

Thermal Processing of Foods
Professor R. Anandalakshmi
Chemical Engineering Department
Indian Institute of Technology Guwahati
Lecture 19

High pressure dialysis, ultrafiltration and reverse osmosis

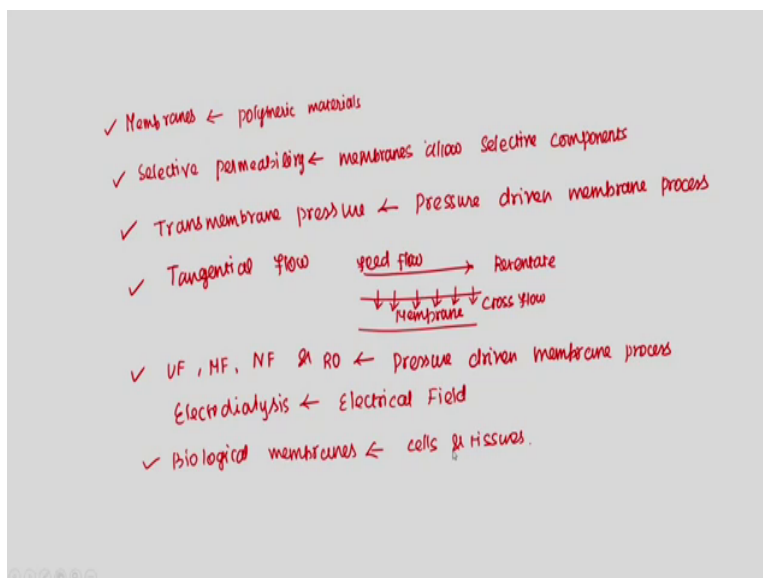
(Refer Slide Time: 0:54)

Types of Membrane Process

- The driving forces of membrane processes
 - Concentration gradient ✓
 - Transmembrane pressure ✓
 - Chemical potential ✓
 - Osmotic pressure ✓
 - Electric field ✓
 - Magnetic field ✓
 - Partial pressure ✓
- Ultrafiltration, microfiltration, and reverse osmosis membranes are classified on the basis of molecular size of pores and transmembrane pressure ✓
- Pervaporation is based on separation driven by partial pressure and selectivity that depends on solubility and diffusivity of species in the membrane ✓

Good morning everyone. So, today we are going to continue yesterday's class so yesterday we have discuss about fundamentals of membrane processes. So, we ended up saying that, there were four major categories of the membrane processes we are going to discuss in next two classes. So they were ultrafiltration, microfiltration, reverse osmosis as well as nanofiltration techniques, and also we are going to discuss about the electro dialysis as well.

(Refer Slide Time: 1:05)



So, if you remember a short recap, so we discuss about the membranes so which is mostly of polymeric mostly but there were ceramic membrane also we were seen. And also the another term is selective permeability. So that means, so that means the membranes having selective permeations. So that means it can allow only selected components, selective permeability or membranes allow selective components.

So, another important thing we talked about is transmembrane pressure, transmembrane pressure in that, if it is a pressure driven membrane process, so this is very much important. Another one thing is we talked about tangential flow so that means, your feed is this is your membrane so, your feed is flowing parallel to the membrane surface and the permeate is, permeating through a membrane so that means it is cross-flow and the retentate will be collected here.

So, this is a feed flow so that is parallel to the membrane surface. So, these were few important terms we have discussed and also whatever we are going to discuss, what is that? Ultrafiltration, microfiltration and nanofiltration and reverse osmosis. So, all of them are pressure driven membrane process and also we are going to discuss about electrodialysis. So that is electrical field driven that means driving forces electrical over there.

Other than that pervaporation also there but we are not going to discuss it in depth. Pervaporation, what happens is? The vaporization through the membrane is a selective process. So the vaporization of molecules through membrane surfaces nothing but a selective

permeability, so other than that there are biological membranes, so which were used to do retain cells and tissues. So this is the short recap.

(Refer Slide Time: 4:53)

Pressure Driven Membrane Processes

- In MF and UF, particle size is practically the sole criterion for permeation or rejection
- RO membranes separate particles at molecular level and their selectivity is based on the chemical nature of the particles
- NF is, in essence, a membrane process similar to RO

| Process | Typical operating pressure range (MPa) | Limit particle size range (nm) or (molecular weight) |
|---------|----------------------------------------|------------------------------------------------------|
| MF ✓ | 0.1-0.3 $\times 10^6$ Pa | 100-10,000 ✓ |
| UF ✓ | 0.2-1 | 1-100 (10^2 - 10^6 Da) |
| NF ✓ | 1-4 | 0.5-5 (10^2 - 10^3 Da) |
| RO ✓ | 3-10 | (10^1 - 10^2 Da) ← Not wt. CB r |

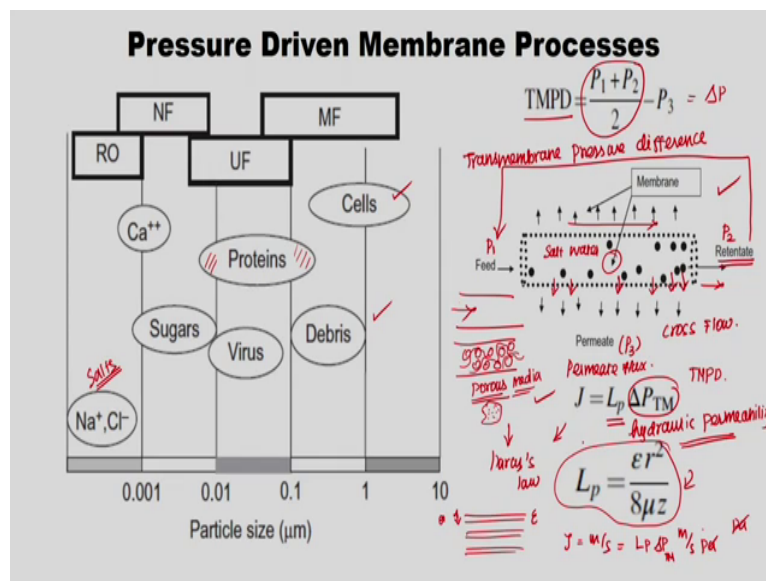
So, now we are going to discuss about pressure driven membrane processes which includes all of microfiltration, ultrafiltration, and nanofiltration as well as reverse osmosis. In micro and ultrafiltration the particle size is a practically the sole criterion for permeation or rejection. So based on the particle size only your permeate will permit through the membrane or retentate is rejected on the membrane surface.

RO a membrane separate particles at molecular level, so reverse osmosis membrane separate particles at molecular level also their selectivity is based on the chemical nature of the particle. For other microfiltration, ultrafiltration particle size is sole criterion but here the molecular level as well as the chemical nature of the particle is important in the reverse osmosis membrane.

So, nano-filtration in sense membrane process similar to RO, but it has also the overlap with the ultra and microfiltration, so here your operating pressure ranges. Normally, in microfiltration it is 0.1 to 0.3 mega-Pascal, mega-Pascal nothing but 10 to the power of 6 Pascal. Ultrafiltration is 0.2 to 1, nanofiltration is 1 to 4, reverse osmosis is 3 to 10. And in terms of particle size limit particle size in the sense, so that is the particle size beyond that it cannot allow.

So, limit particle size which is in nanometers as well as molecular weight is also given. So microfiltration is 100 to 10,000 nanometer the particle range, which it can retain and ultrafiltration is 1 to 100, if in terms of molecular weight it is a 10 to the power 2 to 10 to the power 6 Dalton and nanofiltration 0.5 to 5 nanometer and in terms of molecular weight 10 to the power 2 to 10 to the power of 3 Dalton, and reverse osmosis 10 to the power of 1 to 10 to the power of 2 Dalton, but here it is solely depend upon the molecular weight of the particle.

(Refer slide Time: 7:24)



So, here it is a clear picture, so where you can see clearly the particle size based on that how your micro, ultra, Nano and reverse osmosis membrane processes is categorize. So, if you see the micro filtration which removes the cells and debris but it allows the protein, so if you see the protein, protein has this overlap, it is not clear cut definition, clear cut in the sense you cannot say all protein can permeate and also you cannot say all permeate can be retained in the membrane surface.

But cells and debris normally be removed then if you go to ultrafiltration proteins and viruses and it allows the sugars, but anyway there is no clear cut particle size range. Some of the protein particles go into permeate and some of the viruses are also pass through that means permeate through. And nanofiltration mostly sugars and also it can contribute to calcium ions but if you go to reverse osmosis all are ionic levels, na plus cl minus, so this is mostly salt that is why use extensively in desalination of water over reverse osmosis.

So, microfiltration which comes 0.1 to 10 the micrometer ultrafiltration 0.01 to 0.1 nano 0.001 to 0.01 micrometer then less than 0.001 micrometer is nothing but your reverse

osmosis. So here is the tangential flow membrane process, so you have a feed here, so this is the membrane surface, so this is the membrane surface and your permeate is permeating through the membrane surface, this is the membrane, through the membrane surface your permeate is permeating and retentate will be inside the membrane that is three times, so that is collected later.

So if you see the feed is flowing parallel with the membrane and your permeate flows in the cross-flow that is perpendicular to the membrane surface. And another important thing is that this transmembrane pressure difference, so transmembrane pressure difference. So this is the main driving force because these are all pressure driven membranes. So that TMPD is nothing but a feed pressure is P_1 and outlet when it gets collected so that is P_2 . So, the average of this two is P_1 plus P_2 divided by 2. And P_3 is nothing but a permeate pressure, so that is minus P_3 .

So, this is nothing but a transmembrane pressure difference, which is nothing but average between inlet and outlet pressure minus the permeate pressure. And also remember what happens is, when the feed is introduced into the membrane, so it contains solids plus, for example, if we take the water with salts, so salt water if we take, so the water gets permeated through the membrane. So, what happens, the salt concentration will be increasing towards the end, so this side salt concentration started increasing.

So, what happens is, after some time the flow rate of the suspension, it may not be high enough to drive the flow, so in that case what happens? The salts get deposited, so it cannot flow further through the membrane to get it separated. And to avoid that sometimes normal process can be changed; the retentate is given back to the feed, to increase the suspension flow rate through the membrane. So that is another criteria one may follow to increase the suspension flow rate when it passes through the membrane to avoid the salt concentration which is not carried over by the normal suspension flow.

So, here is the formula forever the permeate flux, J is here nothing but permeate flux, so this L_p is nothing but hydraulic permeability and ΔPM is nothing but your transmembrane pressure difference. So, this is the simple modal so here what we uses? The Darcy law, Darcy law in the sense you might have heard something like porous media flow, so porous media flow is nothing but I have channel, so in which the solid particles are there, so normal

velocity normally if you see the pipe where your fluid is flowing, so there is flow resistance to it when it sees the solid particles so the flow will be somewhat reduced.

The velocity of the flow will be reduced, so it sees the resistance over that so the flow through this solid particle is nothing but the porous media flow or sometimes, the solid particle itself will have pores in it. For example, so this is my solid particles so it is porous so that also contributes the extra resistance, so these kinds of flows are called porous media flow. So, further the governing law is nothing but a Darcy's law, so here also what we consider here is so here we consider them as capillaries.

The membrane materials or parallel to each other so their radius is nothing but r , and it has the porosity of ϵ , so from there the hydraulic permeability is derived as $\frac{\epsilon r^2}{8 \mu z}$. So, r is nothing but the radius of the capillary ϵ is porosity and μ is a viscosity. So, this has unit of J has the unit of meter per second and L_p has unit of meter per second Pascal so your ΔP_m has one Pascal.

So both the side unit is cancelled. So the permeate fluxes in meter per second, so this L_p is the hydraulic permeability depends upon the many factors that we will see but also here remember this is a modal, Darcy Law modal we assumed but there may be a restrictions to use this modal on certain assumptions, so here we are assuming that is Darcy law modal and deriving this, there are certain restriction to use this modal that one should remember.

(Refer Slide Time: 14:38)

Sieving Coefficient and Rejection

- where C_{perm} and C_{retn} are the concentration of the solute in the permeate and in the retentate (measured at the membrane interface), respectively.
- For particles considerably larger than the widest pore, rejection is total, that is, $S = 0$.
- Particles considerably smaller than the smallest pore are not retained at all ($S = 1$).
- For solutes with particle size close to the pore size: $0 < S < 1$.

$$S = \frac{C_{perm}}{C_{retn}}$$

Then the terms called Sieving coefficient as well as rejection, so sieving coefficient is nothing but concentration of the solute in the permeate C_{perm} , and in the concentration of the solute in the retentate that is nothing but $C_{retentate}$. So, S here is sieving coefficient. So for particle considerably larger than the widest pore, widest pore larger than that is rejection is total so that means S equal to 0. So, if the particles considerably smaller than the smallest pore or not retained so in that case the sieving coefficient is one.

But for solute with particle size closed to the pore size than the sieving coefficient may vary. So, that means if I have widest pore my particles get in or if I have very smallest pores my all particles will be retained. So here it is 0 here it is one so in between normal real operations will have sieving coefficients between 0 to 1.

(Refer Slide Time: 15:47)

Sieving Coefficient and Rejection

$$R\% = (1 - S) \times 100$$

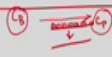
- MF membranes are specified by their average pore diameter (e.g., 0.5 μm) while UF membranes are characterized by their cutout molecular weight (COMW)
- COMW is the molecular weight of the smallest molecule retained by the membrane.
- For the purpose of this definition, $R = 95\%$ is usually accepted as total rejection.
- It will be said, for example, that the cut-out molecular weight of a certain membrane is 100,000 Da

So, then rejection coefficient: So rejection coefficient is nothing but one minus S into 100 that is very much obvious. The microfiltration membranes are specified by their average pore diameter so that is nothing but around 0.5 micrometer. But ultrafiltration membranes are characterized by their cut-out molecular weight that is COMW.

So, COMW is the molecular weight of the smallest molecule retained by the particular membrane. So, for this purpose the definition is, how do I define the smallest molecule retained by the membrane, so that is nothing but retention coefficient of 95 percentage usually accepted as total rejection. So, it will be said for example that cut-out molecular weight of the certain membrane is nothing but 100 thousand Dalton.

(Refer Slide Time: 16:38)

Concentration Polarization and Gel Polarization

- Consider ultrafiltration of a solution of protein ✓
- Assume ^{ideal scenario} total rejection of the protein by the membrane and unrestricted permeation of the solvent
- At the membrane surface, the protein is separated from the solvent.
- A protein concentration gradient normal to the membrane surface is created. 
- Protein concentration near the membrane is higher than in the bulk of the solution, a distance away from the membrane. This situation is called "concentration polarization."

So, then the next important terminology we need to be taken into account in the membrane separation processes, concentration polarization as well as gel polarization. So, if we take the, consider, for example, ultra-filtration of a solution of protein, so here assume the total rejection of the protein by the membrane and unrestricted permeation of the solvent. So this is ideal scenario.

So, at the membrane surface protein is separated from the solvent. The protein concentration gradient normal to the membrane surface is created, so due to which only the solvent is permeating. So, the protein concentration gradient normal to the membrane surface so, for example, here my protein as well as solvent is flowing so the protein gets retained in the membrane surface and your solvent is passing though permitting through, so if you see the bulk concentration in the feed and the concentration on the protein in the membrane surface so I will put it as C_p .

So, between C_p and C_b so there is concentration gradient is developing. The protein concentration near the membrane is higher in the bulk of the solution is higher than in the bulk of the solution, so that means when the feed is flowing along with the membrane surface, the concentration of the retained from the bulk solution is moving towards the membrane surface. So over the time what happen is, the protein concentration near the membrane C_p is higher than the C_b .

So, the distance away from the membrane, so this situation is called concentration polarization. So, C_b is having lower concentration than the C_p , C_p is near to the membrane surface. C_b is away from the membrane surface. So, due to this concentration gradient there is one point where C_p becomes higher than the C_b , so this is nothing but a concentration polarization.

(Refer Slide Time: 18:56)

Concentration Polarization and Gel Polarization

- The high concentration at the upstream face of the membrane causes osmotic backpressure, resulting in backflow of permeate (solvent) toward the retentate. retentate
- This effect is not particularly significant in UF and MF, where the retentates are suspensions of solid particles or solutions of substances with relatively high molecular weight, hence the low osmotic pressure. RO
- The concentrated and hence highly viscous layer at the membrane interface constitutes an additional resistance to flow toward the membrane

And the high concentration at the upstream face of the membrane causes the osmotic backpressure, so if you remember the last class we told, so the solvent moves through the membrane from the higher concentration to lower concentration. So, what happens, so you are in the membrane surface, so your concentration of proteins started developing, the concentration gradient started developing, so what happens?

The solvent is permeating back from the permeate side to retentate side, so that is the osmotic backpressure resulting in a back flow of permeate toward the retentate. So, this effect is not particularly significant in ultrafiltration as well as microfiltration where the retentate or suspensions of solid particles or solution of substances with relatively high molecular weight hence low osmotic pressure.

Actually, so this concentration polarization is very much dangerous in RO, so because we told that there retentate mostly is salt, the salts has high osmotic pressure, so when to overcome that osmotic pressure when the backflow happens then that becomes issue, but here

the this effect is not particularly significant because retentate in this micro or ultrafiltration is suspension of solid particles or solutions of the substance with relatively high molecular weight solid.

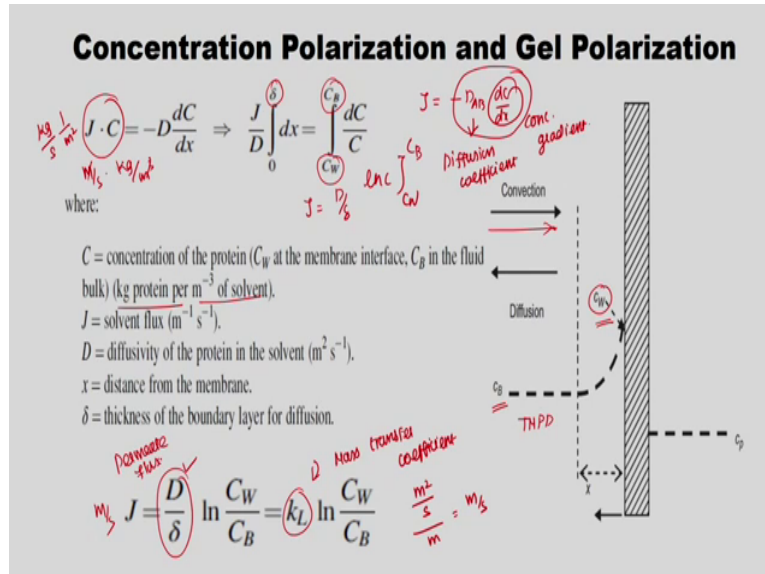
So we need only less osmotic pressure only but that is not the case with the reverse osmosis where salts are having high osmotic pressure compare to the solid particles are high molecular weight components. The concentrated and hence highly viscous layer at the membrane interface constitutes an additional resistance to the flow toward the membrane.

So, once the concentration gradient is developing in the membrane surface near the membrane surface so it forms highly viscous layer because it is protein concentrate so at the membrane interface which constitute are additional resistance to flow, so it stops the or it creates the resistance for the permeate through the membrane surface.

(Refer Slide Time: 21:12)

Concentration Polarization and Gel Polarization

- The transport of solute at the upstream side of the membrane is composed of two components in countercurrent to each other:
 - ✓ Solute transport from the fluid bulk toward the membrane, by virtue of the flow of solvent under the effect of the TMPD.
 - ✓ Solute transport from the membrane interface toward the bulk under the effect of the concentration gradient (back-diffusion).
- At steady state, the local concentration does not change with time; hence, the two effects must be in equilibrium. Assuming Fick's law for the back-diffusion, the steady-state condition can be written as follows:



So, the transport of solute at the upstream side of the membrane is composed of two components in counter current to each other. The first one is solute transport from the bulk, bulk fluid toward the membrane surface, so that means C_B which is the bulk concentration; C_p is nothing but near the membrane surface. So the solute should transport from the bulk phase to near the membrane by virtue of flow of solvent enter the effect of transmembrane pressure difference.

Then the solute transport from the membrane interface towards the bulk under the effect of concentration gradient, so that is nothing but due to the concentration gradient there may be back-diffusion. So, if you see here this is so from what they were telling so from bulk concentration to near the membrane surface follows the convection mode so solute is transport from bulk concentration near the wall membrane where it is here it is told us C_W .

And then the second one is, the solute transport from the membrane interface toward the bulk under the effect of concentration gradient, so this is due to the solute transport from C_B bulk concentration away from the membrane surface to near the membrane surface is due to TMPD that is nothing but trans membrane pressure difference. The back diffusion of the solute near the membrane surface to the bulk is due to diffusion. So, that is due to concentration gradient.

This is what probably in last lecture we told, so the pressure gradient concentration gradient both affects the separation process here. So, at steady state at the local concentration does not change with time, so if the one steady state is reached, hence, the two effects must be in

equilibrium. So, the back diffusion as well as the convection of solute from the bulk to membrane surface, so both are in membrane surface, so both are in equilibrium.

Assuming Fick's law further back diffusion the steady state condition can be written as follows so this equation. So, the Fick's law diffusion nothing but flux is minus $D \frac{dC}{dx}$ so this is nothing but diffusion coefficient. So, this is a concentration gradient and so here it is J into C , so J into C is nothing but a here this is convection process so flux into concentration, so fluxes in meter per second.

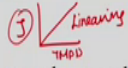
So your concentration is in $\text{KgF protein per meter cube of solvent}$, $\text{KgF protein per meter of solvent}$, so that means Kg per meter cube , so what you get is $\text{Kg per second per meter square}$. So that means so Kg per second is mass flow rate, per meter square is nothing but flux so this is mass flux. This is due to convection. The other side we told that, both are in equilibrium the other side it is minus $D \frac{dC}{dx}$, because the diffusion process is govern here by Fick's law of diffusion minus $D \frac{dC}{dx}$, so the C comes here so that D goes there because the flux divided by d_0 to Δ , Δ here is nothing but a boundary layer, boundary layer for diffusion.

So, dx equals to $C_W - C_B$, C_W is the concentration near the membrane, C_B is the bulk concentration, dC upon C so if you integrate what you get is J is nothing but D upon Δ . This is nothing but $\log C$, $C_W - C_B$. So this is J so this 0 to Δ . So what you get is D upon Δ so what you get is J equal to D upon $\Delta \log C_W$ by C_B . So this is D upon Δ according to film theory, so what you get is here as mass transfer coefficient.

So, this is K , D is in meter square per second, so this is meter so this also comes as meter per seconds. So C get cancelled so J also meter per second so the unit is cancelled. So this is what the theory behind the concentration polarization and how to calculate the flux. Solvent flux or permeate flux. Permeate flux from the concentration of the retented near the membrane of surface and the bulk.

(Refer Slide Time: 26:25)

Concentration Polarization and Gel Polarization

- If the flux is increased (e.g., by raising the transmembrane pressure difference), C_w increases accordingly, resulting in increased resistance to solvent flow toward the membrane. This explains, at least partially, the deviation of the membrane filtration rate from linearity 
- The concentration of the protein in the liquid layer adjacent to the membrane cannot grow above a certain limit (C_G) at which the layer becomes a gel. From this point on, the flux J cannot grow further and remains constant, independently of an increase in pressure. This phenomenon is called “gel polarization”
- The gelation concentration (C_G) depends on the protein and on the operation conditions (ionic strength, temperature, etc.)

So the next one is the gel Polarization, so what happens in the gel Polarization we will see. If the flux is increased that means the flux has to increase, then I can do it by transmembrane pressure difference. So C_w increases accordingly resulting in increased resistance to solvent flow toward the membrane, so what happen here? the further I will increase the TMPD, so then it increases the flux then that is why C_w , C_w is nothing but concentration of protein near the membrane surface that increases and resulting in increased resistance to solvent flow toward the membrane.

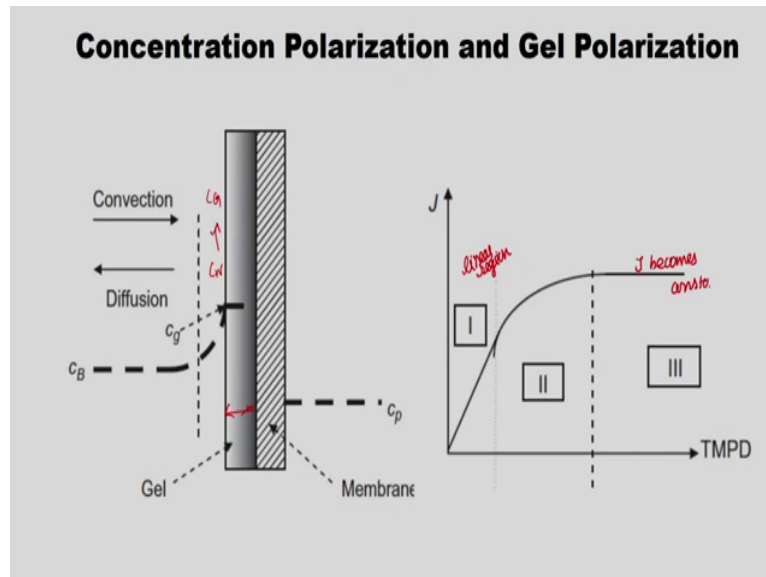
So because of this getting, the layer is getting thickened and thickened, your solvent float through the membrane is getting decreased and this explains at least the partially, the deviation of membrane filtration rate from the linearity. So, Normally what we have is with respect to TMPD, so I have the flux this is a J flux so the line is linear, so when you increase TMPD, the J also gets increase linearly so even gel polarization so that means the C_w increases near the membrane it also effect linearity here.

Linearity between J and TMPD the concentration of the protein in the liquid layer adjacent to the membrane cannot grow above the limit of C_G , so this is nothing but a gel concentration, at which layer becomes gel from this point on, the flux J cannot grow further and remains constant, independently often increase in pressure.

So, what we told? When we increase the transmembrane pressure difference, the J also increases linearly with TMPD, but there is a point where the concentration of protein near the

membrane surface reaches CG. How much ever I increase my transmembrane pressure, my J nothing but a permeate flux will not increase, so this phenomenon is called as gel polarization. The gel concentration CG depends on the protein and also the operation condition and strength as well as temperature.

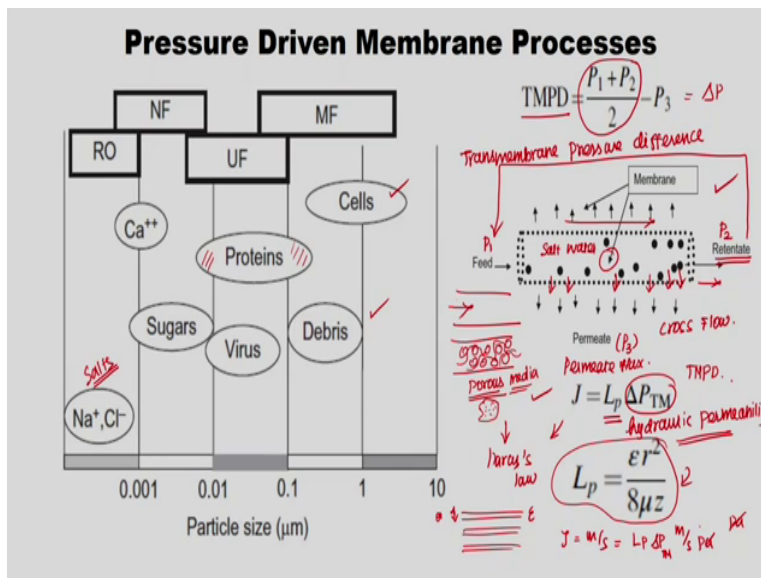
(Refer Slide Time: 28:47)



So, here I have taken the example of protein concentration but that may be true for other filtration as well as other materials as well. So this is nothing but a gel polarization, so this is what happens, so this is the gelation layer. So convection diffusion already you know C_B , so this is near that it is nothing but C_W , but now the C_W increases the critical value of C_G . So, there your permeate flux become constant is become constant, and then further increases in TMPD you will not see any increase, so this is nothing but linear line. Linear region so this is a transition and then after that your J becomes constant.

(Refer Slide Time: 29:40)

| Membrane Process | Membrane Type | Driving Force | Method of Separation | Range of Application |
|-------------------|----------------------------------------------------------|-----------------------------------------------|-----------------------------------------------------|-----------------------------------------------------------------|
| Microfiltration ✓ | Symmetric microporous membrane; 0.1 to 10 μm pore radius | Hydrostatic pressure difference 0.1 to 1 bar | Sieving mechanism due to pore radius and absorption | Sterile filtration clarification |
| Ultrafiltration ✓ | Asymmetric microporous membrane; 1 to 10 μm pore radius | Hydrostatic pressure difference 0.5 to 5 bar | Sieving mechanism | Separation of micromolecular solution |
| Reverse osmosis ✓ | Asymmetric skin type membrane | Hydrostatic pressure difference 20 to 100 bar | Solution diffusion mechanism | Separation of salt and microsolute from solution |
| Dialysis ✓ | Symmetric microporous membrane; 0.1 to 10 μm pore radius | Concentration gradient | Diffusion in convection-free layer | Separation of salt and microsolute from macromolecular solution |
| Electrodialysis ✓ | Cation and anion exchange membrane | Electrical potential gradient | Electrical charge of particle and size | Desalination of ionic solution |
| Gas separation | Homogeneous or porous polymer | Hydrostatic pressure concentration gradient | Solubility, diffusion | Separation of gas mixture |



So, the shape of the curve describing the variation of flux as a function of TMPD can be explained in the light of three models. The first one is linear model where I told Darcy law, so this is what I probably told here, so this model only applicable when you are having a linear line, so linear line in the sense J versus TMPD follows the linear line so there only you can apply the Darcy Law model that is what the restriction I told there.

And intermediate flux segment which is nothing but deviation from the linearity due to gradual buildup of resistance as result of concentration polarization. The high fluxes nothing but a saturation so this is an intermediate range, intermediate flux. So, the third one is the high flux where it is getting saturated, so the gelation, gel polarization is the reason for that so

practically constant flux, what it is? Independent of TMPD, even if you increase the pressure nothing happens to the flux.

For a given set of material properties KL, KL is nothing but the mass transfer coefficient whatever we have seen, that depends upon the tangential velocity so hence on the overflow rate through the module, so it depends upon the tangential velocity and the flow conditions which is nothing but tangential velocity, turbulence and the system property shape and dimensions of the flow channel and material properties which is nothing but a viscosity, density everything KL depends upon everything.

(Refer Slide Time: 31:21)

| Driving Force ✓ | | | |
|------------------------|-----------------------|-------------------------|-------------------|
| Pressure | Concentration | Temperature | Electropotential |
| Gas-vapor separation ✓ | Membrane extraction ✓ | Membrane distillation ✓ | Electrodialysis ✓ |
| Reverse osmosis (RO) ✓ | Dialysis ✓ | | Electroosmosis ✓ |
| Nanofiltration (NF) ✓ | | | |
| Ultrafiltration (UF) ✓ | | | |
| Microfiltration (MF) ✓ | | | |
| Pervaporation ✓ | | | |

So, these were few things we get to know before going to see different membrane process, so today's lecture we probably cover two, which is nothing but micro as well as ultrafiltration. So, here are the driving force for different membrane processes so based on the pressure we will have all four varieties, what we are concentrated on, particularly in food processing.

So pervaporation and gas evaporation separation also comes under pressure driven category. The concentration driven is membrane extraction as well as dialysis, and temperature driven membrane distillation process, electro potential electrodialysis, electro osmosis probably we are going to see little bit about electrodialysis as well.

(Refer Slide Time: 32:11)

| Membrane Process | Membrane Type | Driving Force | Method of Separation | Range of Application |
|-------------------|------------------------------------------------------------|-----------------------------------------------|-------------------------------------------------------|-----------------------------------------------------------------|
| Microfiltration ✓ | Symmetric microporous membrane; 0.1 to 10 µm pore radius ✓ | Hydrostatic pressure difference 0.1 to 1 bar | Sieving mechanism due to pore radius and absorption ✓ | Sterile filtration clarification ✓ |
| Ultrafiltration ✓ | Asymmetric microporous membrane; 1 to 10 µm pore radius | Hydrostatic pressure difference 0.5 to 5 bar | Sieving mechanism ✓ | Separation of micromolecular solution |
| Reverse osmosis ✓ | Asymmetric skin type membrane | Hydrostatic pressure difference 20 to 100 bar | Solution diffusion mechanism ✓ | Separation of salt and microsolute from solution |
| Dialysis ✓ | Symmetric microporous membrane; 0.1 to 10 µm pore radius | Concentration gradient ✓ | Diffusion in convection-free layer ✓ | Separation of salt and microsolute from macromolecular solution |
| Electrodialysis ✓ | Cation and anion exchange membrane | Electrical potential gradient ✓ | Electrical charge of particle and size ✓ | Desalination of ionic solution |
| Gas separation | Homogeneous or porous polymer | Hydrostatic pressure concentration gradient | Solubility, diffusion | Separation of gas mixture ✓ |

So, this gives you the overall picture, so the microfiltration, ultrafiltration, reverse osmosis, dialysis, electrodialysis, where it is used, what are all the membrane type, whether it is a symmetric membrane or what is porous nature, and what is the pore radius, pore radius already we have seen and the driving force so everywhere the hydrostatic pressure and dialysis it is a concentration, electrodialysis it is the electrical potential gradient.

And method of separation sieving mechanism due to pore radius and absorption, microfiltration we already told because the protein concentration the sieving mechanism in ultra and reverse osmosis solution diffusion mechanism, and dialysis diffusion in convection free layer and electric charge of the particle and size in the case of electrodialysis. And microfiltration is used for sterile filtration and clarification.

So, sterile filtration, the separation of micro molecular solution that is nothing but ultrafiltration, separation of salt and micro solute from solution that is reverse osmosis, and separation of salt and micro solutes from micro molecular solution that is dialysis. Desalination of ionic solution is using electric field that is electrodialysis. Separation of gas mixture comes under the category of gas separation, but anyway we are going to concentrate only the first to five.

(Refer Slide Time: 33:43)

| Product | Application | Range | Module | Membrane | |
|------------------|-----------------------|------------------------|--------------|---------------|---------|
| Dairy ✓ | Milk concentration ✓ | RO | Spiral | CA, TFC | |
| | Whey concentration ✓ | RO | Spiral | CA, TFC | |
| | Whey fractionation ✓ | UF | Spiral | PS, PVDF, PES | |
| | Lactose concentration | RO ✓ | Spiral | CA, TFC | |
| | Milk pasteurization | MF | Tubular | Ceramic | |
| | Desalination | NF | Spiral | TFC | |
| | Clarification | MF | Tubular | Ceramic | |
| Juice ✓ | Clarification | MF | Hollow fiber | PS, PE | |
| | | UF | HF | PS | |
| | | MF/UF | Tubular | PVDF, PS | |
| | | RO/NF | Spiral | TFC | |
| | Concentration | RO/NF | Tubular | TFC | |
| | | RO/NF | HFF | TFC | |
| | | MF | Tubular | Ceramic | |
| | Gelation ✓ | Concentration | UF | Spiral | PS |
| | Corn sweetener ✓ | Dextrose clarification | MF | Spiral | PS/PES |
| | Sugar ✓ | Clarification | MF/UF | Tubular | Ceramic |
| Preconcentration | | RO | Spiral | TFC | |

Cellulose Acetate
Thin film composite
PS = polystyrene
PVDF = polyvinylidene fluoride
PE = polyethylene

So, this is another in terms of the application which industry, if your product is dairy which one you choose and if your product is juice which to be choose, and gelation and corn sweetener, sugar this also very minimal product only I have taken. You can further refer the reference and additional source I am going to give you at the end of lecture. The application is milk concentration, whey concentration, whey fractionation these we are going see today.

And lactose concentration milk pasteurization desalination everything comes under the dairy processing. The milk whey and whey concentration and fractionation, we used RO and ultrafiltration, the lactose concentration also RO, but milk pasteurization microfiltration will be used nanofiltration will be used desalination as well.

So here, whatever we have seen the different modules like spiral or tubular or hollow fiber or one more is platen frame we have seen but that is not being used here, most of the applications spiral membranes were used. And membrane here are the cellulose acetate membrane, which type of membrane we used cellulose acetate membrane, and TFC is nothing but thin film composites.

So, lot of research is also going on in the suitable membranes for the various applications, so this composite is now picking up the area, composite is nothing but instead of one single membrane, so we will have one membrane advantages as well as disadvantages. So to make this disadvantages into advantages, the another membrane is also sandwiched with this membrane to give the premium quality, so that kind of thing is this thin film composite.

So then polysulfone this is polyester, PE is nothing but polyester PVDF is nothing but, so polyvinylidene diester, then ceramic membrane you know that is all. So, here gives the, which product and which application and which range of filtration technique with which module and membrane material I will be using it. So this we are going to see mostly today the milk concentration as well whey concentration.

(Refer Slide Time: 23:38)

Microfiltration


- Alternative for dead-end filtration and centrifugation *↓ energy*
- In the food industry, MF is extensively used for the clarification of cloudy fluids.
Ex: clarification of juices
- Membranes with nominal pore sizes on the order of 0.1–0.5 μm or less produce permeates that are practically free of microorganism cells.
- MF is being increasingly used for the purification of drinking water and of water for the production of soft beverages.
- Food fluids clarified by MF include clear fruit juices, wine, vinegar, beer, and sugar syrups.

So, now microfiltration, so the microfiltration is alternative for dead-end filtration as well as centrifugation, so here you need to use lot of energy, but that is avoided when we use microfiltration technique. In food industry microfiltration is extensively used for the clarification of cloudy fluids so that means clarification of juices is the best example. So, the membranes with nominal pore sizes on the order of 0.1 to 0.5 micrometer or less produce permeates that are practically free of microorganism cells.

This is what we are told the cells and debris are retained in the microfiltration operation. Microfiltration is being increasingly used for purification of drinking water, and of water for the production of soft beverages, so here we need the ultra-pure water so there microfiltration technique is used. Food fluids clarified by microfiltration include clear fruit juices, wine, vinegar, beer and sugar syrups. So, the clarification operation can be done in fruit juices, wine, vinegar, beer and sugar syrups as well.

(Refer Slide Time: 38:09)

Microfiltration

- MF frequently serves for the pre-treatment of fluids before UF or RO. 
- Removal of suspended particles and colloidal material by MF is essential for reducing the rate of fouling in subsequent UF or RO steps.
- Oil droplets and particles of fat (e.g., in whey) are also removed by MF. Membranes made of hydrophobic polymers, such as polyvinylidene fluoride, are particularly suitable for that application.
- Brines used in the manufacture of cheese and in fish processing contain suspended solids and fats, which must be removed before recycling or disposal of the fluid.
- Other wastewater streams generated in food processing often require prefiltration before further treatment.

Microfiltration frequently serves for the pre-treatment of fluids before ultra and RO. So here see the membrane process is nothing but a filtration process, so instead of directly going to ultra or RO here the pore size used is 0.1 to 0.5 micrometer. So, more than that, whatever the bigger size molecule gets retained in the microfiltration techniques so that is what it serves us the pre-treatment for the fluid before ultrafiltration and reverse osmosis.

So, for example, so instead of directly going for ultrafiltration, so if I do microfiltration and go for microfiltration my cells and debris get eliminated in the microfiltration itself. So, it is a pre-treatment for ultra and RO and removal of suspended particles and colloidal material by microfiltration is essential for reducing the rate fouling in subsequent ultrafiltration or reverse osmosis system.

So, as I told just now when you do this microfiltration operation unnecessary this large size molecule get eliminated here, but if it is goes directly to ultrafiltration, unnecessary fouling or it gets clogged in the membrane surface. So, that can be avoided if we used microfiltration before ultrafiltration or reverse osmosis. Oil droplets and particles of fat that is example is whey are also removed by microfiltration.

The membranes made up of hydrophobic polymers such as polyvinylidene fluoride are particularly suitable for that application, which application, oil droplets and particulates of fats. So, when we wanted to remove this so we suppose to used hydrophobic polymers, so

which is nothing but polyvinylidene fluoride, so that as membrane we can use in the microfiltration technique.

And brines used in the manufacture of cheese and fish processing containing suspended solids and fats which must be removed before recycling or disposal of the fluid. So, this is not only used for the direct food processing but these techniques are used for the waste material which comes out of the food industry, because they have high biological oxygen BOD so the waste disposal it also contribute so that is the one example here.

Brines used in the manufacture of cheese and in fish processing contain suspended solids and fats which must be removed before recycling or disposal of the fluid, so before going for disposal so that should be removed, what is that, that brines. The suspended solids in the brines which is used for manufacture of cheese and fish processing. Other waste what is generated in food processing industry often require pre-filtration before further treatment as well as the disposal, there also microfiltration is used.

(Refer Slide Time: 41:13)

Ultrafiltration

- Dairy applications:
 - ✓ UF has become a major separation process in the dairy industry ✓
 - ✓ The two largest areas of application are the preconcentration of milk for cheese manufacture and the production of protein concentrates from whey. Both applications are related to cheese-making. ✓
- Whole cow's milk contains
 - ✓ 3.5% protein ✓
 - ✓ 4% fat ✓
 - ✓ 5% lactose ✓
 - ✓ 0.7% inorganic salts (ash) ✓
 - ✓ 87% is water ✓

| Product | Application | Range | Module | Membrane |
|------------------|------------------------|-------|--------------|---------------|
| Dairy ✓ | Milk concentration ✓ | RO | Spiral | CA, TFC |
| | Whey concentration ✓ | RO | Spiral | CA, TFC |
| | Whey fractionation ✓ | UF | Spiral | PS, PVDF, PES |
| | Lactose concentration | RO ✓ | Spiral | CA, TFC |
| | Milk pasteurization | MF | Tubular | Ceramic |
| | Desalination | NF | Spiral | TFC |
| Juice ✓ | Clarification | MF | Tubular | Ceramic ✓ |
| | | MF | Hollow fiber | PS, PE |
| | | UF | HF | PS |
| | | MF/UF | Tubular | PVDF, PS |
| | Concentration | RO/NF | Spiral | TFC |
| | | RO/NF | Tubular | TFC |
| | | RO/NF | HFF | TFC |
| | Caustic recovery | MF | Tubular | Ceramic |
| Gelation ✓ | Concentration | UF | Spiral | PS |
| Corn sweetener ✓ | Dextrose clarification | MF | Spiral | PS/PES |
| Sugar ✓ | Clarification | MF/UF | Tubular | Ceramic |
| | Preconcentration | RO | Spiral | TFC |

← Cellulose Acetate
 thin film composite
 PS = polystyrene
 PVDF = polyvinylidene difluoride
 PE = polyethylene

The next is ultrafiltration, so ultrafiltration mostly in the dairy applications, so UF has become a major separation process in the dairy industry so this is very much important, so if you see here also so the ultrafiltration, whey fractionation so this also whey concentration also we used ultrafiltration membrane. So, most of the application goes to dairy industry.

The two largest areas of application are the pre-concentration of milk for cheese manufacture as well as the protection of protein concentrate from whey, so this is a major two applications we are going to see for the ultrafiltration. The both applications are related to cheese making processes only. So, if we see raw cow milk, I think we have discussed this in aseptic processing as well I guess, the 3.5 percentage protein and 4 percentage fat and 5 percentage lactose, 0.7 percentage inorganic salts and remaining 87 percentage is water, the raw cow milk composition.

(Refer Slide Time: 42:26)

Cheese Making Process

- In the traditional cheese making process, milk, usually after adjustment of the fat content and pasteurization, is coagulated through a combination of lactic acid fermentation and enzymatic reactions.
- After cutting and temperature adjustment, the coagulated mass separates into solid particles (curd) suspended in liquid (whey).
- The curd contains the major milk protein-casein and most of the fat.
- The whey is essentially a dilute aqueous solution of lactose, mineral salts, and noncasein milk proteins (e.g., lactalbumins and lactoglobulins).
- The curd is subjected to various methods of treatment and curing, resulting in the vast variety of different cheeses.

And in the traditional cheese making process, what happens is? The milk usually after adjustment of the fat content if any adjustment needed and after pasteurize, it is coagulated through combination of lactic acid fermentation or enzyme reaction. So, any one of the technique it is coagulated. After cutting and temperature adjustment the coagulated mass separates into solid particles which is called curd and suspended liquid which is called whey. So, the curd contains major milk protein-casein and most of the fat the curd, the curd part contains the protein-casein as well as the fat part.

The whey is essentially a dilute aqueous solution lactose minerals salts and non-casein milk portions that is nothing but lactalbumins and lactoglobulins, so these are milk proteins in the whey. And the curd is subjected to various methods of treatment like cutting resulting in the vast variety of different cheeses. And the whey whatever is left out that is mostly used for the further processing or most of the time process a waste that is where I told but the treatment process would be little bit expensive because it has high biological oxygen demand.

So, this is the traditional cheese making, after the pasteurization of milk we will coagulate using any of the lactic acid fermentation technique or enzymatic reaction then two things are separated curd and whey. Curd is containing milk protein-casein and fat and whey contains aqueous solution of lactose minerals salts and non-casein milk proteins so the curd is further process cheeses and whey is thrown out or sometimes used for further treatment as well.

(Refer Slide Time: 44:24)

Cheese Making Process

- UF of milk retains the fat globules and the proteins. The inorganic salts and the lactose, along with some of the water, are partially removed as permeate
- The resulting retentate is a partially concentrated milk with reduced lactose and mineral content. The concentration ratio is often three- to fivefold.
- If further reduction of lactose and mineral content is desired, the retentate is diluted with water and ultrafiltered once again (diafiltration)
- The pre-concentrated milk is mainly used in the manufacture of many types of cheeses (most commonly soft cheeses).

But in ultrafiltration, how we do this? Ultrafiltration milk retains the fat globules and the proteins in the inorganic salts and the lactose along with some of the water but partially removed as permeate. So the same as that of traditional cheese making process. But the difference is, the resulting retentate is partially concentrated milk with reduced lactose and mineral content and also the concentration ratio is often three to fivefold.

So, that is the advantage. So further reduction of lactose or mineral contain is desired then the retentate is diluted with water again and ultrfiltrate once again. So this we call it as diafiltration. And the pre-concentrated milk is mainly used in the manufacture of many types of cheeses, so the concentrated milk whatever we got here so that is used in the cheese making process.

(Refer Slide Time: 45:45)

Advantages in Cheese Making by UF

- Increased yield, probably because of the inclusion of some of the noncasein proteins in the curd ✓
- Lower energy consumption ✓
- Reduced volume of whey ✓

So, the advantage with respect to traditional making is the increasing yield probably because of the inclusion some of the noncasein proteins in the curd, so here we told in the traditional cheese making non-casein milk proteins goes into whey. But when we use the ultrafiltration, so this also gets with the curd part itself and low energy consumption compared to normal cheese making process and reduced volume of whey that is what I told. So here almost 90 percentage goes as a waste in the traditional cheese making process, but that volume gets decrease when we use the ultrafiltration.

(Refer Slide Time: 46:10)

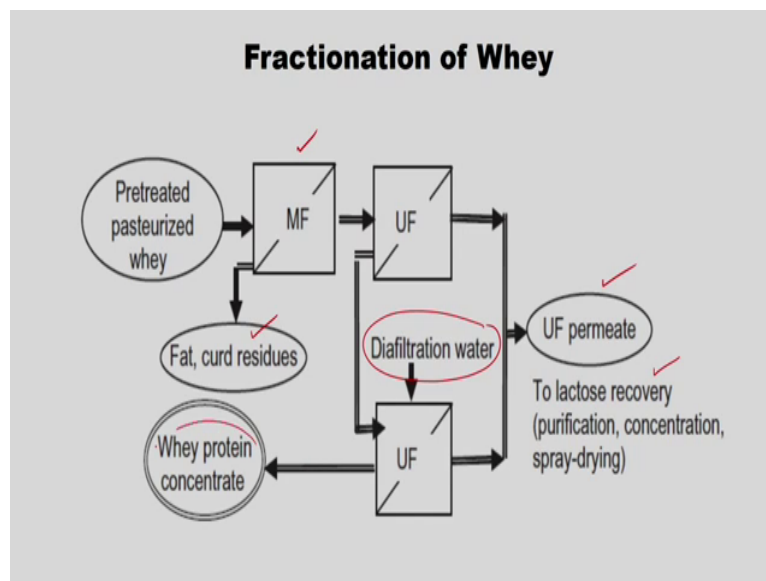
Fractionation of Whey

- Whey contains the major part of the noncasein proteins, together with low-molecular weight solutes (lactose, mineral salts).
- UF of whey produces a valuable whey protein concentrate as the retentate and a protein free permeate containing mainly lactose and minerals.
- The retentate is usually concentrated further by evaporation and spray-dried.
- Whey proteins find extensive use in the manufacture of cheese and a considerable array of food products and health food specialities

The fractionation of whey, this is another application which is done using ultrafiltration. The whey contains the major part of the noncasein proteins, so the curd is taken and it goes for further cheese making process. The removed whey further we used fractionation and technique using ultrafiltration membrane. So whey contains the major part of noncasein proteins together with low molecular solute which include lactose as well as mineral salts.

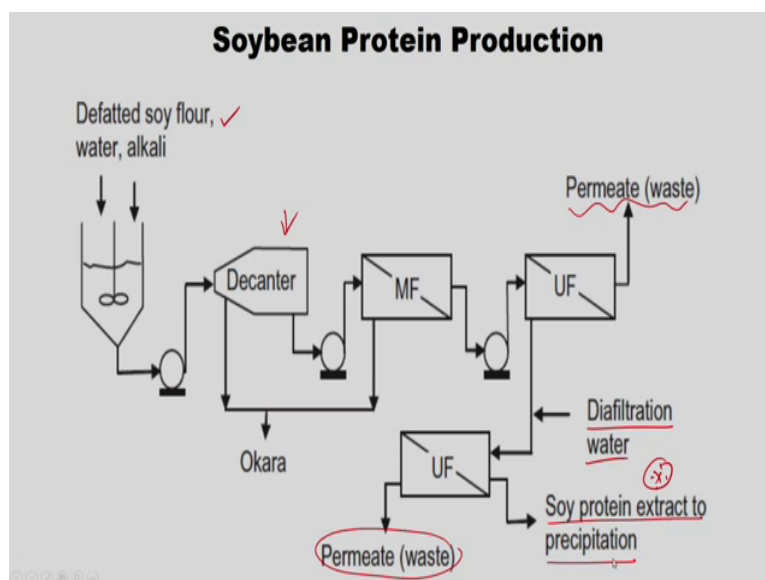
So, ultrafiltration of whey produces the valuable whey protein concentrate as the retentate and protein free permeate containing mainly lactose as well as minerals. The retentate is usually concentrated further by evaporation or spray-drying. And the whey proteins find extensive use in the manufacturing of cheese and a considerable array of food products in a health food specialist. So that means the whey protein further fractionating the whey using UF so we will get whey proteins. So that is further used for manufacturing of cheese as well.

(Refer Slide Time: 47:16)



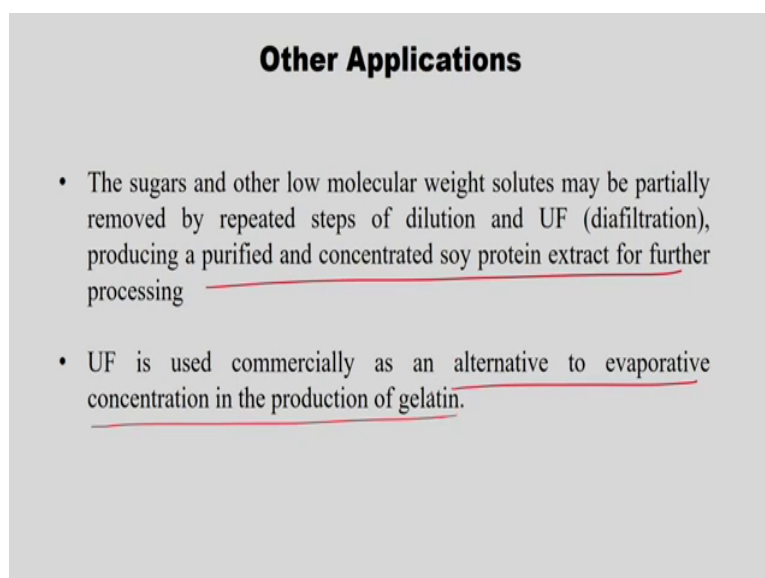
So, this is the flow chart where your microfiltration is first used here pretreated as well as pasteurize whey it is fed into the microfiltration, so which gives you fat curd residues and the whey further goes to ultrafiltration, so we use diafiltration water here if it needed second time it is ultrafilter. So, here what we get is ultrafilter permeate to lactose recovery it goes further purification concentration as well as spray-drying. So, whatever we get out of the ultrafiltration is nothing but whey protein concentrate.

(Refer Slide Time: 49:23)



The flowsheet is here, so we have defatted soy flour with water and alkali, so that is pumped into decanter, based on the density it removes the concentrated part, so that is nothing but high density part, then it further pumped to the microfiltration, then further ultrafiltration, so here we get the permeate as a waste. And the further processed with the ultrafiltration membrane using diafiltration water, so here we get the soy protein extract to precipitation and the permeate goes as a waste. So this is the soy protein extract for further precipitation.

(Refer Slide Time: 50:04)



And also the other application also includes sugars and other low molecular weight solutes, maybe also partially removed by repeated steps of dilution and diafiltration which produces

purified as well as the soy protein extracts for further processing. And ultrafiltration also used commercially as an alternative to evaporative concentration in the production of gelatin, so this are also included in the ultrafiltration applications.

(Refer Slide Time: 50:49)

References and Additional Resources

- Fellows, P.J. 2000. Food Processing Technology-Principles and Practice. 2nd ed. Wood head Publishing, Cambridge.
- Richardson, P. (Editor). 2004. Improving the thermal processing of foods. CRC Press.
- Berk, Zeki. 2018. Food process Engineering and Technology. Academic press.

So, here I will stop today's lecture. Then we will discuss about two techniques which is nothing but Nanofiltration and reverse osmosis in next class. And these are all resources and additional references what I have used in this lecture. Thank you.