane distillation to concentrate milk to the final 50% solids.
- The used air gap membrane distillation (AGMD) has the advantage of internal heat recovery and is therefore often preferred over direct contact membrane distillation.
- Reverse osmosis is favorable until its maximum achievable concentration.
- Air gap membrane distillation is, despite the low operational temperatures, energy intensive for the concentration of milk.
- Reducing recirculation rates gives a major reduction of operational costs.

So concentration of milk starts with RO, so RO is concentrating it almost 18% solids RO is followed by a membrane distillation to concentrate the milk to final almost an year 50% of the solid. So, that the used air gap membrane distillation as the advantage of internal heat recovery and is therefore open preferred over direct contact number in distillation. So, AGMD has the better in case of this and enrichment of Milk products.

As a reverse osmosis is favourable until its maximum achievable concentration, air gap membrane distillation is despite low operational energy intensive temperature for concentration of milk, reducing circulation that gives a measure reduction in operational cost. This is a classic example of use of membrane distillation in dairy industries.

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Bilge water separation by membrane distillation

- A ship is a closed system; hence the wastewater originated from washing, leakages of holds or from condensation of water vapor is collected in the lowest part of a hull (ship bilge).
- The bilge water contains all the pollutants generated on the ship, including significant amounts of oily pollutants generated mainly from the engine room.
- Moreover, a composition of bilge water differs significantly for each ship, but as a rule, besides a mixture of fresh and seawater, the oily bilge water also contains diesel fuels, oily fluids, grease, suspended solids, heavy metals and surfactants.
- A volume of bilge water increases during ship exploitation; therefore, it must be discharged into sea.
- In accordance with MARPOL 73/78 convention, the oil concentration in the discharged water cannot exceed 15 mg/L.

Courtesy: Marek Czepa, Separation and purification Technology, 2020

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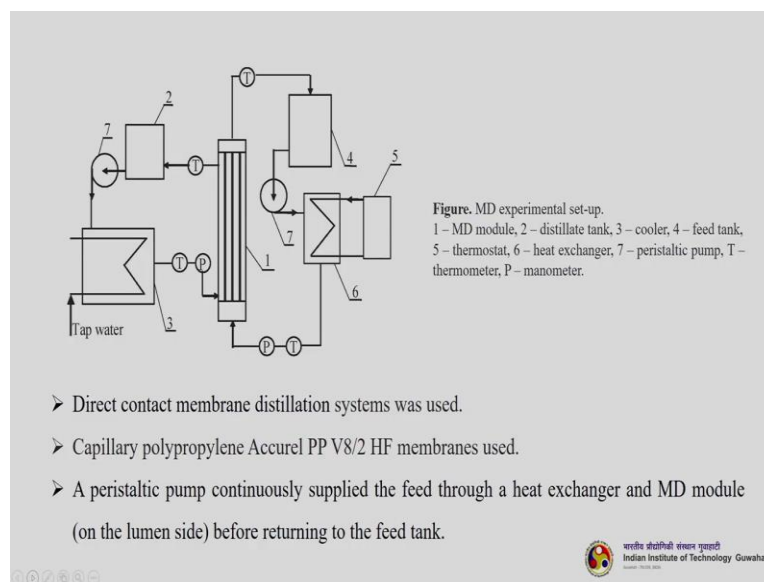
So in another application is bilges water separation by membrane distillation, you know a ship is a closed system and when wastewater are generated from different sources in ship whether it is washing asking whether it is coming from leakages of holes are from condensation of water vapour. Everything is getting collected in the lowest part of a whole which is called the ship bilge. This bilges water contents all polluted generator on the ship including significant amount of oil pollutants generated mainly from the engine room.

Oils in gresation and all this things more over the composition of bilge water differ significantly for each ship but as a rule you can say that besides the mixture of fresh and sea water the oily

bilge water also contains diesel fuels, oily fluids grease, some suspended solids, heavy metals and some surfactant. Now a volume of bilge water increases during the ship exploitation that it must be discharged into the sea. Now when you are talking about discharging this bilge water into the sea you need to take care of the discharge recommendations.

So, accordance with the MARPOL 73/78 convention which recommends how do you actually basically discharge the bilge water in to sea, if the oil concentration in the discharge water must be less than 15 milligrams per litre of 15 PPM.

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So, this is an MD experimental setup for treatment of the bilges water or bilge wastewater. So, the first one is you see this is a hollow fibre type of capillary type of system here is a membrane distiller module system second is this is the distillation tank distiller tank, third is cooler, this is a cooler and fourth is a feed tank ok here it is feed. Fifth is thermostat, ok this is a thermostat sixth is heat exchanger, seventh is peristaltic pump and eight is thermometer and P monometer.

Now a direct contact membrane distillation system was used in this case. A capillary polypropylene accurel PPV 8 by 2 hollow fibre membrane used for this treatment of the bilges water or wastewater. A peristaltic pump continuously supplied the feed through heat exchanger in the MD module and on the lumen side before returning to the feet tank.

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- The hydraulic pressure at the module inlets was below 1 kPa.
- The cooled distillate was recirculated on the external membrane surface with a similar volume flow.
- The distillate temperature was maintained at 293 ± 1 K.
- The feed temperature was 323 K or 343 K during bilge water treatment experiments or in the range of 323–353 K during the MD studies with standard NaCl solutions.
- During the last series of MD experiments the continuous feed concentration was carried out to achieve more than 80% water recovery.

Now the hydraulic pressure at the module inlet was below 1 kilopascal. So the cooled distillate was re-circulated on the external membrane surface with a similar volume flow. The distillate temperature is maintained at $293 + \text{or} - 1$ Kelvin. The feed temperature was 323 Kelvin or 343 Kelvin during bilge water treatment experiments or in the range of 323 to 353 Kelvin during MD studies which standard sodium chloride solution.

During the last series of MD experiments the continuous feed concentration is carried out to achieve more than 80% of the water recovered.

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Other MD Applications

ii) The concentration of solutions

Membrane distillation can be used for the concentration of solutions in some cases.

Ex. Waste water treatment concentration of salts, acids, etc.

iii) The removal of volatile bio-products

Volatile bio-products, such as ethanol, butanol, acetone or aroma compounds can be removed by membrane distillation.

So, apart from this, you know, there are the applications of membrane distillation where also reported you can read more from literature. There are fantastic review papers available on membrane distillations and so you can download those and you can go through. So, the concentration of solution, membrane distillation can be used for concentration of solution some in some cases just like wastewater treatment concentration of salts, acids etc where we remove salts and acids and recover them.

The removal of volatile bio products, the volatile bio product such as ethanol, butanol, acetone or aroma compounds. So, these are produced during fermentation usually if you talk about this, this produced by AB fermentation. Ok, so this needs to be removed. OK when the fermentation is going on because beyond certain concentration of ethanol, butanol and acetone or any of the solvents. Ok the microorganisms will become dormant they will not become active to carry out the fermentation. So this needs to be removed by using membrane distillation.

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- In the simplest type of construction two compartments are separated by a membrane.
- Evaporation occurs on the high-temperature side and hence the temperature of this liquid will decrease.
- In contrast, condensation occurs on the low-temperature side and the temperature will increase.

- In commercial installations the process will be carried out in a counter-current flow, which allows a constant temperature difference to be set up across the membrane.
- The temperature of the feed solution decreases but the temperature of the permeate increases.
- A substantial portion of the heat is transferred from the feed side to the permeate side and part of this energy can be recovered.

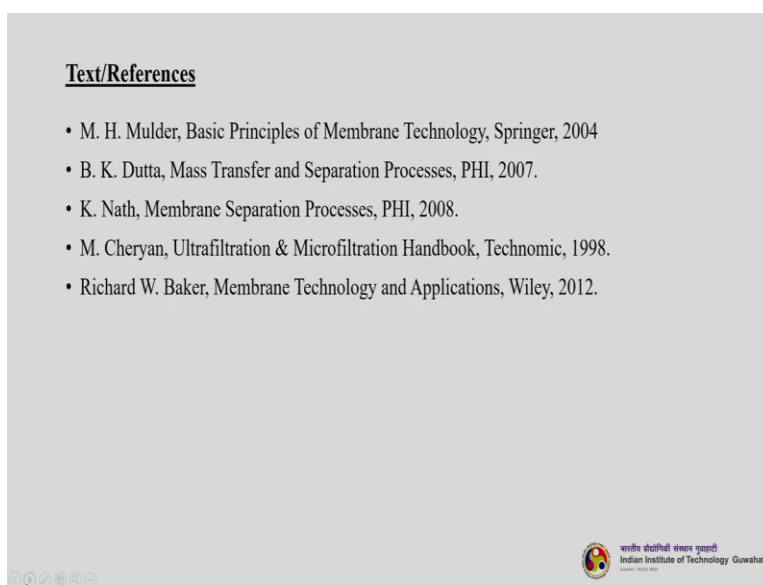
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So you can see that the simplest types of construction of the two compartments are separated by a membrane that is simple type of membrane distillation. You can just imagine that there is a membrane in between and there is upstream takes place there is a downstream takes places. It is a simple module. So evaporation takes place in the high temperature side ok the feed side and the temperature of this liquid will decrease.

Then in contract condensation occurs the lower temperature side and the temperature will increases, please refer to this figure you see feed is at 90 degree centigrade OK when it leaves the module it becomes become 50 degree almost 40 degree centigrade temperature is being lost this in the form of evaporation. You can see the permeate is entered as 45 degree centigrade and the same 40 degree has been added to hear you get 85 degree centigrade is permeate in commercial installation.

The process will be carried out in counter current flow which allows a constant temperature difference to setup across the membrane. The temperature of the feet solution decreases, but the temperature of the permeate increases. A substantial percentage of the heat is transferred from the feed side and permeates side and part of this energy can be recovered.

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So, with this I conclude today's lectures you can refer books all of these nowhere membrane distillation is given in detail. So there are two beautiful this one review papers which I have given the references also in the slides you can see those, please refer those to read for membrane distillation and this much whatever I have discussed today is for a basic understanding. So, with this I conclude so thank you very much.

In case you have any doubt please feel free to write to kmohanty@iitg.ac.in. In the next lecture that is the last module so will have three lectures under this module 34, 35, 36 in the next this

module facilitated transport mechanism, membrane contactor and other membrane processes. So in the first lecture of this module that is lecture 34 under Model 12 we will discuss the mechanism of copper transport, carrier agents, active transport passive transport and their applications. So thank you very much.