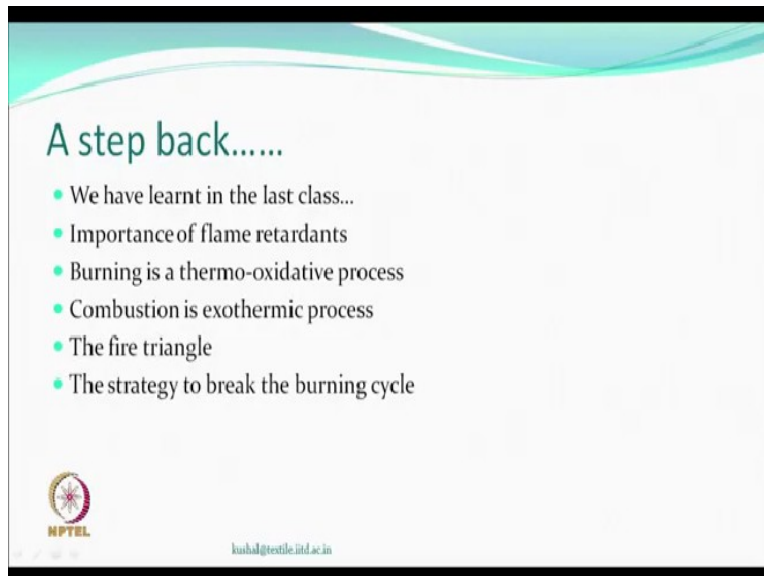


**Textile Finishing**  
**Prof. Kushal Sen**  
**Department of Textile Technology**  
**Indian Institute of Technology-Delhi**

**Lecture-21**  
**More Flame Retardants and Evaluation of Fire Retardancy**

Welcome back to our class in textile finishing, we shall just revise as to what we did in the last class.

**(Refer Slide Time: 00:32)**

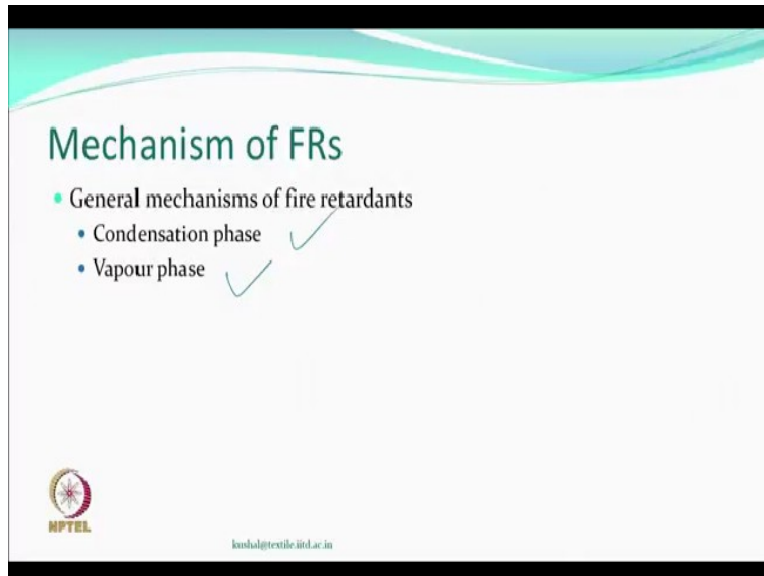


So in the last class we learned about the importance of flame retardant, why we must do some treatment, so that a good number of textiles could be made flame retardant and we understood that most of them succumb to flame and fire but what it means therefore is that they actually are fuels. We also learned that this whole burning process is thermo oxidative process, so you have to give certain amount of energy to initiate the new need somewhere oxygen.

And then this process starts and finally you have combustion where you can see the flame most activities are in the gas phase. So there is a fire triangle and the strategy is what one has to adopt is whether at what level at the condensation level or at the gas phase level we must intervene. So

that the flame is not propagated. So that is how we learn some strategies to break the burning cycle.

**(Refer Slide Time: 01:55)**



We also learnt general mechanisms which we called as condensation phase mechanisms or vapor phase mechanisms. So condensation phase means that the flame retardants will during the burning process alter the process of burning you know path of decomposition and the vapor phase is where all the gases are now there in the vapor phase free radicals are being formed and so you have to worry about either the free radicals or the heat release has to be taken away.

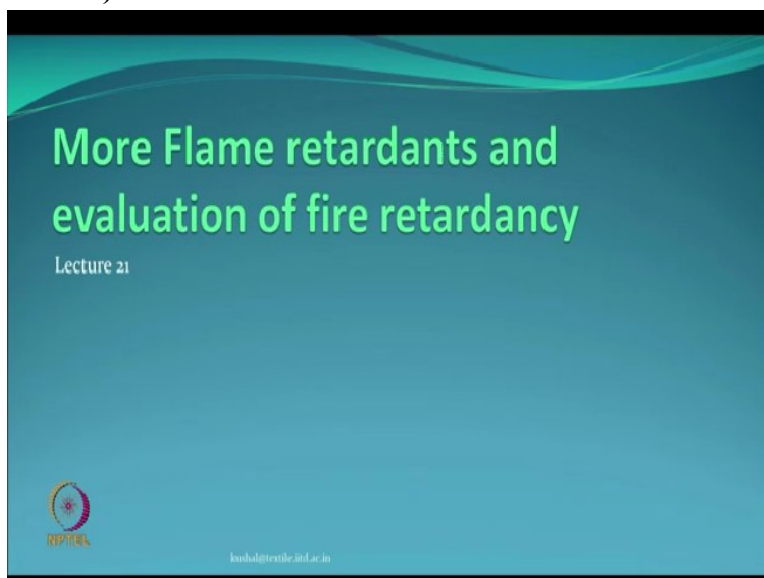
All these things therefore become quite important, of course there can be some flame retardant agents which may be working with the mechanisms. So it is possible.

**(Refer Slide Time: 02:38)**



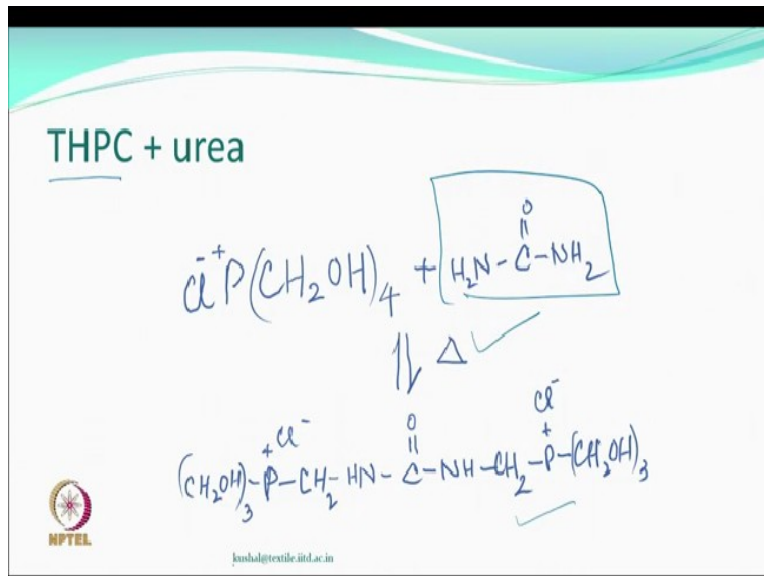
Also we did learn something about chemistry of some of the fire retardants.

**(Refer Slide Time: 02:45)**



Today we will go a little further and learn little more about the fire and flame retardant compounds and also will dwell upon as to how flame retardancy is evaluated.

**(Refer Slide Time: 03:11)**



So one of the compound which we talked about last time was THPC tetrakis hydroxy methyl phosphonium chloride right. So in the presence of heat and in the presence of urea some such compounds are formed. So it's a large molecular compound and one can neutralize this also with another alkaline compound may be you could use ammonia itself, if you use ammonia then you can get cross-linked products.

**(Refer Slide Time: 03:59)**

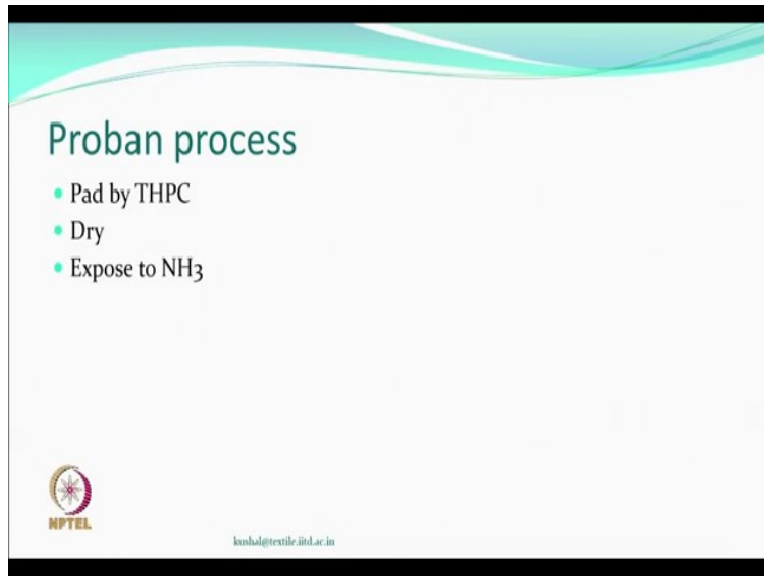
### THPC + urea + ammonia

- Ammonia neutralizes
- The previous compound makes crosslinked structure
- Effective FR
- Improves wash fastness.

If you use THPC, urea and also ammonia which we neutralized so that chloride part will be taken care of reactions can take place, they can make cross-linked structures and therefore the wash fastness can be improved and this combination particularly for the cellulosic material have been

found to be very very effective of flame retardant. Obviously we can appreciate this works generally by the condensation phase mechanism.

**(Refer Slide Time: 04:52)**



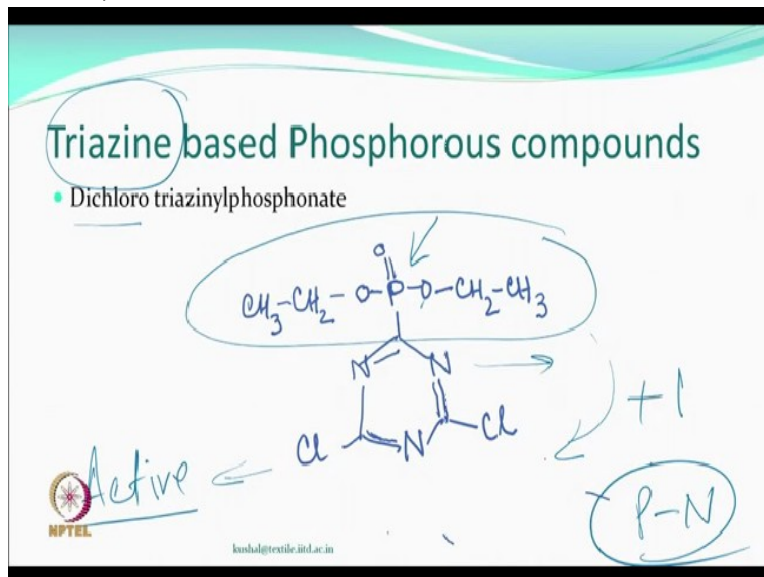
Another process which was commercialized and was known as a Proban process also used THPC ok, this Proban process also used THPC, so what you had to do was padded by THPC a solution dry and then during this process you expose it to the ammonia vapors and then the reaction would be there and in some sense there will be polymerization and cross-linking between the molecules and therefore the size will be more and it works.

So basically the main component is the THPC and within the made THPC it is the phosphorous which is the most important component. So one would actually like to have as much phosphorus as possible in a compound, the more it is the better it will be this is expected because this is the active compound from the condensation mechanism point of view. We learn little more about some compounds which are phosphorus compounds.

But you want to make them reactive, so that they can react with the the substrate and so will be permanently present till it is required when actually fire happens, you see we expect the fire is not gonna be happening everyday you see it will be once in a while and that will be the last time the fabric is going to be used, till that time we will be washing it off. So therefore wash fastness becomes quite an important things.

So that the effectiveness of the flame retardant is kept, so one of the ways in which this can be achieved is using our tried and tested you know group called the triazine group you know this triazine group.

(Refer Slide Time: 06:58)



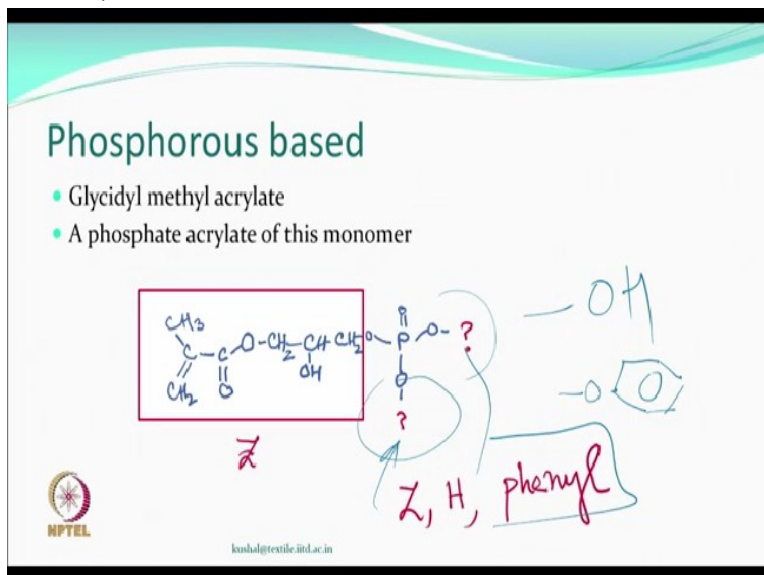
You are familiar these type of groups are used for reactive dyes, you know monochloro dichloro type of reactive dyes one can use and therefore reactions can occur from here in you know approximately alkaline conditions for the hydroxyl groups like the cellulose has hydroxyl groups or they can actually have reactions with amino group amino n groups of protein based fibers. So what you will attach is a phosphorus permanently.

Now this like for example we this is a dichloro triazinyl compound if you add this particular compound on this side as well one more, then you will have a monochloro compound. So reactivity will be reduced but you still have will have one so this chlorine will be replaced by this so you will have monochloro compound but this will be the active group which can react right. That is how it works. So you can get a reactive compound and once it is reacted.

So it is covalently bonded and so will be retained for a longer period of time and what we are finally interested is obviously this phosphorous compound. So you can have one molecule of this group or another one. Obviously you can understand will not have 3 ok, then how will it react but it will become a very large molecule, it can obviously will work as a flame retardancy is concerned but may not be fast, so fast as they chemically reacted one.

So in this compound you also visualize in the previous also that we do have something like a nitrogen and phosphorus. So phosphorus and nitrogen synergism can be expected in this type of a compound as well. So it should be a very effective compound.

**(Refer Slide Time: 09:33)**



So another compound which is also based on a different group, but this is a reactive group and this reactive group again is an n methyl group and you have phosphorus here and other of course are the compound which have been attached or the groups which have been attached to the phosphonate to make it compounding. This became a very popular commercial compound as well called the Pyrovatex CP, that this was CP so there may be based on what you add to different parts of the hydroxyl group.

So say with the phosphorus you will have different compounds, so they would have different names. So various compounds this group must have tried that is very effective flame retardant for cellulosic fabrics, it also in some sense has some nitrogen and phosphorus of course the phosphorus is the main compound added in a support system which people believe that it works as a synergistic effect. It also supposed to work obviously by a condensation phase mechanism. Interesting compounds where the phosphorus still remains the key.

But you have this group which we are referring here as Z, this is an acrylic based group but it is got double bonded structure is that it has got double bonded structure and so it can react by

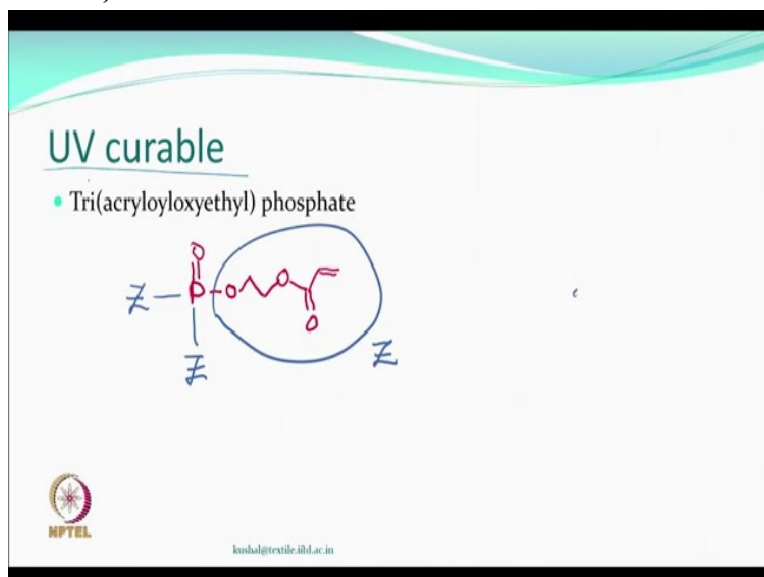
addition reactions with any functional group like hydroxyl or amino groups ok, you get the point. So it can react also, so the reaction can take place from here, the reaction therefore this type of a group depends on how you react sometimes it may be curable by irradiation also.

And so it will become a new compound, now the other part of the phosphonium based system you can have either the Z going there or the hydrogen which will mean this will become a hydroxyl group or a phenyl group can also be attached here which would mean that this would become something like that. So you can make different compounds based on this type of a general structure.

And the reaction if at all will be happening from this side ok, this will be an interesting reaction reacting group. So it has only phosphorous does not have nitrogen, so maybe it will become more effective if you have protein as fiber, it can work on the condensation phase mechanism because the nitrogen can come, from the protein itself. But otherwise because the phosphorus that it is there it is going to be an effective compound.

Similar compounds people have tried which they actually believe can you know react as UV-curable. So the previous one as well as this one if you have this kind of groups available they can be cured by additional reaction by irradiation as well.

**(Refer Slide Time: 13:31)**



And so they can be in some sense reacted and so wash fastness etc. would be better.



(Refer Slide Time: 13:41)

**Burning behavior of wool**

- We had learnt that wool is inherently more flame retardant than cellulosic fibre fabrics
- But it burns under the VFT conditions, i.e., burning from the bottom.
- Why?

The diagram shows two vertical rectangular pieces of fabric. The left piece has a flame at the top, with a curved arrow indicating the flame's movement. The right piece has a flame at the bottom, with a curved arrow indicating the flame's upward movement. The word 'Burn' is written in blue cursive next to the bottom flame. In the bottom left corner, there is a logo for NPTEL (National Programme on Technology Enhanced Learning) and the email address knshah@textile.iitd.ac.in.

Next now get into another interesting fiber that we did in the beginning looked at it is burning behavior. We did learn that as such wool is relatively much more shall we say in inherently flame-retardant compared to later cellulosic material cotton and viscose, but we also found that it also burns if you ignite it from the bottom right, if you have this fabric and you ignite from the bottom then will burn completely.

But if you burn from the top then it may stop, it would stop actually it burning if you burn from the top. So why it burns on the bottom remember because the heat obviously and the hot air and the flue everything has a tendency to go upwards. So it becomes more and more vulnerable to this burning process, but from the top when you burn it is the slowest in this kind of a test and so if the fiber resists which the wool fiber resists then it gets extinguished the flame gets extinguished.

So this is how the burning behave, but what it therefore means is if you really are interested in making the woollens also safe if you will have to treat them you cannot rely on this small little resistance that it offers and say well everything is fine from the flame-retardant point of view that is not true. So why does it behave in this manner that is it is relatively more resistant from polyester, from silk, from viscose on burn why?

So one the reason one can appreciate it that wool is covalently linked as far as the intermolecular linkages are concerned which other fibers are not. So that can provide some resistance through the disulfide link, also it is presumed that sulfur is also going to play some role and in some cases it has been also seen that if sulfur is also present. For example in nylon if sulfur is present it becomes flame retardant as well.

Therefore phosphorous play the role sulfur also has to play some role and wool has sulfur it has got cross links and to get thread together it becomes relatively more flame retardant.

**(Refer Slide Time: 17:13)**

The slide is titled "Wool" and lists phosphorous based compounds:

- Phosphorous based compounds
  - Phosphates and polyphosphates
  - Phosponates
  - Phytic acid (inositol polyphosphate)

A chemical structure is drawn showing a central phosphorus atom (P) double-bonded to an oxygen atom (O) and single-bonded to three hydroxyl groups (OH). This phosphorus atom is also single-bonded to an oxygen atom that is part of a cyclic phosphate ester. The cyclic phosphate ester consists of a six-membered ring with five carbon atoms and one oxygen atom. Each carbon atom in the ring is bonded to a hydrogen atom (H) and a sulfur atom (S). The sulfur atom is also bonded to a hydrogen atom (H). The phosphorus atom is bonded to the oxygen atom in the ring and to a hydrogen atom (H). The structure is labeled with "Z" at various points, indicating the presence of sulfur atoms.

NPTEL  
india@textile.iitd.ac.in

So we look at some interesting compounds other than whatever we talked about the phosphorus based compounds whether they are phosphates which we have seen before or phosphonates, they will be also effective on wool by the condensation phase mechanism, one very interesting compound people have been talking about these days is phytic acid I am not sure if you have ever heard about this term called phytic acid.

But let us learn today, the important thing is that in a cyclic way this is the phosphorus moiety which is based on the phosphoric acid. This is all over everywhere, so this is repeated this is we called Z, the Z is there, Z is here, Z is here, Z is here and Z is here. So all over there is Z, so this means that this wool compound has got large amount of phosphorous and in some sense

phosphoric acid once it will be released in during the burning process a lot of phosphoric acid will be released.

So per unit mass you will have more phosphorous, so let us say carbon to phosphorus ratio here will be high and so it should be more effective and so people have tried this for various limited application but the phosphorus based compound interesting compound it can be and if you can make sure that this group reacts with the let us say amino groups by linkages or otherwise then you would see relatively more permanent or you can use other cross-linking agent which can react with this and the wool and hopefully become more fast.

**(Refer Slide Time: 19:28)**

The slide is titled "Wool" and contains a bulleted list under "Sulfur based compounds" with "Sulphamic acid" as the only item. To the right of the list is a hand-drawn chemical structure of sulphamic acid,  $\text{H}_2\text{N}-\text{S}(=\text{O})_2-\text{OH}$ . The slide also features the NPTEL logo and the email address [kushal@textile.iitd.ac.in](mailto:kushal@textile.iitd.ac.in) in the bottom left corner.


There is another compound you see the structure do you recognize this compound it is a sulfur-based compound will already a sulfur but it is sulfur based compound which is called sulphamic acid right, this sulphamic acid has also been seen to be a very effective compound for flame retardancy of wool as well as silk. The difference between silk and wool is the sulfur content and the crosslink that is a disulfide cross-link which wool has, but silk does not have right.

So that is the difference and if he tried to provide sulfur it does help in these 2 cases as well, but the most commercially successful in some sense the finish for wool has been based on zirconium, titanium based compounds and it was developed by IWS.

**(Refer Slide Time: 20:41)**

**Zirpro finish**

- Developed by IWS
- Hexafluorozirconates ( $ZrF_6^{2-}$ ) and Hexafluorotitanates ( $TiF_6^{2-}$ ) attach onto positively charged sites in wool under acidic conditions
- Along with dyeing at around pH 3
- Very effective FR

 [www.nptel.ac.in](http://www.nptel.ac.in)

india@textile.iitd.ac.in

The slide features a light blue header with a wavy pattern. The title 'Zirpro finish' is in a bold, teal font. The bullet points are in a teal color. Handwritten blue circles and lines highlight the chemical formulas  $ZrF_6^{2-}$  and  $TiF_6^{2-}$ , the pH value '3', and the phrase 'Very effective FR'. The NPTEL logo is in the bottom left, and the website URL is at the bottom center.


And called as zirpro finish, it is expected that in the acidic condition wool would have positive charge and this compound the fluoro compound with a zirconium and titanium are negatively charged. So they do get attached to the wool pH maybe if around this pH or maybe it is around that you can theoretically do exhaust dyeing as well as exhaust this finishing agent along with the dyeing.

So you can have dyeing as well as finishing together as for this but look about concern, a very effective flame retardant it has been seen and works very well.

**(Refer Slide Time: 21:32)**

# Wool

- Tetrabromophthalic anhydride (TBPA)

 [kushal@textile.iitd.ac.in](mailto:kushal@textile.iitd.ac.in)

Other compounds also have been tried one of these compounds which suggested which called the TBPA tetra bromophthalic anhydride, so you have this anhydride and there are bromine groups all over. So the bromine groups obviously are suggesting to you I hope understand that they are going to be released in the flame and they will be more active in the gas phase when the burning and so will be less active in the condensation phase. For such type of compounds can be for the blends of wool and polyester for that matter can still work.

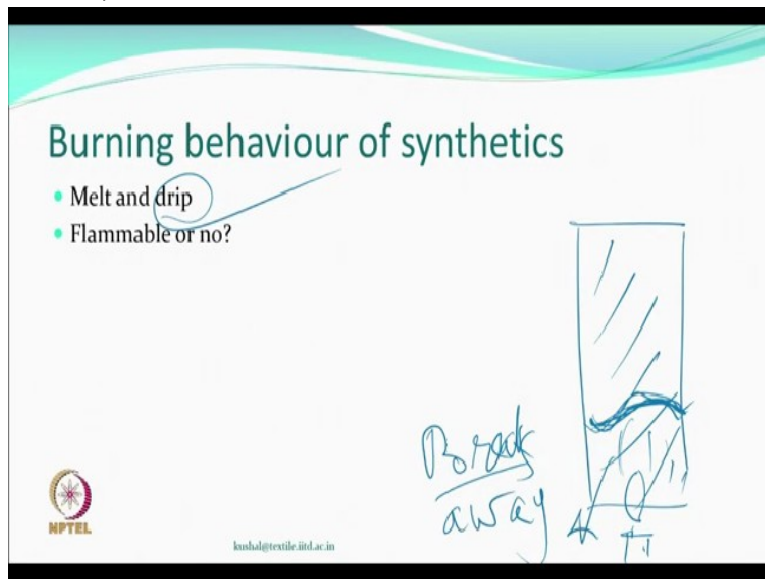
**(Refer Slide Time: 22:21)**

# Synthetic fibres

 [kushal@textile.iitd.ac.in](mailto:kushal@textile.iitd.ac.in)

Before we finished the flame retardant of finishing topic, let us look at some synthetic fibers also. Now synthetic fibers the names we know, what are the names polyester, nylon, polypropylene, they very easily burn, but important thing is they melt and drip.

(Refer Slide Time: 22:52)



Let us say if you have fabric a being burnt from the bottom, so as it burns obviously rapidly but because these materials melt let us say a polypropylene will melt somewhere 170 degrees, nylon 6 for example at 210, 215 degree centigrade, the polyester around 250 to 260 degree centigrade. Now if they are softened and they are actually melt, so there is always a possibility because the heat is going up.

Before the burning takes place of the polymer, before the polymer actually starts burning at temperatures which are obviously less than 250 degree centigrade, the burning has not started if the portion of the fiber melts and then breaks away, what it means is drips melts and then drips. So if the polymer started burning from bottom the heat went all the way up polymer became soft, the polyester or polypropylene and they just fell down before the rest of the matter material could catch fire.

So this burning portion will come down in case you are lucky and the floor something which does not burn, then you are in some sense safe, they just has fallen down, so this behavior is different than let us say the wool, silk behavior, cotton behavior which do not melt and drip, the way the synthetic fibers would do that. So if you burn the cotton fabric from the bottom and just completely finish off in no time.

But you can have a situation where a portion top portion above the flame will become so soft and after melting just falls down and so further propagation on the upward direction may not take place. So in some sense you can say well they can extinguish because the top portion has burned so it will not keep supporting, but if you do it from the top then obviously everything will burn because then you have the solid thing over which the burning and the flame is there.

So in some applications as I said you may get some benefit that let us say the curtains all around, the walls they may start burning from the bottom part of the thing this falls down and let me stop burning because the burning portion has been taken away by this dripping and so further burning may not take place. So that way it can extinguish in a different mechanism. But can we call this as safe obviously not it is burning ok.

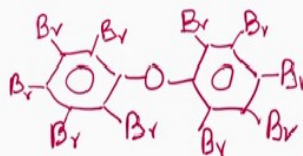
People may say well this is you know safe in that sense that is not true actually and difficulty also comes know people also sometimes say the cotton fabrics are safer from the point of view of flame retardancy, only one reason they burn very fast so there is no safety there, here in the synthetics this shrink also not before melting they would shrink and if they are next to skin they shrink, the heat is there the heat will come directly to the skin.

And you not be able to throw the garment out while in the case of let us say cellulose you may be able to remove something which is burning it is not sticking and not that there is not going to be damage will be there, they will be from wearing point of view they will shrink and actually transfer all the heat to the skin and the body. So nothing is safe here, so you have to treat them.

**(Refer Slide Time: 27:33)**

## Bromine compounds

- Decabromo diphenyl oxide
- Will work on polyester nylon, PP etc
- And their blends as well
- Can be added to the melt



kushal@textile.iitd.ac.in

Bromine compounds I mean they are halogenated halogen compound, so instead of chlorine you know most compound the flammable compounds where they bromine based compounds, very successful compounds as for the flame retardancy is concerned, this particular compound you can see has got so many bromine atoms and so when then the degradation will start this compound will degrade and release let us say that HBR and so you would have that free radical quenching ok.

And so flame will not propagate further, so very very effective compound it could work because works on the gas phase as long as the compound is on the textile it will go up in the flame and extinguish the flame, some of these compounds can be added during the manufacture of the fibers also as additives, they stay there, so they become more permanent in that sense. So once they are there inside they will not be leached out every time you wash.


And would be available when it is actually required but that is a process which has to be conducted by the fiber manufacturer not in the textile finisher ok, that is what we talked about.

**(Refer Slide Time: 29:00)**



## Polyester

- Tris 2,3 dibromo phosphate
- Very effective

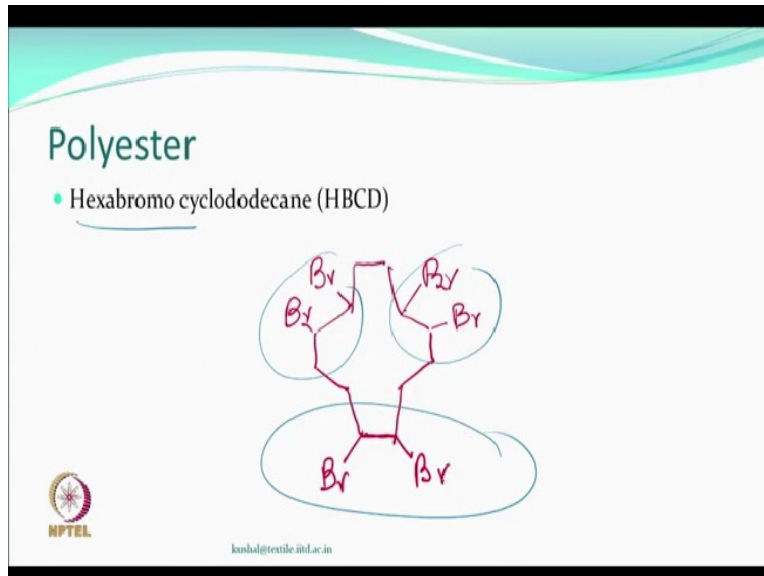


[kushal@textile.iitd.ac.in](mailto:kushal@textile.iitd.ac.in)

Another very interesting compound which is phosphorous compound, why we gain one and go to the phosphorus, the phosphorous and bromine combination also can be an interesting compound so part of it will work by the condensation phase mechanism, the other part would work with the vapor phase. So you have a dual action right. So if this compound is either as a finish is available on the fabric. So you remember there this is 3, so Tris 2 3 dibromo phosphate ok.

So looks on one side but there are 3 such groups on all sides of phosphorus, so interesting compound actually at right kind of conditions one can be sure that phosphorus will also be available, bromine phosphorus will act in the condensation phase, bromine in the gas phase and so can be a very safe compound. People have tried lot of compounds with more and more bromine.

**(Refer Slide Time: 30:38)**



This is one another compound which is hexabromo cyclododecane HBCD and so it is got this bromine can be you know little flexible in whichever type of area they are there they will be released. So the question that remains is if you want to think to be more effective then you go to have let us say if you want to work in the gas phase then more bromine type of elements would be there, with time people have somehow not happy with the halogens.

But they are very effective flame retardants, but from the environment point of view the halogens as long as they are attached there is no problem but even when they burn out the gases that they produce the mechanism is very clear, but people feel that this is not a good idea. So a good number of organo chlorine compounds or organo halogen compound like bromine based compounds they are very successful in the gas phase.

And very good flame retardants today we are having a relook and therefore people want to shift to the phosphorus nitrogen combinations and sometimes we are talking about fluorine based compound which also when they burn there can be some difficulty silane based compounds people are now looking at. So that the phosphorus is there and other mechanisms can be also available and so the whole flame retardant chemistry is having a relook.

And going instead of working from the gas phase you may find more and more compounds being used which will be more active in the solid phase or the condensation phase.

(Refer Slide Time: 32:57)

**Blends with synthetic fibres**

- What do you think?
- Say PET/Cotton
- Scaffold

 NPTEL

[kushal@textile.iitd.ac.in](mailto:kushal@textile.iitd.ac.in)

One important thing which may spend some time here is if you use the blends let us say with polyester and cotton kind of a situation we know polyester burns, we know cotton burns, what do you think would be happening if we have the blends ok, if suppose we have a blend of polyester and cotton or polyester viscose and you burn, what would you expect. So the experiment they were done they found that the blend from the point of burning is less safe compared to the 2 fibers actually both fibers are not safe.


But the blends is worse, why because one of the mechanisms which we thought when the polyester burns it can melt and drip but when cotton burns it does not melt but when cotton and it is ash and the microstructure is available the dripping of the polyester does not take place in fact the cellulose based char becomes like a scaffold and holds the polyester there and there and so polyester also burns more.

Together they burn more faster, more dangerous, so you have to treat both that is one important thing do not have to have situation where you treat only one and the other is not to be treated. So in general we understood that some of the compound which were there which we talked about can work for the synthetics the bromine are concerned they will work for synthetics, but if we do not want bromine then phosphorus nitrogen based combinations will have to be tried.

(Refer Slide Time: 35:14)

## Nylon

- Thiourea
- It works very well
- Use of formaldehyde with thiourea ?


 NPTEL  
knshd@textile.iitd.ac.in

One interesting compound is thiourea, it is got sulfur and it is been found that if you apply thiourea and in some area the flame retardancy of nylon improves tremendously, we talked about sulphamic acid yes, the thiourea also has sulfur and it works very well, people have suggested that in case permanency required maybe you could make a thermoset resin with thiourea and formaldehyde well, formaldehyde had some problem that we have seen earlier but it can be used right.

(Refer Slide Time: 35:55)

## Application

- Pad-dry-cure
- Spray-dry-cure
- Coat-dry-cure
- Pad-dry-UV cure

 NPTEL  
knshd@textile.iitd.ac.in

So how do we apply well, we have simple in case we believe it is a permanent thing which we want some reaction take place, the best is pad dry cure conditions and with all type of groups you know about conditions and if you have one type of a surface which has to be treated one way and the other way then you can do the spray and dry, if you can have resins latexes where the flame retardant has been included in the recipe.

Then you can do the coating, after coating you can do the drying and of course if you have UV curable resin then you will say well I will do pad dry UV cure right. So you will want to some reaction will take place, some polymerization, some networking within the molecules must take place, so that wash fastness is also improved, so curing is important in all cases whenever permanency will be required.

**(Refer Slide Time: 37:06)**



Although this part of discussion does not form the part of finishing, that is more towards the chemistry of the fiber itself, more related to the chemistry of the fiber or the chemistry of an editor which lets say during the formation of a fiber I am talking about manufactured fibers where the synthetic are regenerated, you can add the flame-retardant during the manufacture itself either as a co monomer or as an additive.

That way definitely there will be more permanency, but some fiber structures are by themselves so nicely conjugated that they really resist the fire quite a lot and or in a way they are more stable at higher temperatures. These type of materials would come in the category of inherently flame-

retardant fibers. In some cases where we expect that the fire could be very very strong you may like to use such fibers.

And not any other finish, for example the upholstery in an aircraft for that matter, you would not like to take any chance so some use in railways or in buses and you may like to use them or in auditoriums also you may like to use inherently flame-retardant fibers, but remember what we said this is not a finishing treatment, what is this, this is actually either by the chemistry itself the fibers are quite resistant to degradation by heat or you have added a flame retardant during manufacture.

**(Refer Slide Time: 39:22)**

**Inherently flame retardant fibres**

- Aramid fibres ; Nomex and Kevlar. Aramid fibres Because of their chemical structure, these don't break down into combustible molecular fragments.
- Polyphenylene sulphide fibres (PPS) are produced by melt-spinning. These does not support combustion under normal atmospheric conditions.

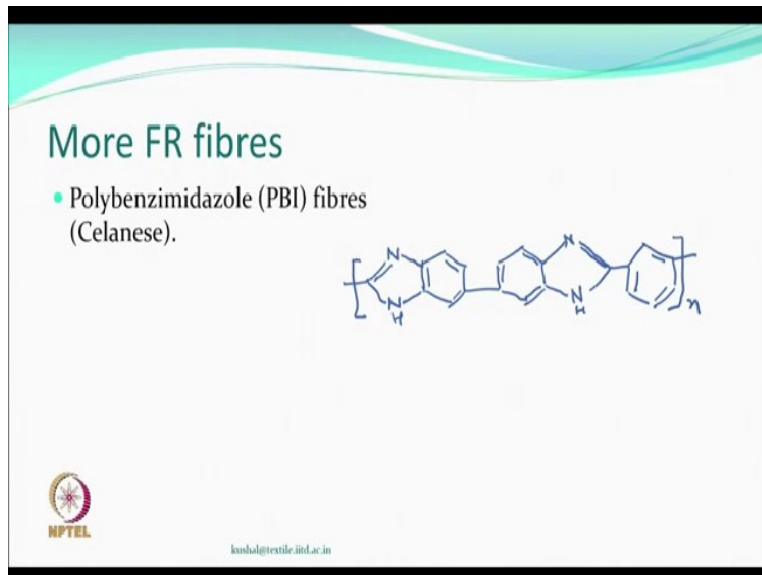
The slide features three chemical structures: a black and white structure for Aramid (top), a green structure for another Aramid variant (middle), and a red structure for Polyphenylene sulphide (bottom right). The Aramid structures consist of benzene rings linked by amide groups, while the PPS structure consists of benzene rings linked by sulfur atoms. The slide also includes the NPTEL logo and the email address [kushal@textile.iitd.ac.in](mailto:kushal@textile.iitd.ac.in).

So you must have heard about these words like Nomex, Kevlar, so they are by nature more resistant ok, because of the structure that they have and it does not break down easily into the molecular fragments which will start supporting the flame you see what I told you earlier is also important that. In case somebody sees or some automated stuff is there, there is a fire something will happen you only require a little bit of a time.

And they resist to that and so they will be pretty safe from that point, if you they are very heat-resistant material. So these are the 2 structures of the Nomex and Kevlar which one is Nomex, which one is Kevlar, any idea. So this structure is Nomex and this structure is Kevlar alright. So because of these aromatized rings quite a lot of them. Therefore they are called the aramids or by themselves very much resistant.

Kevlar is known for other uses like in bulletproof jackets and so on so forth as well, another interesting fiber based on sulfur is the polyphenylene sulphide fibers, the special fibers and they also are very very resistant to heat, maybe you have not heard both heard of these fibers they not as popular like Nomex and Kevlar. But they are inherently flame retardant in case they have to be used.

**(Refer Slide Time: 41:10)**



Not a very popular fiber but the PBI fibers are also very very highly heat resistant fibers you can see the leather structure, double bonded structure quite a lot and so by structure itself they are pretty stable and therefore inherently fire retardant.

**(Refer Slide Time: 41:34)**

## Other Inherently FR fibres

- Noncombustible cross-linked polyacrylate fibre (Courtaulds)
- Panox is an oxidized, thermally stabilized polyacrylonitrile (PAN) fiber.
- Recron FR (polyester), contains organophosphorus compound.

knusba@textile.iitd.ac.in

Other inherently fire retardant fibers, for example cross-linked polyacrylate fibers, so people maybe got motivated to learn that the wool has cross-linked and little bit more resistant and therefore you have cross-linked non combustible fibers polyacrylate fibers, they were commercialized by Courtaulds, one of the fiber manufacturing companies. The other fiber which was quite heat-resistant fiber, it is called Panox made from polyacrylonitrile.

So it acrylonitrile at a certain condition can get cyclized, so the CN CN groups can come together and make a cyclic structure and once you make a cyclic structure that is an oxidized fiber which is much more stable to any heat degradation and actually this is a pretreatment one has to give to the fibers before you can actually make a carbon fiber. So this Panox or this preoxidation sequence which has to be used.

Because to make carbon fibers from acrylonitrile fibers finally the carbonization done a 1000 degrees 1200 degrees centigrade which is required to carbonize them but if you do not do this oxidation correctly the fiber will be smoked, then now I appreciate carbon fiber can withstand 800-1000 degrees centigrade more also. This is one example where the either the organophosphorus compounds.

Now remember one thing it does not have bromine now, no bromine but it is got organophosphorus compound which are either additive or as co-monomers during the



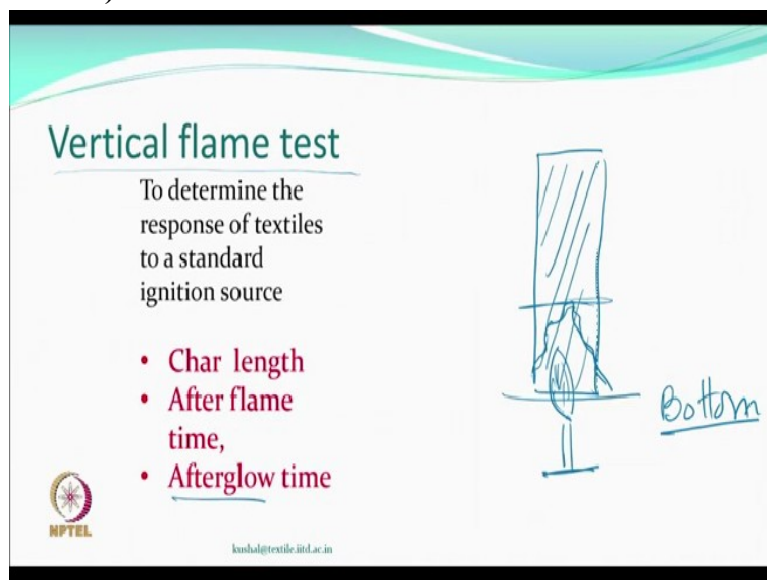
polymerization itself can be added. So one fiber which we know for example is an inherently flame-retardant polyester fiber where the flame retardant is in the fiber itself, during the manufacture either the polymer in polymerization trail or during the spinning stage during the melt. One can add these compounds and so it is becoming more eco-friendly as well you can understand bromine is not here.

**(Refer Slide Time: 44:21)**



Finally, we come to what we call as an evaluation, so there are standard tests available for evaluation, we will just go through some of the principles which are used to test the fibers or the fabric exactly.

**(Refer Slide Time: 44:44)**



One is the vertical flame test you saw a small little video before which where the test was being done, a vertical flame test is one of the very severe tests a fabric can be subjected to burn from the bottom, what it therefore means is, if you burn from the bottom it is the serial it will go up and up and up, so if you burn the fiber for a certain period of time fabric for a certain period of time remove the flame you remember definition remove the flame and then measure.

If it extinguishes or it does not, so even if it is a flame retardant fabric it may burn up to a certain thing and then you can measure the length or time how much time after flame has been removed it gets like you extinguished or afterglow time if it is a cellulosic based material use I told you, you have seen it also, you see some glow which at that point the temperatures are pretty high and so you check them the time. So that is how some of these parameters can be measured.

**(Refer Slide Time: 46:22)**

**LOI: Limiting Oxygen Index**

- Burning from top
- Minimum concentration of **oxygen**, expressed as a percentage, that will support combustion of a polymer.
- Measured by passing a mixture of **oxygen** and nitrogen over a burning specimen, and reducing the **oxygen** level until a critical level is reached.

Handwritten notes on the slide:

- Cotton
- LOI ~ 18
- Wool
- ~ 25
- N<sub>2</sub>
- O<sub>2</sub>

The diagram shows a vertical specimen being burned from the top, with a gas inlet at the bottom. The top of the specimen is labeled 'Top'. The gas inlet is labeled with 'N<sub>2</sub>' and 'O<sub>2</sub>'.

NPTEL logo and email: kudoh@textile.iitd.ac.in

Another is a little more quantitative test more reliable called the limiting oxygen index test, so in this case you have created system where you can change the environment let us say the sample has been mounted inside a jar, this jar, this is the sample and you pass 2 gases oxygen and nitrogen, you know nitrogen does not burn oxygen helps to burn right. So mixture in the normal environment we have approximately about 21% oxygen, rest is nitrogen and other gases alright.

Now what we have to find out is how much minimum amount of oxygen is required to burn the sample and remember in this test the burning is from the top, so you have a flaming jet which will be brought in, so that you start the burning from the top, this jar will ensure that the gas or a

mixture is coming which you monitor. So all over here is the mixture of the nitrogen and oxygen, this proportion you can vary by different control systems, will have control systems.

When you open the valve accordingly and measure, how much is going into the chamber or this glass case and then see the burning behavior. So one important thing is you are burning from the top, so this is the slowest way of burning. Therefore you can measure if there are any changes that happen because if a treatment. So you keep burning changing the concentration if the concentration is low, it will stop burning.

You increase the oxygen concentration at a level will start burning and the burning does not stop till all the fiber or the fabric has been burnt out, that is the minimum level of oxygen required. Let us say cotton if you burn from the top you saw it burns because its LOI is let us say cotton the LOI value may be near 18 and you have oxygen in the environment which is 21%. So there is no way it could sustain alright.

Wool on the other hand has an LOI close to 25% 25 value, 25 value is more than the available oxygen in the environment. So if you burn the wool fabric from the top it would not burn but rest of the fabrics can burn depending upon whether they are above this 21% or below the 21% LOI value if you use this particular test. So what we do obviously I told you that you have a mixture of gases where the mixture ratio has been controlled.

And then you allow it to pass through this jar upwards then you burn once you are sure about the ratio and then check if it burns then you either decrease, so that it does not burn later ok. So that's how I will find the minimum oxygen or limiting oxygen index, little more sophisticated tests can also be performed in equipments called the or instruments called cone calorimeters.

**(Refer Slide Time: 51:11)**

## Cone calorimeter

- A cone calorimeter is primarily used as a standard test and as a research tool
- Measure heat produced
- Heat release rate ✓
- Reduction in mass ✓
- Gases produced CO, CO<sub>2</sub> and others.
- Smoke

Handwritten labels in the diagram: cone, gases, sample, NO<sub>2</sub>, CO, mass measurement.

Logo: HPTeL

URL: kushal@textile.iitd.ac.in

So these cone calorimeter actually burn the sample completely, they believe in that, so they are called cone because there is a cone where there are heating elements and certain amount of heat flux is generated a certain temperature is generated which could be 400 degrees 500 or whatever and theoretically there is a platform or a sample holder which is on some type of weighing machine, it is burnt because the heat flux is coming after the burn happens.


Then there we flame gases etc., they will go through the exhaust and they can be collected at some point to do certain measurements, if you want to find out how which gas has been evolved or which is more older than or which is less evolved things like carbon monoxide you have interested carbon dioxide you have interested you may also be interested in other gases like nitrogen dioxide or sulphur oxides of sulfur. So you may be interested in them, but this cone calorimeter there is a sample here.

So sample you place it this is the heating cone and this is the weight measurement or mass measurement system and so you can measure rate of heat release, you can measure the reduction in mass with time as it is burning you can also see how the carbon monoxide carbon dioxide other gases are formed, if you have your smoke density measurement system you can measure how much the soot or other thing of percentage obscuration can also be measured.

**(Refer Slide Time: 53:27)**

## Oxygen consumption

- Heat release is calculated based on oxygen consumption during combustion.
- Heat Release Rate (HRR) is calculated from the oxygen concentration in the flue gases



kushal@textile.iitd.ac.in

So this instrument in some sense relies on oxygen consumption, so whenever they measure how much oxygen is going in the exhaust, if the oxygen is less that must have burned more or used more so oxygen depletion. Otherwise if exhaust goes in certain way then certain amount of oxygen must be always there. If it is less than that then there is a depletion which can be related to if so much oxygen so much oxidation product and that means whatever you are going to be getting the heat flow. So this is what they do.

**(Refer Slide Time: 54:08)**

## Effect of fabric construction

- ?



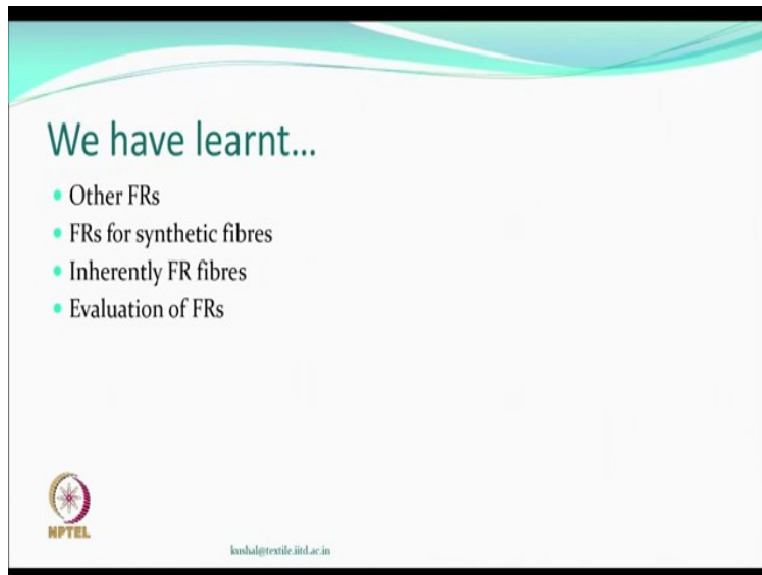
kushal@textile.iitd.ac.in

A final word here will the construction of a fabric make any difference in the burning behavior, the chemistry being the same very lightweight fabric versus a very tightly woven fabric like a canvas, you think you make any difference. Of course it will make a difference because you say

you require diffusion of oxygen for the burning also. So if it is very open oxygen is available everywhere like a ball of just got fiber for that matter.

this is a burn like this because everywhere there is oxygen but if a dense tight constructions, the inside will be relatively more difficult to penetrate and so you might just have less of burning or the rate of burning may be slow it does mean they are safe, but construction would have some effect ok.

**(Refer Slide Time: 55:17)**



So what have you learnt today, we have learnt that there are other efforts that is flame retardants which can be used could be bromine based for synthetics very effective inherently flame-retardant fibers, because of their chemistry and of course we learned something about how to evaluate the flame retardant behavior of or treated or untreated fabrics. In the next class then we meet we will change the topic and go to another finishing treatment called antimicrobial finishing ok, till then have fun, see you thank you.