Membrane Technology
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Lecture No 6
Preparation of Synthetic Membrane, Phase Inversion Membranes

Good morning students today is lecture 6 of Module 2. In today's class,

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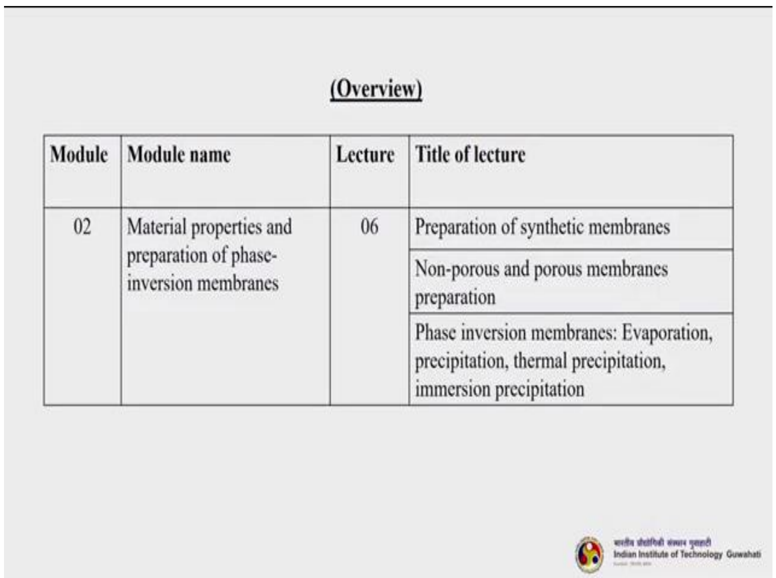
(Overview)

Module	Module name	Lecture	Title of lecture
02	Material properties and preparation of phase-inversion membranes	06	Preparation of synthetic membranes
			Non-porous and porous membranes preparation
			Phase inversion membranes: Evaporation, precipitation, thermal precipitation, immersion precipitation

We shall start discussing the preparation of various membranes will start from synthetic membranes of course we will discuss all the synthetic membranes, as I told you earlier is that biological me mbrane is out of our scope. So we will discuss non porous and porous membrane preparation different methods and. Today's focus will be mostly on phase-inversion membranes.

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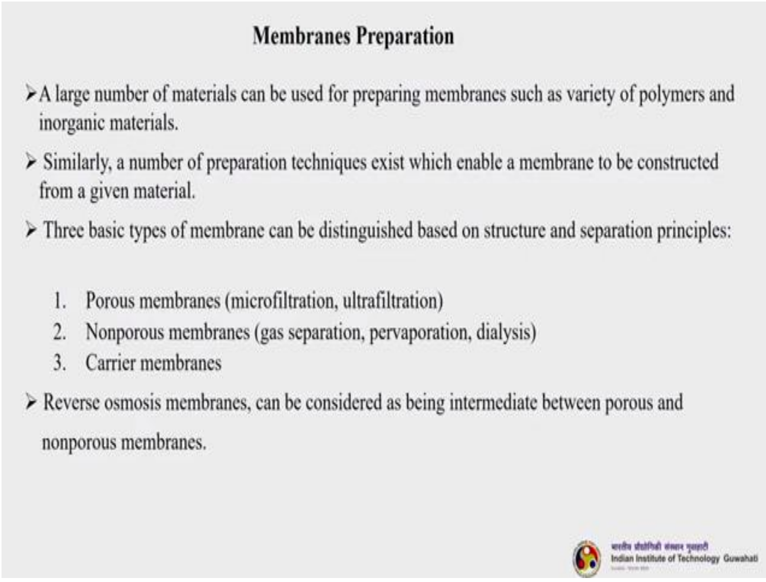

As you know, there are a large number of materials that are available for preparing a membrane we have discussed all such membrane materials, such as polymers and inorganic, materials, the different properties. And there are advantages and disadvantages of using these materials for membrane preparation. Similarly, there are so many different varieties or types of membrane preparation techniques are available,

Which can be used for preparing a particular membrane for a specific task. Now, based on the structural separation principle, we can divide the membranes into three classes. First is porous membrane which is of course our microfiltration, ultrafiltration. All these membranes, then we have non porous membranes, which are also called dense membranes. so those are used for gas separation, pervaporation,dialysis and all.

And then we have carrier membranes, carrier mediator membranes carrier membranes, which basically works on facilitated transport, in a nutshell, we have discussed this but in a, in our subsequent lecture will devote to one full class to discuss facilitated transport. So, RO membranes, actually it lies in between poros, and non porous membranes.

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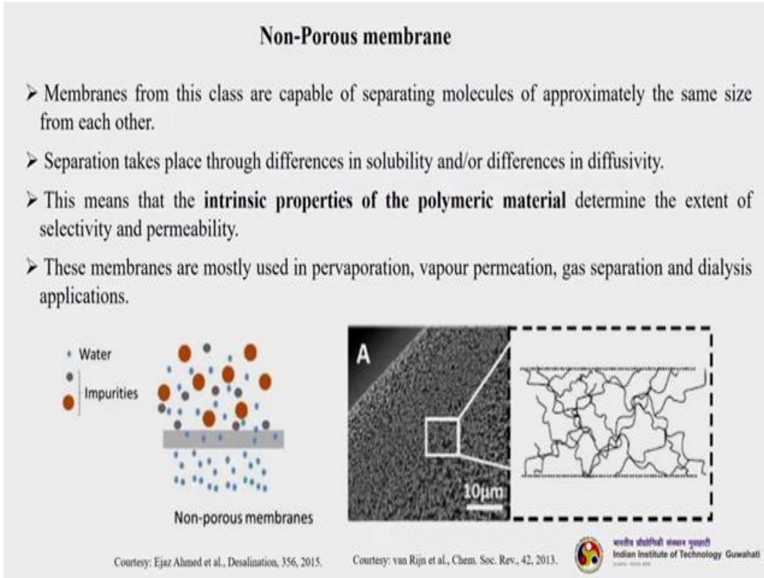

So porous membrane, the diamension of the pore mainly determines the separation, the type of membrane meterial being crucial importance for chemical thermal and mechanical stability, but the type of material is not at all contributing to the flux and overall rejection. So, membranes of this class actually induced seperation by discriminating between particle size. So, that is why these are called size based separation basically..

And such membranes are, microfiltration and ultrafiltration so we can obtain very high selectivities on the solute size or particle size is very, very large relative to the pore size, so all of them will be subsequently written, you can see how a micro porous membrane looks like so this is a polypropylene membrane, and this is image actually a PACM image, membranes of non porous nature will separate the molecules approximately the same size from each other.

That is the beauty of non porous or dense membranes. So actually separation is taking place, by virtue of the differences, either in diffusivity or solubility, so that precisely means the polymeric material of the membrane is determining the extent of selectivity and permeability. So, these membranes are mostly using in pervaporation application as well as gas separation and dialysis membranes, you can see a PACM image of a non porous or dense membrane. Here,

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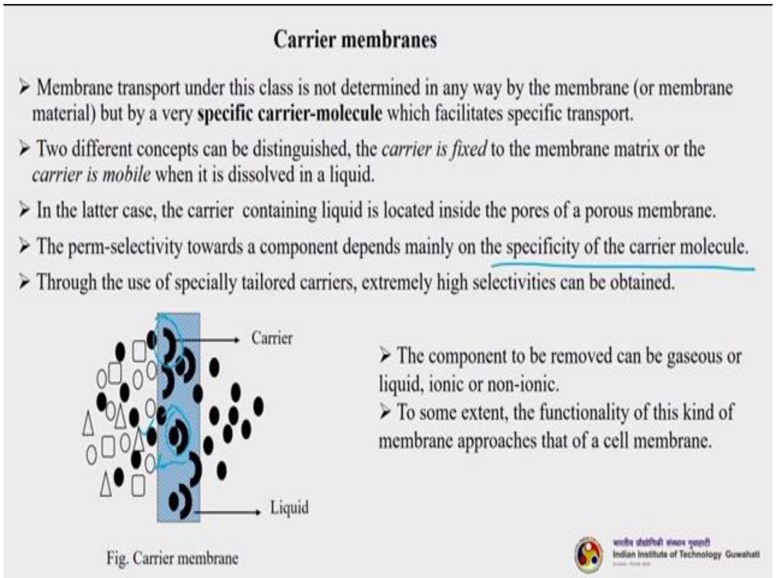

The next class of membrane is carrier membranes. So here, this is a specific class of membranes in which this transport is through facilitator mechanism. So here the membrane transport under this class is not determined by any way of the membrane material is not playing any role, who is helping in transport is basically the carrier molecule which is present inside the pores of the membrane.

So we can have two different mechanisms here. Either we can have a carrier that is fixed to the membrane Metrix or we can have a carrier which is mobile, When it is dissolved inside the liquid and that liquid is filled in the pores of the membranes. And the membrane itself, the solid membrane itself is providing a mechanical support for such purposes. The perm-selectivity towards the component depends mainly on the specificity of the carrier, a molecule.

So, What is the carrier molecule what is its nature that means its physical and chemical nature. So all these things will determine the overall selectivity so through the use of specific tailored carriers, extremely high selectivities can be obtained. The meaning of this sentence is basically for a particular target seperation, so I can choose a carrier molecule in such a way that I can get very high, selectivities, see, in this particular skech.

These are the carriers, okay, how they are helping and they are binding to this solute. This is another solute which I want to transport. So, This is a carrier and solute intermediate complex, when it is reaching the membrane surface the permeate side, then it is dissociating itself and the carrier is again back inside the membrane for press binding of the solute. Okay, so the component to be removed can be gaseous, liquid, ionic or non ionic, to some extent.

The functionality of this kind of membrane and approaches that have a cell membrane. So basically, most of the biological membranes. They work in such mechanisms.

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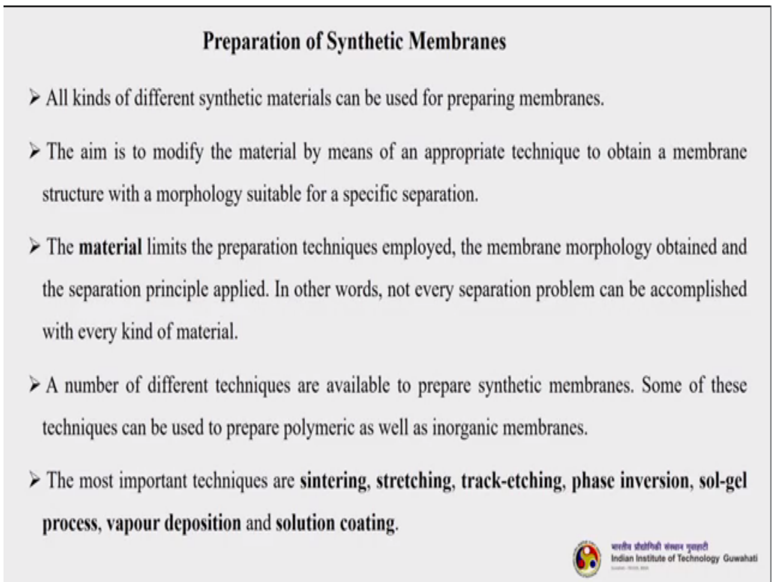

So let us now discuss preparation of the membranes. So all kinds of different synthetic materials can be used for prepairing membranes. Actually, the aim is to modify the memory by means of an appropriate technique to obtain the memory structure with a particular morphology, which will suit a particular seperation. So the material actually which the preparation technique involves so what is the meaning of this?

Meaning is that I cannot use any material for any preparation technique, let us say there is a particular technique called sintering, I am just giving an example. So I cannot use any type of material for sintering though most of the polymer ceramics are used, but there are restriction Similarly, there are restrictions for track-etching there are restrictions for interpersonal polymerization okay.

So, number of different techniques are available to prepare synthetic membranes. Some of these techniques can be used to prepare polymeric as well as inorganic membranes so that means, some of the techniques can be used for both polymeric and inorganic metals. However, there are a few techniques which are exclusively made for either polymeric or either ceramic membranes or inorganic membranes, So we will see one by one today.

The different techniques to prepare membrane. So, some of them are sintering, stretching, track-etching, phase inversion,sol-gel, vapor deposition and solution coating. So today we will discuss few of them.

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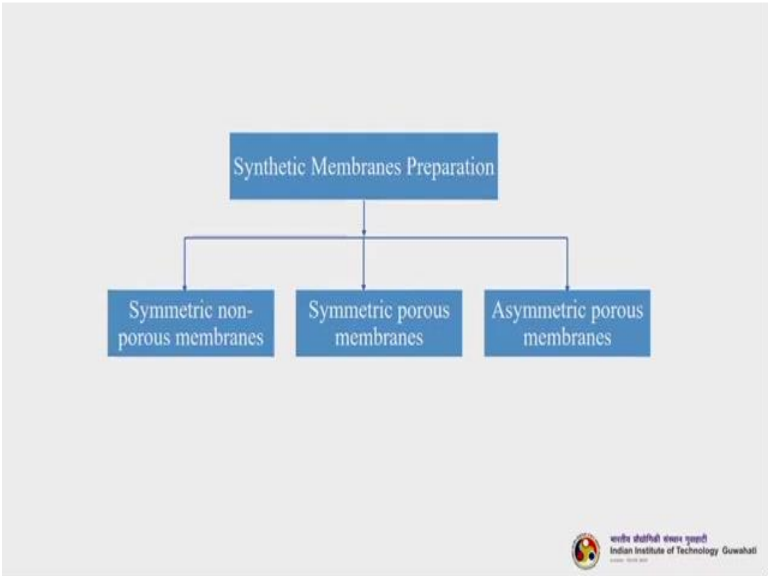

So first we will discuss the symmetric non porous membranes. Okay then followed by symmetric porous membranes, and then we will discuss the asymmetric porous membranes which are basically the dense membranes.

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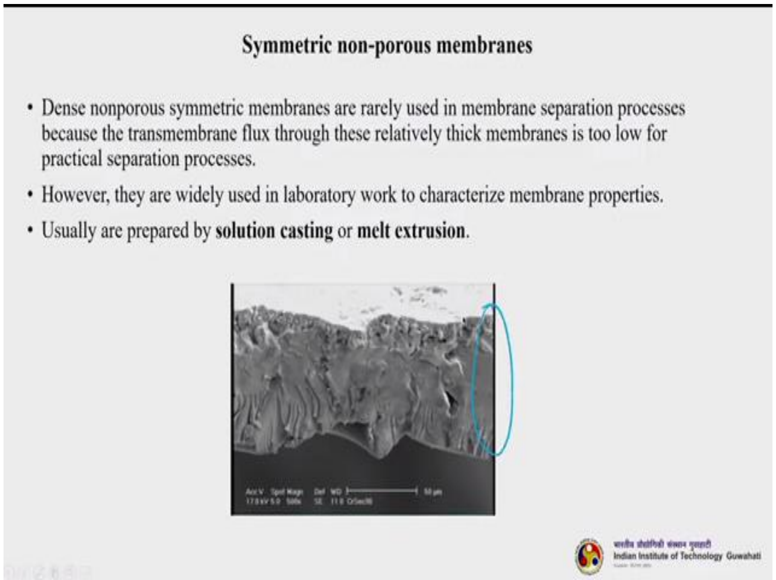

Okay, so let us discuss the symmetric non porous membranes, or dense membranes, so dense non porous symmetric membranes are rarely used in membrane separation processes, because the flux through these relatively thick, they are very thick membranes. Okay, it is too low for practical seperation purposes in one class if you remembr as I told you earlier that the membrane thickness should be as much as less as possible.

Otherwise the membrane material, and its thickness is providing additional resistance to the seperation, so it will be very thin then the resistance will be less, it is very very thick then the results will be more. So ultimately the separation will be low, so this is very important. And that is why these non porous membranes which are usually thick than other porous membranes. Okay, they are not giving high flux and the selectivity is also less.

So how about their widely used in laboratory work to characterize membrane properties. So there are two simple way we're discussing there are many other ways also. The first one is called solution casting. And the second one is called melt extrusion. So you can see actually how thick actually a dense membrane or this one, non porous membrane is.

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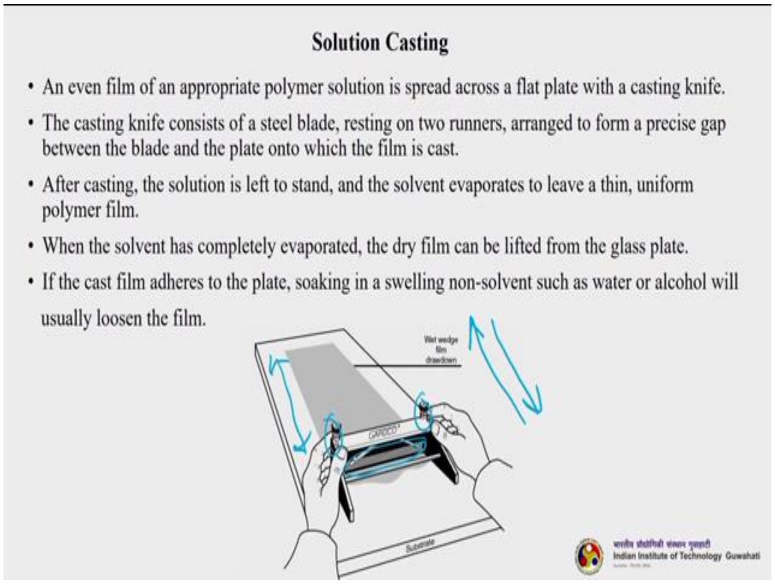

So solution casting is one of the most easiest way, it is very good way to do actually. And we can do it in a large scale. So, what is being done in this, film of a appropriate polymer solution is spread across a flat plate, with a casting knife please look at the figure now. So, this is actually the casting knife. Okay. So, and this is the film. Right. So the casting knife consist of a steel blade resting on two runners.

Okay, so this is one runner, this is another runner. So basically, moves, it moves, we can move it in this direction, I can again further move it in this direction. So, the knife consists of a steel blade resting or two runners, adheres to form a precise gap between the blade and the plate onto filim is cast. And what is the role of knife, so I can adjust the line height in such a way to deserve to obtain a desired thickness of the membrane.

And after casting the solution is left to stand for some time. Okay. The reason is that let the solvent evaporates, okay when the solvent evaporates, then it will a thin uniform polymer film will be precipitated and it will be collected. Now, once all the solvent evaporated the film becomes dry we can take it up. Now, it happened many times, the film. After the solvent evaporation is become very sticky to the surface of the blade.

So that time we have to use a swelling and non solvent usually water or alchohol that will easily lose the film so that I can take the film, without disrupting it structure further.

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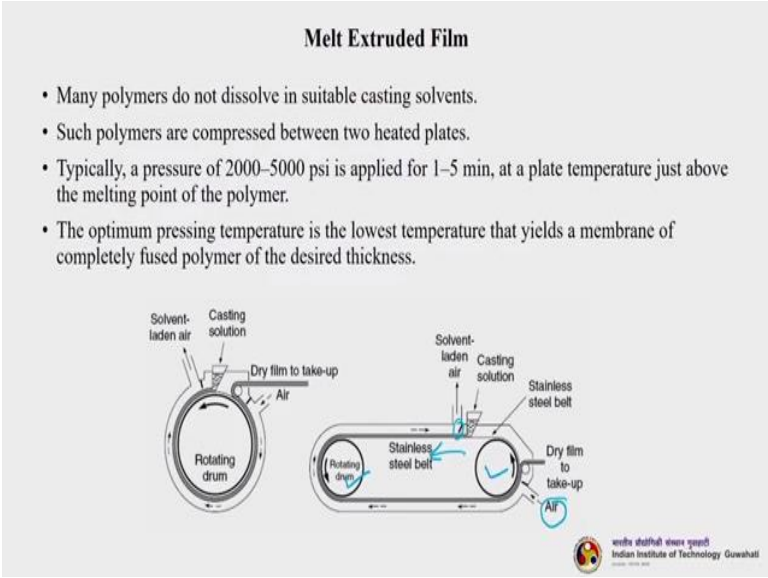

So the next one is actually melt extruded film. So this is exclusively used for certain polymers, which do not dissolve in some suitable casting solvents, like, a nylon. Okay. Such polymers accomplished between two heated plates, there are two heated gloves, under which this polymer sir. Actually compressed and so typically a pressure very high pressure is applied easily 2000 to 5000 psi pressure is applied for very small time is really 1to 5 minute.

Okay, at a plate temperature, just above the melting point of the polymer, not many times what happens if the polymers, which I want to use for this non porous membrane preparation is available in the forms of bits small bits, then we need to decrease the particle size so we can go for some pressing to decrease the particle size or grinding. Okay, then we can put it under the Blitz.

So the optimum pressing temperature is the lowest temperature that yields a membrane and not completely fused polymer of the desert thickness. So basically, we want temperature pressure in such a way that the entire polymer material which is supplied between these two plates, has been melted and resulted in a desired thickness. So you can see this is actually a melt extrusion system in which there are two drums.

One is here and one is rotating. Okay, in this direction, and the casting solution is pored here, you can see a small knife type of arrangement here. Okay, which is just like the blade which we discussed the earliest the slide, okay that distributes the thickness of the membrane. Okay, and air is being pushed from the bottom here. Okay, and the air will actually help in evaporating the solvent.

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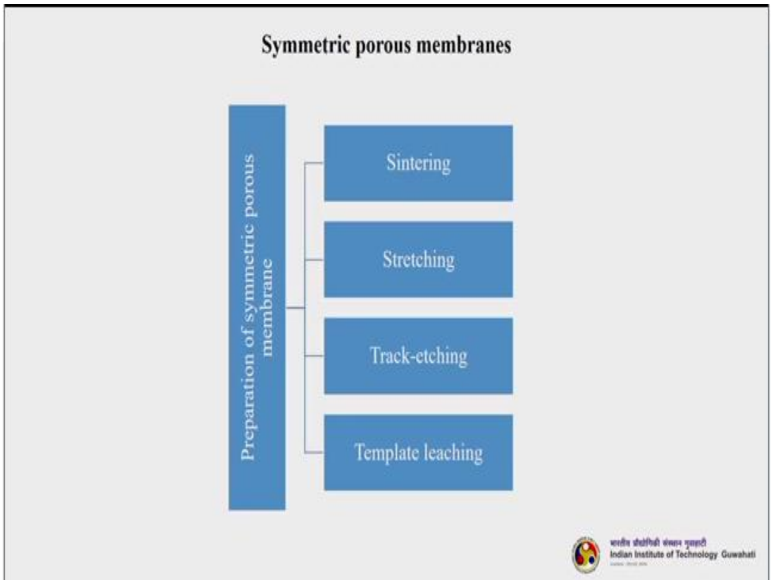

So the next is symmetric porous membrane. So we will discuss four different types of preparation techniques here. The first one is centering then stretching, then track-etching and then template leaching. So these are the present techniques for symmetric porous membranes.

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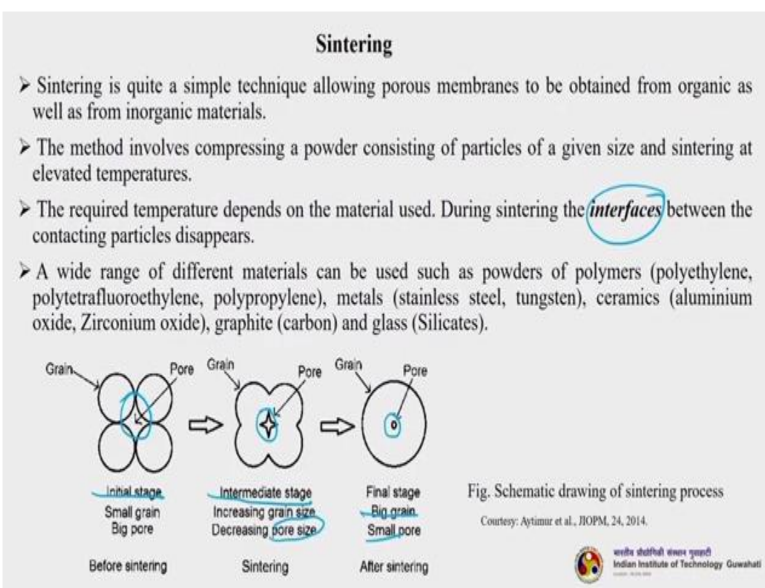

So sintering is a very old is metallurgical process actually it is. So it's a quite simple technique, allowing porous membraness to be obtained from organic as well as inorganic material so the beauty of this particular technique is that I can use both polymers as well as inorganic materials. The method involves compressing a powder consisting of particles of the given size and sintering it at elevated temperature.

Here sintering means you are putting them under very high elevated temperature in atmosphere of a very high elevated temperature and the required temperature depends on the materials to be used what temperature you are going to set, actually, that depends upon the material which you are using. Okay, there is no thumb rule for this. So during the sintering the interfaces between the contracting particles disappear.

The interface is not phases Okay, so please see this figure. You can see I have only four different granis. Okay. This is the first. This is the initial stage here the granes are small and the pore, okay whatever it is available here. They interspersed between these pores. These pore granes. Okay that is actually big, and in the intermediate stage, what is happening. So the grain size increases so the grains.

This grains, they are fusing together, right, and the fuse together, the gravesize increases however the pore says decrease as you can see, the pore sizes become small. Okay. Then in the final steps, the grain sides remains. We get a big grain. Okay, and we get a small, very small pore. This is how actually the pores are developed using a sintering process so wide range of different materials can be used, such as powders of polymers. Okay, metals, ceramics and even glass silicates.

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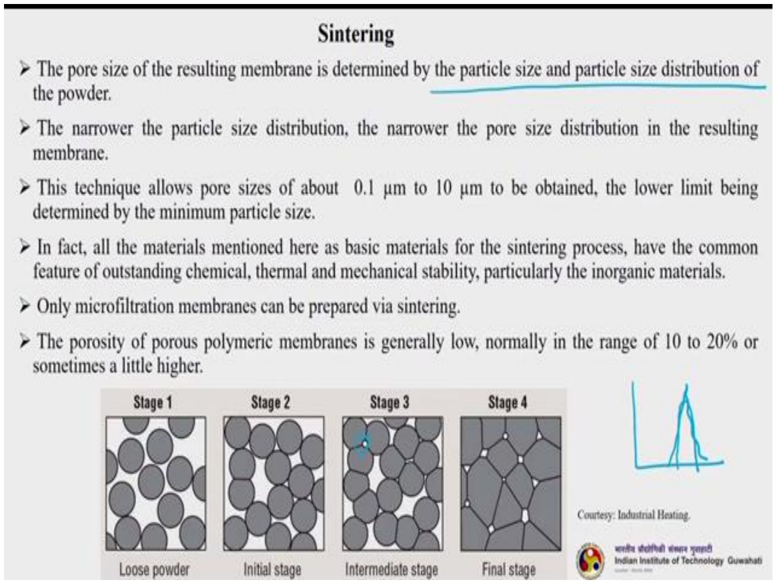

So this is, this scheme is actually interesting and will be more clear. So, we will discuss this see let us see the pore size of the resulting material actually a membrane is determined on the particle size and particle size distribution of the powder so please remembrane this is very important. So the pore size is determined by the particle size and particle size distribution of the powder powder material, your material which from which you're going to make your membrane.

So narrowed the particle size about distribution. The narrowed the pore size distribution in the resulting membrane. So, if we get a very, very narrow particle size distribution something like this. Okay, then we will also get a very narrow membrane pore size distribution. So this technique allows a pore size of about point one micron to 10 micron. Okay. And all the materials mentioned here are basic materials for sintering process.

Have the common picture of outstanding chemical thermal and mechanical stability, particularly the inorganic materials, since we are talking about a very high elevated temperature process. So, the materials that are supposed to be used for this process should have very good thermal and mechanical stability. So, only one problem is this we can only make microfiltration membranes out of this.

It is very difficult to make ultrafiltration or nanofiltration range. So, it is not possible, basically. So the porosity of the porous polimeric membrane is generally low. Normally in the range of 10 to 20%, or sometimes a little higher also. Now we have little advanced techniques. So you can see how nicely this particular scheme, make you understand of the sintering process, usually initially the powder will be loose.

And then there becomes the close, that come close together. Okay, and then intermedius as you can see small small, the interfaces is growing. Okay, sorry, disappearing. And then, when most of the grains are fused together. Okay, we get the pores of different sizes.

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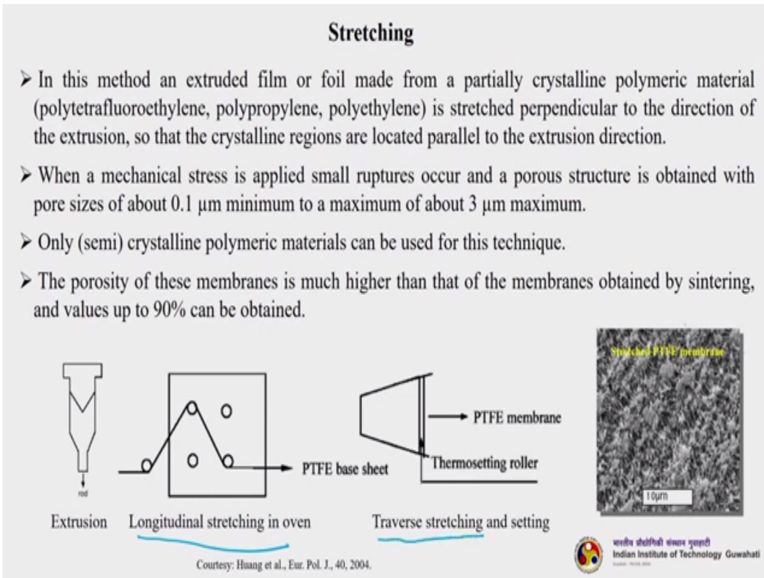

So the next one is stretching. So in this method and extruded film or foil made from a partial crystaline and polymeric material usually polytetrafluoroethylene PTFE, polypropylene PP or polyethylene is strech perpendicular to the direction of the extrusion so that the crystal regions are located parallel to the extrusion direction. Basically, there is a film, which is casted. Okay.

And you are supposed to strech that streching can be done by various methods. Okay, so now you're applying a mechanical stress, basically, right, what is the intention, to create cracks or ruptures. Okay. So, when you create a crack or ruptures by giving mechanical stress, the later on you have to go for a process in which you are going to wash this membranes, so that whatever. These loose or adhered particular set attached to the surface of pores.

Will get washed away and the pores becomes clear So only semi crystalline polymeric materials can be used for this technique, the porosity of these membranes is much, much higher than that of the that often by sintering and almost we can get up to 90% porous, so you can have a longitudinal stretching. Inside a oven, or you can have a traverse stretching and setting and this particular same image is for a stretched polytetrafluoroethylene membrane.

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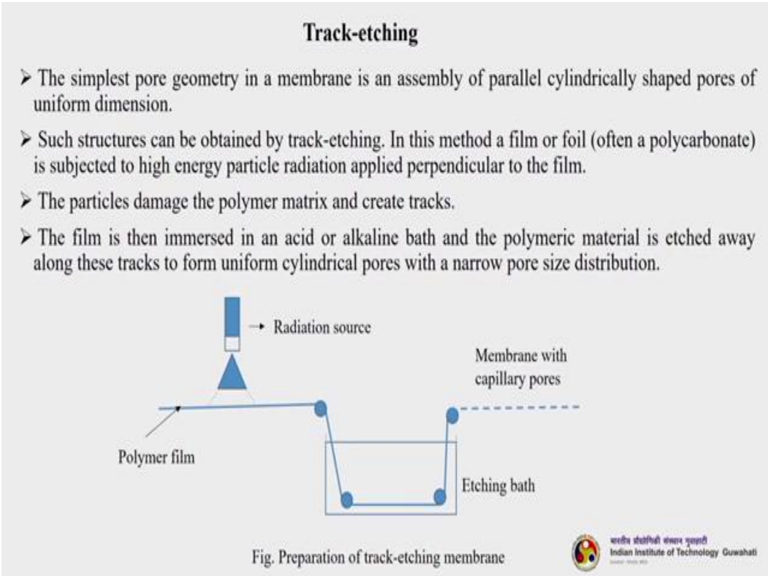

So the next method is track-etching. These are old techniques but are also used nowadays for making commercial membranes. So the simplest pore geometry in a membrane is an assembly of parallel cylindrically shaped pores of uniform diamensions, we get very nice pore structure in this method. Okay, such structures can be altered by track-etching. In this method, a filim which is already casted actually, when I am talking about that a film.

Okay, that means a polymer, which is already dissolved in the solvent. Okay. And then it is casted in the film. So now that film is subjected to high energy particle radiation. Okay, applied perpendicular to the film. So what this radiation is doing, the radiation is damaging the polymer matrix, basically by creating tracks, or cracks whatever you can call them. So, in the earlier slide, we discussed about stretching, that the streching was done by mechanical here actually.

We are not doing streching, we are creating the same tracks or cracks by radiation. So the film is then immersed in a acid or alkaline bath and the polymeric material, it is their way along this stretch to permeate uniform cylindrical pores with narrow pores as distribution. So you get a very nice uniform for cylindrical pores, with the narrow pore size distribution.

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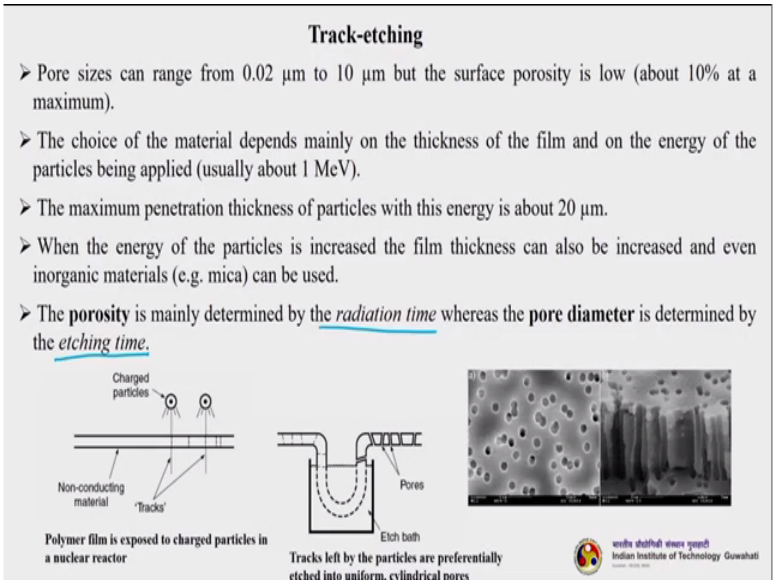

So, usally pore sizes of 0.2-10 micron are often however the surface porosity is low, about 10%. The choice of polymeric material depends mainly on the thickness of the film, and on the energy of the particles, that is being applied okay how much radiation, you are applying actually. So the maximum penetration thickness of the particles with this energy is about usually 20 micron. Right?

When the energy of the particles is increased the film thickness, can also be increased. And even inorganic materials can be used, usually polymeric materials are used. However, in recent some advancements have taken place where they have used subs inorganic materials such as for example mica. Okay, so the porosity is mainly determined by the radius and time. Okay, whereas pore diameter is determined by that etching time.

Okay, so, etching is done to remove whatever the loose adhere particles that is attached to the track. Okay, then the posts will be cleaned.

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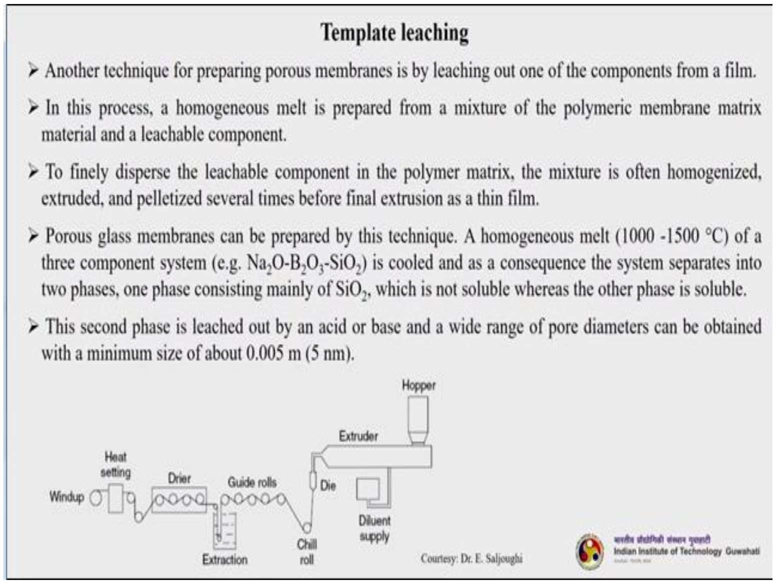

Next method is template leaching. So, this is a another technique for preparing porous membrane by the name is template leaching, so what has been done here actually. We are leaching out one of the component from a film. Okay, so in this process inhomogeneous melt actually is prepared from the mixture of polymeric membrane matrix, material, and a leachable component. So the leachable component is already mixed while we are making a film. Right?

So to finely disperse the leachable component in the polymeric matich the mixture is open homogenized extruded pelletize several times we put the final extrusion as a thin film. You can see the schematic actually. So one example is porous glass membraness, so you can prepare porous glass membranes by this technique, so he prepared a homogeneous melt at a temperature of usally 1000- 1500 degree centigrade and figure.

Okay, of a three component system. So, this is your sodium oxide, barium trackside and silicon dioxide. Okay, this is a three component system. It is coold and as a consequence, the system separates into two phases. So what are these two phases. So the one phase containing mainly silicon dioxide. Okay, so that is not soluble right other phase is soluble. Now this second phase is leached out by an acid or base.

And a wide range of pore diameters can be obtained with a minimum size of about 0.005 meter. So basically what is happening when the leaching is happening. So this leaching is creating the pores. But anyway, again we wants to leaching processes. Over the film will be subjected to certain washing techniques, or you can again put them to some bath, in which using some solvent, or non solvent.

You can wash out the loosely adhered particles which are there inside the pores, basically are cleaning the pores.

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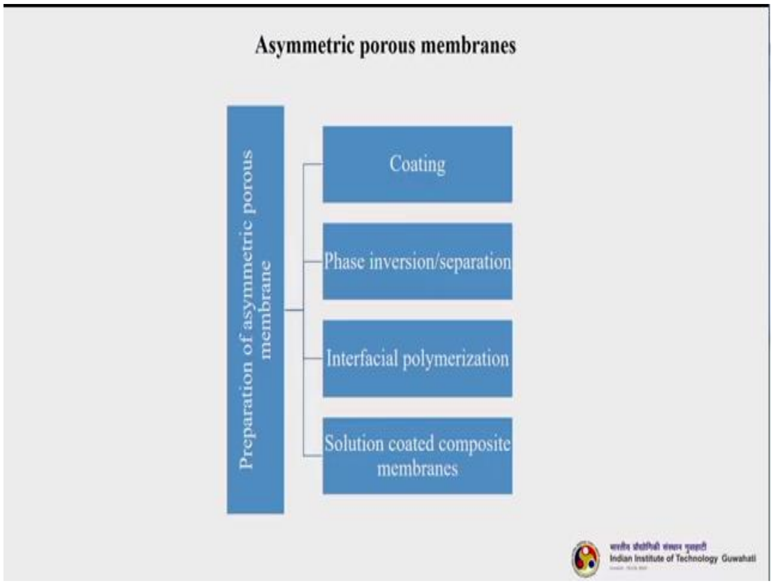

So the next one is asymmetric porous membranes, under this will discuss various processes, such as coating, phase inversion or separation, interfacial polymerization and solution coated composite membranes. So today will restrict our discussion to coating and phase inversion. Next methods will discuss in our next lecture.

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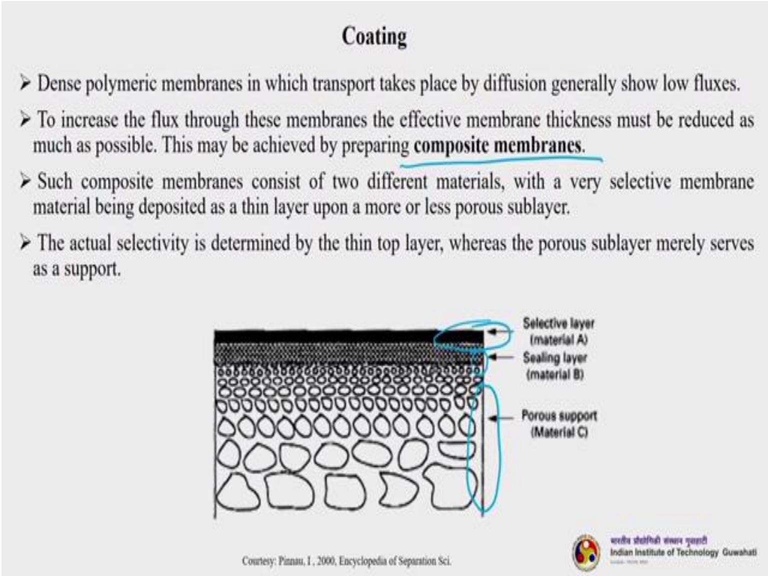

So, usually we can prepare dense polymeric membranes in which transport takes place by diffusion generally show low fluxes. Okay, to increase the flux through these membranes that effective membrane thickness must be reduced as much as possible, so that is what I was telling few minutes we put also, but this may be achieved by preparing competent membranes. So, competent membranes or as symmetric membranes right?

So such competent membranes consists of two different materials with a very selective membrane material being deposited as a thin layer. Okay, on a porous clear said this is a thin layer selective layer. Okay, which is actually doing the seperation, then we can have another sealing layer here. Okay, we are having very very low pore size distribution, then another layer here. Okay, which is highly micro porous, so that is providing your support.

So, the actual selectivities determine the top layer. Where as the porous sub layer serving the support, support, it is providing mechanical support.

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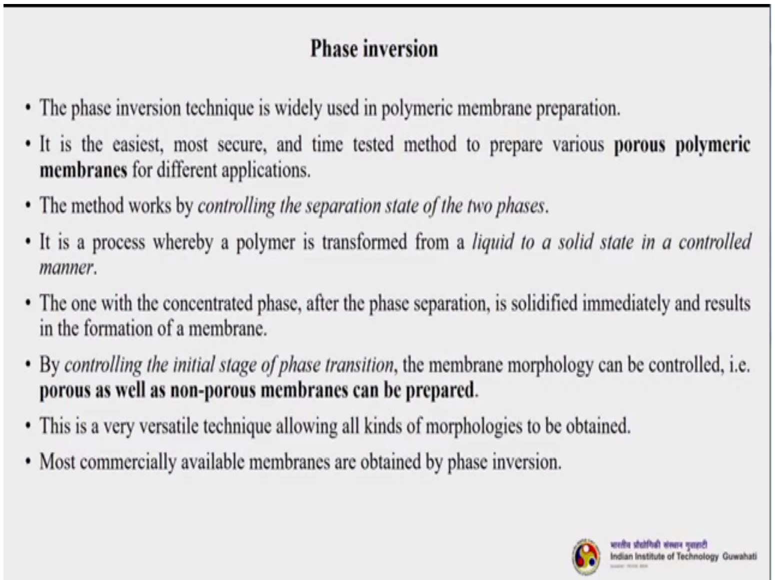

So several coating procedures can be used, such as deep coating, plasma polymerization, interfacial polymerization, and in-situ polymerization of these membranes. Another type of coating is also possible where the coating layer plugs the pores in the sub layer. So in this case actually properties of the sub layer, rather than those of the coating layer mainly determine the over all properties.

So this is a different technique with sintering stitching leaching out and track-etching techniques only porous membranes can be obtained, whatever we have discussed today. So these membranes can also be used as a sub layer for composize membrane so that their application can be extended to other areas. So through the use of phase inversion technique, it is possible to obtain open as well as dense structures so phase inversion is one of the most important process.

Okay, that technique can be used to obtain both open means pores, as well as dense membranes coating techniques are normally used to prepare thin but dense structures, possessing a high selectivity and relatively high flux. So the basic support material for all composite membrane is often an asymmetric membrane obtained by phase inversion. So today we will discuss about phase inversion in detail.

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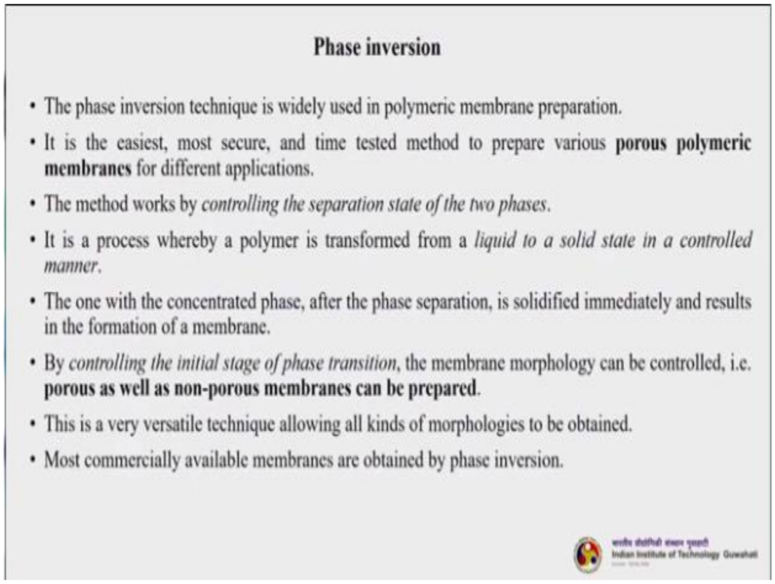

So phase inversion is widely used for polymeric membrane and preparations. So it is the easiest, most secure and time tested method to prepare various poros polymeric membranes for different applications. So, the method works by controlling the separation states of the two phases. Right. It is a process in which the polymer actually is transferred from a liquid state to a solid state.

Okay, under a controlled manner or atmosphere. So the one with the concentrated press after the phase inversion is actually solidified immediately and results in the formation of the membrane, and by controlling the initial stages of the phase transition, the membrane morphology can be controlled. That is how we can either have a porous membrane, or we can also prepare non porous membrane, using this particular technique.

So this is a very versatile technique, allowing all kinds of morphologies to be obtained, and most commercial membranes are obtained by phase inversion techniques.

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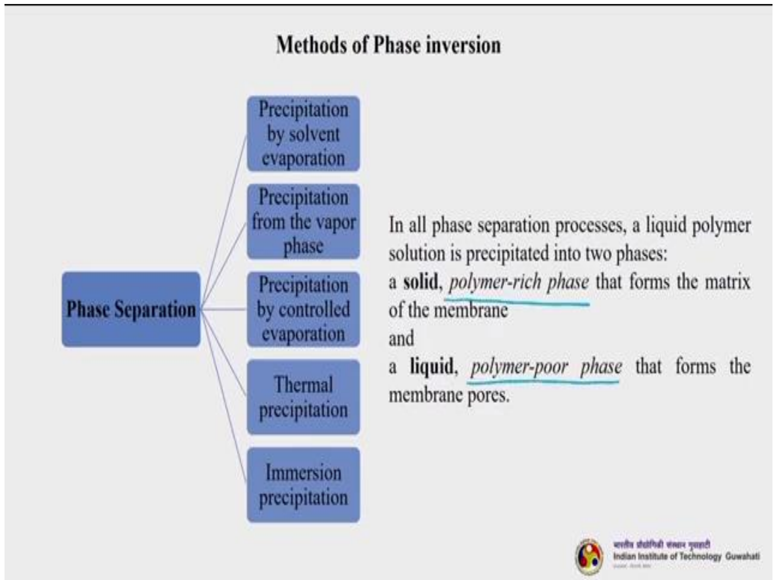

So, let us see what are the different types of phase inversion, in many books you will see in the writing phase separation this is the same basically. Okay, so we shall have precipitation by solvent evaporation, we can have precipitation from the vapor phase, we can have precipitation by controlled evaporation, then we can have thermal precipitation then we have immerson precipitation. So in all phase inversion or phase separation process.

There are two phases, one phase is precipitating, and that is forming the solid phase, okay and later on the membrane. So, in all this phase seperation process a liquid polymer solution is precipitant. Okay, to one solid phase, which is polymer-rich phase, another its liquid phase. Okay, which is polymer-poor phase.

So this polymer-rich phase will form the matrix of the membrane. Where is the polymer-poor phase will form the pores. Let us say, one by one, these techniques.

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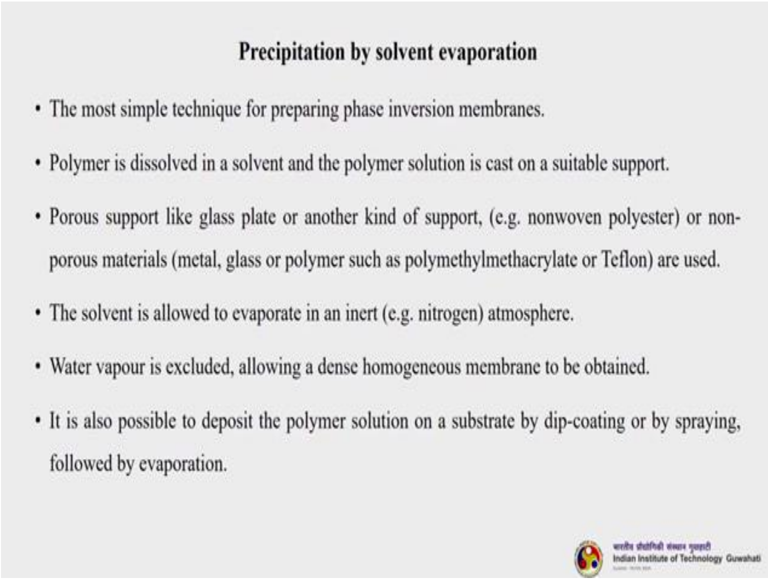

So precipitation by solvent evaporation Okay, so how we are precipitating by evaporating the solvent. So this is the simplest of the technique as the name indicates, okay polymer is dissolved in a solvent and the polymer solution is casted on a suitable support. Okay, so I can use a stainless paper plate or a glass plate, okay to cast the polymer solution support a support like glass or stainless steel. Okay.

Or non porous support also can be used, the solvent is used to evaporate in any inert atmosphere, easily nitrogen is used. So water vapor is excluded, allowing a dense homogeneous membrane to be obtained. So it is also possible to deposit the polymer solution on a substrate by deep coating, or by spray. So we will discuss deep coating later on.

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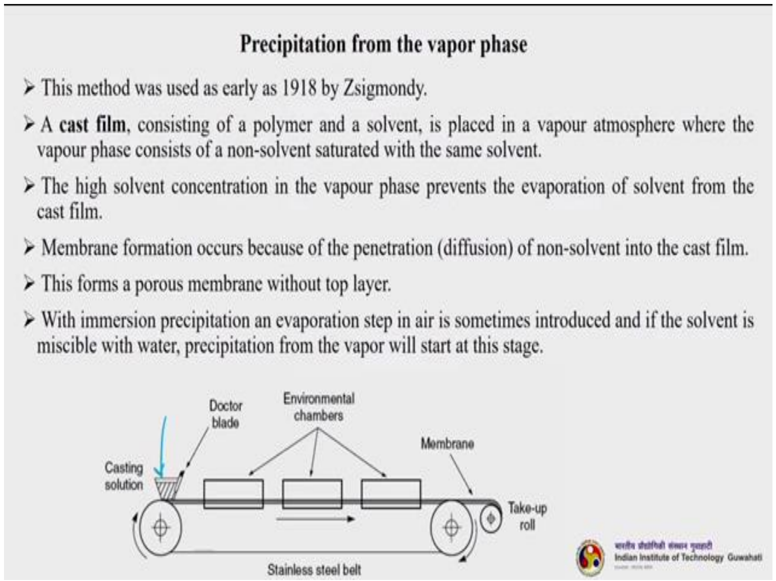

So the next one is precipitation by vapor phase. So this method was used long back in 1980 by Zsigmondy, who is prepared actually polymeric membrane using this particular technique. So a cast film, consisting of a polymer and solvent is placed in a vapor atmosphere, or we can call it is a humid atmosphere many books is written that humid atmosphere, but the vapor phase consist of a non solvent saturated with the same solvent. Okay.

So the high solvent concentration in the vapor phase prevents the evaporation of the solvent from the cast William. Okay, the membrane permission occurs because of the penetration of the non solvent into the cast film. So basically, the non solvent which is present in your vapor phase will try to penetrate to the matrix of the film. Okay, thereby creating the pores. So, with the immersion precipitation an evaporation step in air is sometimes introduced.

And if the solvent is miscible with water precipitation from the vapor will start at this stage so you can see this is a stainless steel conveyor belt arithmetic. In this particular schematic diagram. Okay, so you can have a doctor blade again the doctor blade, is the job is to increase or decrease the thickness of the particular membrane okay here you are casting solution can be poured here, then it is rotating stainless steel conveyor belt as well.

And this film is gradually passing through various chambers. So these are enveronmental chambers, some will have this vapor phase containing the non solvent but some will have the air, and in some chambers. drying will also takesplace just before we will take up the final membrane.

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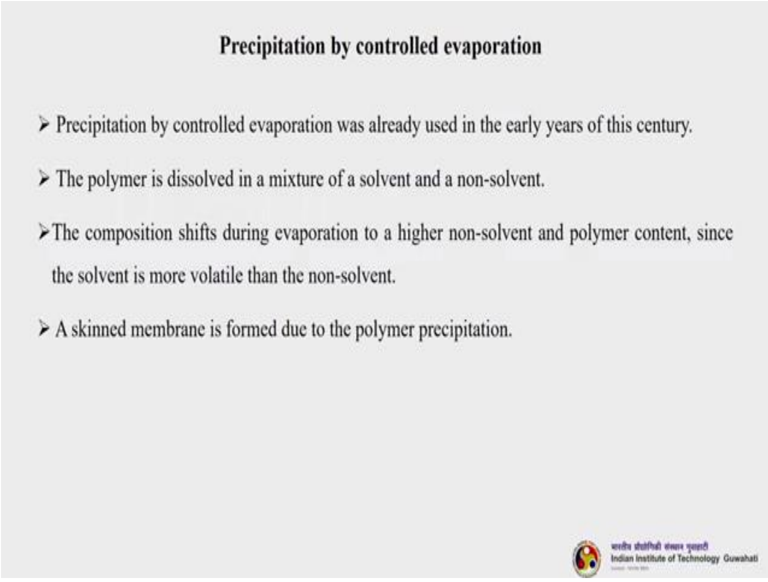

So, the next one is precipitation and by controlled evaporation. So precipitation by controlled evaporation was already used in the early years of this century. So the polymer is desolved in a mixture of a solvent and a non solvent. When I am preparing the cost solution that means the polymer is dissolved in the solvent, which will dissolve the polymer. And here I am adding a non solvent, at this stage.

Now the composition of this particular solution, in which there is a solvent and a non solvent will shipped during evaporation to a higher non solvent and polymer content, since the solvent is more volatile than the non solvent, for your understanding. So, that means, when I am making the mixture of a polymer, then its solvent as a non solvent, then what will happen when I am casting it then. seems the solvent is readly evaprated, it will evaporate.

So, the composition will be more towards the non solvent and the polymer polymer content. So that will form metrics of the membrane. So skin membrane is formed due to the polymer precipitation.

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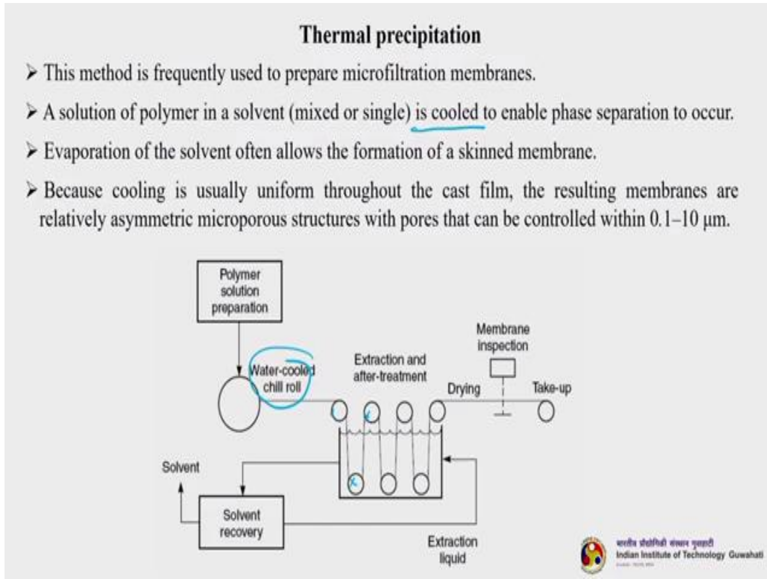

So the next technique is called thermal precipitation. So this method is frequently used to prepare microfiltration membranes, so a solution of a polymer in a solvent but either it is a mixed or a single. Okay, is coold, to enable phase seperation to occur so this is basically is a cooling technique. Okay, so, evaporation of the solvent, often allows the permission of the skin membranes. So in every process the solvent is getting evaporated.

And finally precipitation of the polymer membrane is happening or the metrix is happening. Okay. And because the cooling is uniform throughout the cast film usually it is uniform. Okay. The resulting membranes are relatively, asymmetric micro porous structures with pores that can be controlled within point 0.1-10 micron. Very smaller pore size can be obtained. So you can see this particular diagram.

There is the polymer solution is actually pored on a water cooled, chill roll, this is a water cooled chill roll. Okay, which is rotating in this direction. Okay. And once the chill role is cooling the casting solution, then it is resulting into a polymeric film. The film is passed through a extraction chamber where it is passing, so these are actually this whatever round shape things you are seeing these are this conveyor belt type arrangements. Okay.

And the film is passing through this chamber. Okay, then it goes through drying okay and the extraction solvent, can also be recycled.

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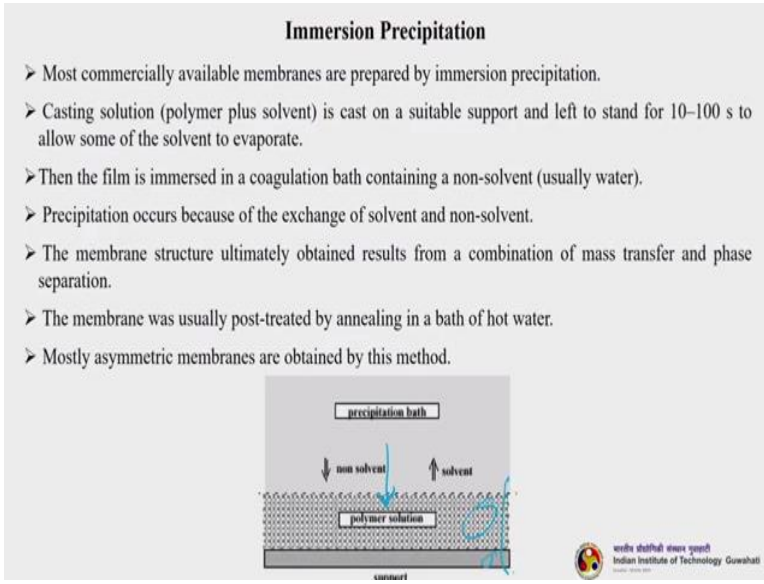

So the next technique, which is one of the most important technique on most important this one. Commercially adapted technique for membrane in preparation in most of the industries that is actually immersion precipitation. So, most of the commercial membranes are manufactured using immersion precipitation. So, here the casting solution, that means the polymer plus the solvent was dissolved in.

Okay is cast on a suitable support and left to stand for 10-100 seconds very small residents time such to allow some of the solvent to be evaporated. The idea is not to evaporate all the solvent was to just allow some of the solvents to evaporate. Then the film is immersed in a coagulation bath. Okay containing and not solvent. Right, so the non solvent can be usually water, we can have some other solvent also.

Non solvent also the precipitation occurs because of the exchangeof the solvent and non solvent. Right, so you can look this figure what is happening. So this is a polymer solution right, okay on the support, there is a support on there. So, this I am putting it in a precipitation bath, containing a non solvent. Now what is happening, you can see when the processes on the non solvent is trying to deffused inside the polymeric membrane whereas the solvent is diffusing out of the polymeric film.

Now this structure, actually creating pores. Okay, the membrane is usually post-treated by annealing in a bath of water. Okay, annealing is a metallurgical process. Right. So mostly asymmetric membranes are obtain by this membrane you can understand that we are talking about a symmetric membrane here. There are two layers here. Okay.

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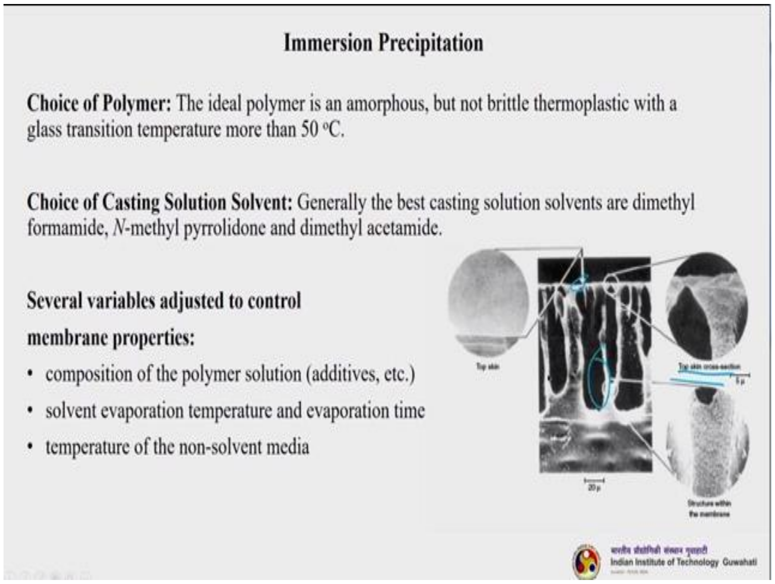

So, the choice of polymer and choice of casting solution solvent are very important for this particular process. So the ideal polymer should be an amorphous, but not retail thermoplastic okay with a glass transition temperature more than usually 50 degrees centigrade. And we can have varieties of casting solutions solvent, usually the best casting solution solvents are dimethyl formermamide, N-methyl pyrrolidone and dimethyl accetamide.

Apart from this, there are several variables that are adjusted to control membrane properties. Okay, some of them are composition of the polymer solution. So are you going to have some other additives. Okay, which can help in firming for a particular size or. So then solvent evaporation temperature and evaporation time and temperature of the non solvent media. So all of these parameters.

These are process parameters can be optimized to get a particular desired pore size and pore size distribution, you can see how it actually looks like. This is the top skin. Okay, this is the large image of the top screen. Okay. And this is the top skins, cross section. Right. And these are actually the pores.

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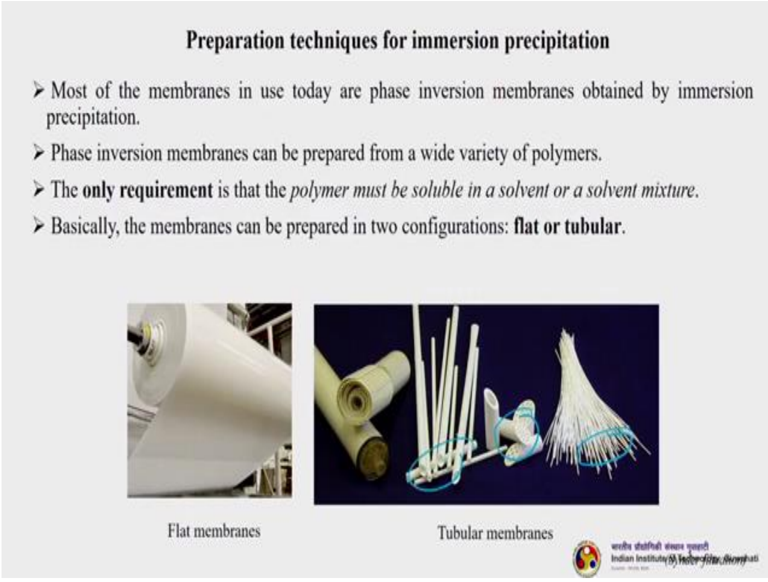

So, most of the membranes in use today are phase inversion membranes. Okay, they are obtained by this immersion precipitation techniques. So phase inversion membranes can be prepared from a wide variety of polymers. The only requirement is that the polymer must be solution in a solvent, or solvent mixture so that is the only criteria for using this phase separation or phase inversion technique.

Basically the membranes can be prepared in two configurations either we can have a flat sheet membrane. Okay, either we have a tubular membrane. So, you see how the flat sheet membrane, these are commercial membraness available. Okay. And these are hollow fibers. These are ceramic membranes. Okay, these are tubular membranes.

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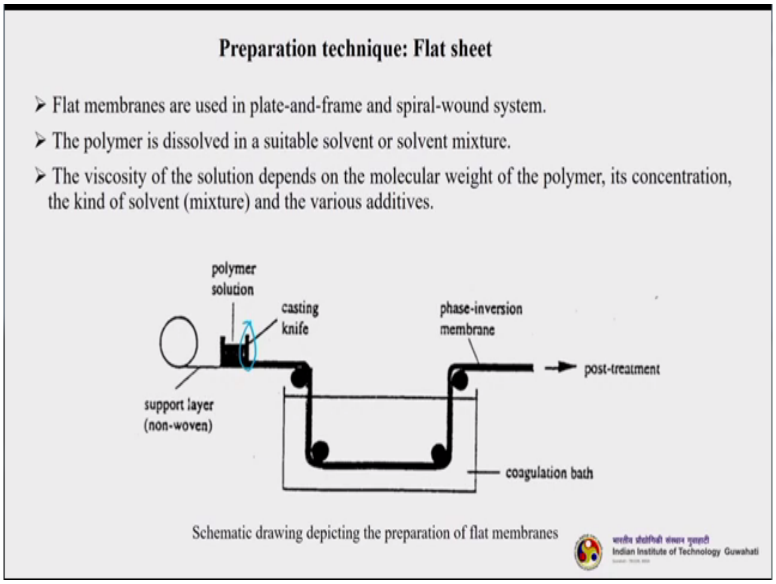

So, let us see how the plastic membrane can be prepared. Okay, the flat membranes are used in a plate-and-frame spiral wood system, this we have discussed when we have discussed the membrane modules. Okay, the polymer is dissolved in a suitable solvent or solvent mixture, the viscosity of the solution depends on the molecular weight of the polymer, its concentration, the kind of solvent and the various additives.

So polymer solution is actually pour on the conveyor belt arrangement again, mostly the same type of arrangements here. There are some conveyors usually having this stainless steel belts, right, then we can have a casting knife here, the height of the knife is determining the thickness of the membrane. Then it goes through a coagulation bath. Okay. And then after that also after it is cleaned.

And comes out of the coagulation bath okay we can go for, We will have to go for different post treatments. Okay. To before we get a dry membrane.

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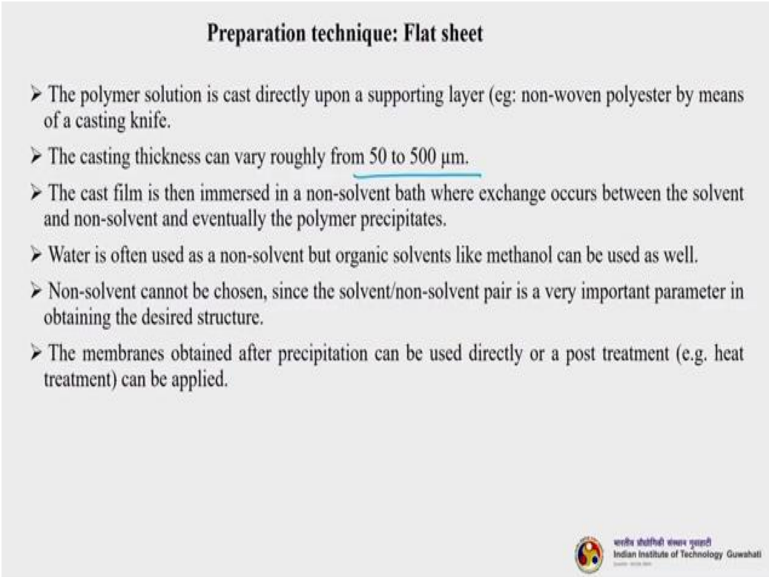

So the polymer solution is cast directly on a supporting layer, and the casting knife will actually help in deciding the thickness of the membrane, the casting thickness can roughly vary from 50 to 500 micron, so the cast film is then immersed in a non solvent bath, whatever the film that is casted using a polymer and a solvent, and the film is formed. Now that will goes to a now solvent bath. Okay.

When the exchange of course between the solvent and a non solvents, and usually the polymer precipitates. So the non solvent will try to diffuse through the membrane, or takes on the Polymer. Okay. On the film, and the solvent which is already present in the film will try to evaporate. So, this is how actually it happens. So, one is diffusing out another is diffusing in. So water is often used as a non solvent, but organic solvents take methanol okay.

Can also be used for different polymers. Now non solvent cannot be choosen. Since the solvent/ non solvent pair is very important parameter and obtaining a desired structure. So, the membrane obtained after precipitation can be used directly or post treatmen when that is heat statement can be applied said depends actually, what is the what is your intention and what is your intended application.

So depending on that, but we will decide, basically whether you wil use directly after the membrane is prepared, or it comes from the bath, or whether you are going to take it for some thermal treatment, such as drying, or some other processes like annening and cooling.

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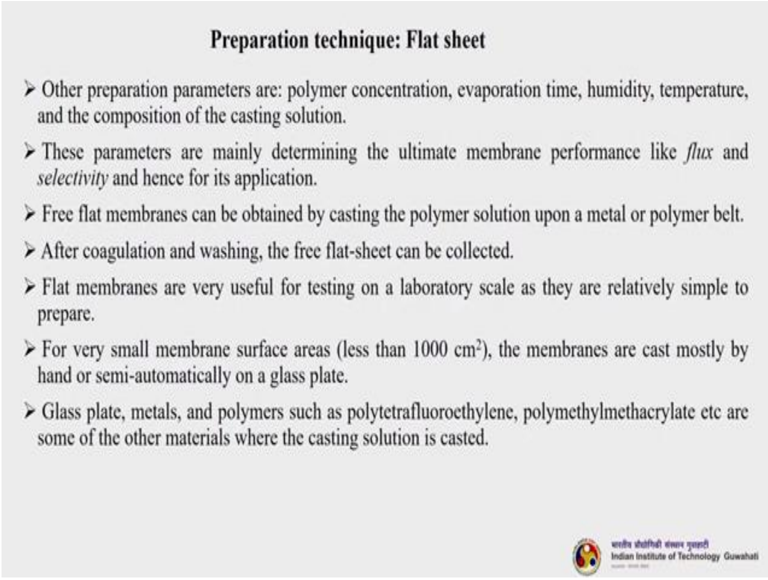

Other prparation parameters that helps in preparing flat sheet membrane are polymer concentration evaporation time, humidity, temperature and composition of the casting solution. Now, these all these parameters are mainly determining the ultimate membrane performance, which is flux and selectivity and hence its application. Right. So, what is the what is the flux and what is the selectivity of the prepared membrane.

Ultimately that will decide where this particular membrane will be used, what type of application. So a free flat membranes can be obtained by casting the polymer solution upon a metal or a polymer belt. So after the coagulation and washing the free flat sheet can be collected. If I do not want any other treatment processes that I can usually collect it after the coagulation and washing and use it.

So flat sheet membranes are very usefull for testing on laboratory scales as they are relatively simple to prepare for very small membrane surface areas. so it is less than 1000 centi meters square. The membranes are cast mostly by either by hand or semi automatically on a glass plate.

So glass plate metals and polymers such as polytetrafluoroethylene, polymethylmethacrylate PMMA, are some of the other materials which are with the casting solution is casted

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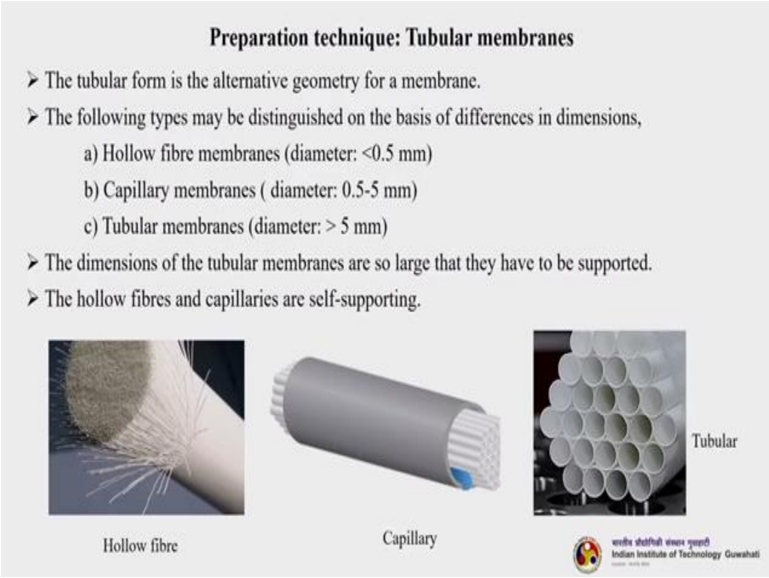

Let us see how the tubular membranes are prepared. So the tubular form is another alternate geometries and very good in terms of commercial application, so that we can have either a hollow fiber membrane so the diameter is <0.5 millimeter or you can have capillary membrane for the diameter is usually 0.5-5 mm or we can have tubular membranes.

So diameter is usually >5 mm. So the diamensions of the tubular membranes are so large that they have to be supported. So the hollow fibers and capillaries are self-supporting. Okay, this also we have discussed during our modular configuration discussions.

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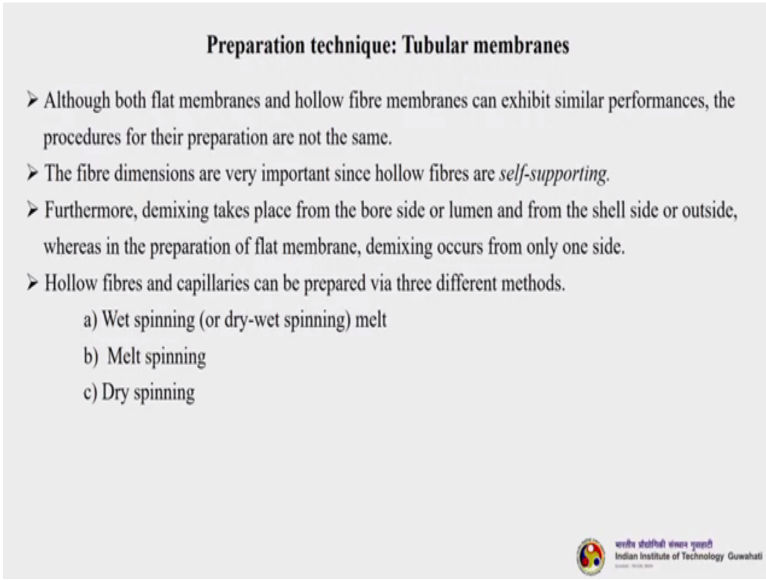

So all the both flat membranes and hollow fiber membranes can exhibit similar performance, the procedures for their preparations are not the same. This we have learned today. Okay, the fiber diamensions are very important since hollow fibers are self-supporting, since the hollow fibers are self supporting. So, the mechanical strength is actually less for them. And furthermore, the demixing takes place from the bore side or lumen. Okay.

And from the sell side or outside, whereas in preparation of the flat membrane demixing occurs for only one side. So, trying to understand what is the meaning of this. So when I am preparing a flat sheet membranes, the mixing that means the diffusion of solvent out, and not solvent in actually is happening from one side of the membrane and only, whereas when we are talking about tubular membranes or hollow fiber membranes. Okay.

So there is this cell side and there is a tube side, Okay, so there is something called inside of the tube and something is the outside of the tube. So from both size, actually, that the demixing is occurring so that is actually it is complicated, so we can prepare hollow fibers and capillaries by three different methods. One is called wet spinning or it is also called dry wet spinning method or melt. Then metal spilling and dry spinning. So we will discuss this first one

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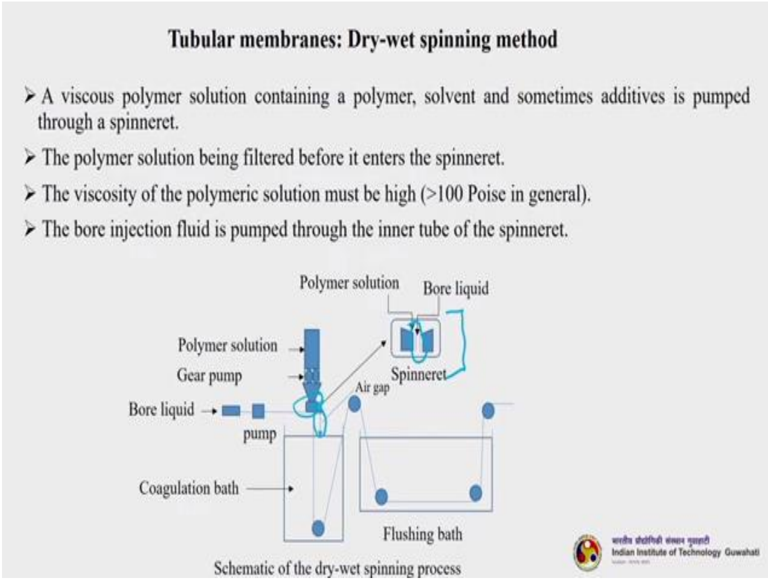

This is very important. Preparation technique. So a viscous polymer solution containing a polymer is solvent and sometimes additives are pumped through a spinneret. Okay, so this is actually the spinneret you can see this is the last version of this. And the polymer solution is being filtered, before it enters the spinneret. The viscosity of the polymeric solution must be high, usually it is should be>100 poise in general. Okay.

The bore injection fluid pumps to the inner tube of the spinneret. So this is the bore, actually, through which bore fluid is injected, and the polymer solution is passed outside the bore. Okay. There is an air gap. The air gap is plays a lot of role in deciding what is the type and quality and thickness of the membrane. And then it goes through a coagulation bath and flushing bath then before it goes to final dry.

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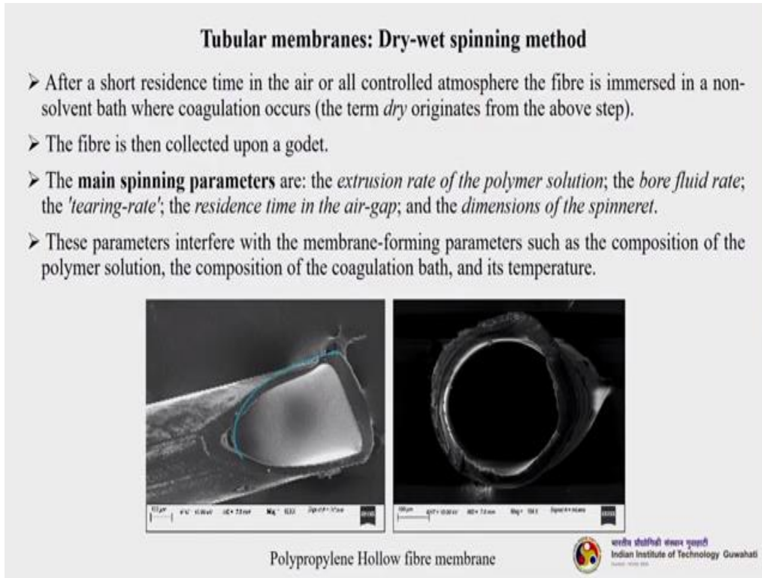

So, after a short resident time in the air, are all controlled atmosphere the fiber is immersed in a non solvent bath, where coagulation occurs, so that term actually dry originates from the above steps. Okay, we are calling it a dry wet spinning method. So the fiber is then collected upon a godet. The main spinning parameters or extrusion rate of the polymer solution at what rate, the extrusion is happening through the spinneret.

The bore fluid rate, the tearing rate, the residents time in the air gap. Okay and the diamensions of a spinneret, so these are the parameters which all can be optimized to produce a particular tearing rate membrane. So these parameters interfere with the membrane forming parameters, such as the composition of the polymer solution, the composition of the coagulation bath, and its temperature, you can see the scanning electron microscope images of a polypropylene hollow fiber membrane here. Okay,

So, this is a cut, actually. And this is the cross section. Okay, this is the inside of the tube, and this is the membrane thickness.

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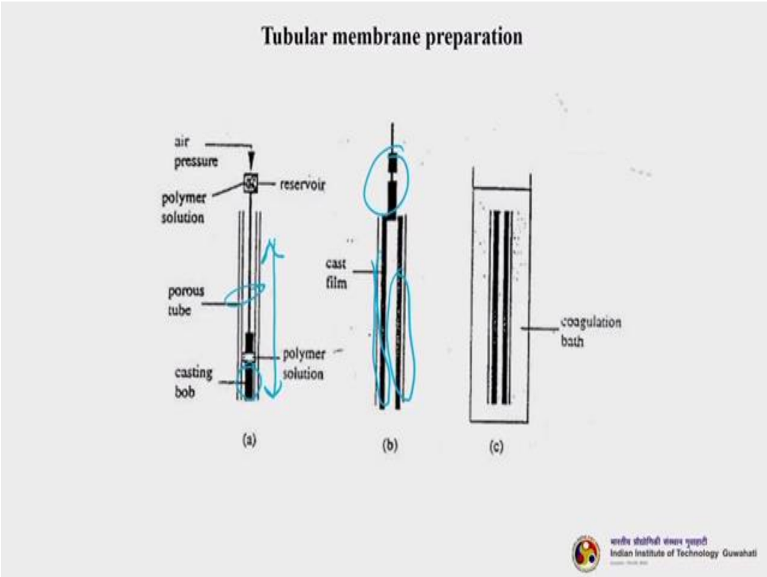

Student, This is the last slide. I'll try to explain this how it tubular membrane is prepared here, see the first one. Here we have a porous tube, this is the this is the porous tube actually. Okay. Right. And there is a casting bob, this one. Okay, this one, the casting bob. So the casting Bob is actually poros. So what we are doing or what is being done is actually, there is a reservoir here which is having the polymer solution.

So this polymer solution is pushed through the casting bob. So since the casting board is porous in nature. So the bore will allow the. This one polymeric solution to pass through the casting bob, and the casting bob is can be moved up and down. Right. So when it is moved up and down. So the polymer solution that is coming out from the casting book will get attached to the porous tube and the film will form, like this. Okay.

Once that is done, the casting bob was taken out, then we have to take this porous tube and put it inside a coagulation bath, so during this coalition bath what is happening again this solvent and non solvent diffusion will takes place. Okay. And after a certain time, you will allow certain residents time in the coagulation but what will happen that the membrane which is found inside this porous tube you will become loose, then we can take it down for further processing.

We can get over some other processing like a aneeling or some heat treatment, it is required. It depends upon what is the intended application so students this is how tubular membranes prepare. So in today's class we have discussed basically how non porous or dense membranes are prepared, then how symmetric and asymmetric micro porous or porous membranes are prepared, as well as you have seen how flat sheet and tubular membranes are prepared.

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So, these are some of the references that I am using. Okay.

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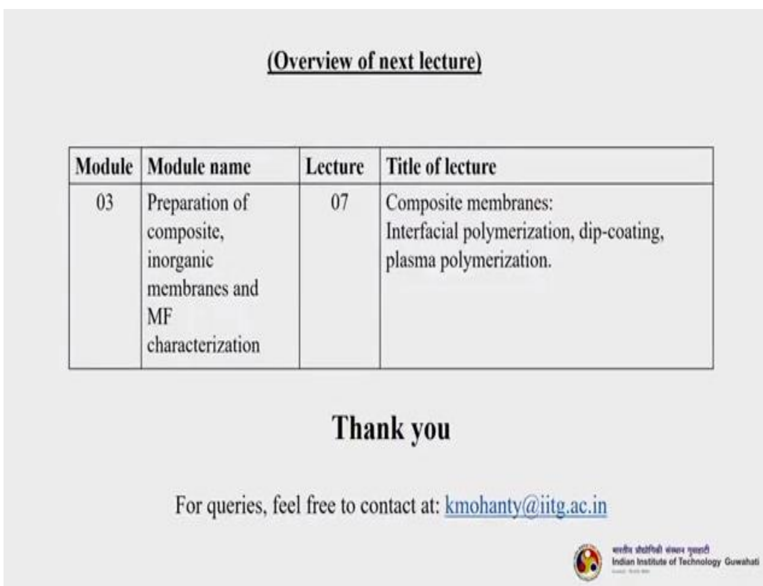

And then in the next class, we will discuss about the preparation of the composite membranes. So, and by varrious technology, techniques, which is interfacial polymerization dip coating plasma polymerization okay. interfacial polymerization is one of the most important breakthrough in preparing this integrity skin remembrance. Okay, so please keep reading. And if you have any doubt. You can always drop a mail to me at kmohanty@itg.ac.in. So thank you very much.