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Lecture - 32 Texturing of spun yarns contd

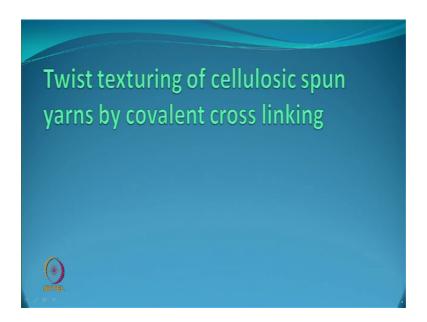
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A step	o back			
	e learnt that twist t techniques,	exturing of spun ya	arns can be accomplishe	ed by
• Sheath	– core technique			
• Introdu	action of thermopla	sticity		
• Decrys	tallization-recrysta	llization		
• Interfa	cial polymerization			
• Covaler	nt crosslinking			
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So, we continue from where we left on the Texturing of spun yarns. And to recall we learned that the twist texturing of spun yarns can be done by some of the following techniques sheath core which means that in the core you have a elastomeric yarn and the sheath is any other yarn which is spun on to that or we can take in the case of cellulosic can introduce thermoplasticity and use the thermo mechanical means. We can do simply by solvents decrystallization, recrystallization in the case of any fiber for that matter cellulosic material as well.

For cellulosic materials you can change the surface by coating thermo plastic polymer which could be done by interfacial polymerization and also covalent cross linking which today we will consider little more in detail.

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So, we focus today on twist texturing of cellulosic spun yarn. So, we are concentrating on cellulose spun yarns of course, regional thing and using covalent cross linking among the many method that we saw that are possible, so we are looking at covalent cross linking.

There was a time when cross linking and various kind of cross linking agents became very important because we wanted to improve the crease recovery properties of cotton or viscose fiber fabrics. A lot of research was done on various agents, it is only after the advent of synthetic fibers coming into the market.

Once they were in the market, then it appears that the crease recovery properties of thermoset thermoplastic fiber fabrics were much better. And so some emphasis was reduced on the cross linking agents, cross linking process which used to give washing wear fabric or durable press fabrics and so on so forth. But of late it has been noticed again that for the reasons that these fiber fabrics are biodegradable and therefore, they become much more important than the synthetic fibers.

And so today again you will see a large number of cellulose based material, dress material, suiting shirting material will be available which says durable press, permanent press and so on so forth. And therefore, emphasis has come back on the cellulosic material and also the way you make them grease resistant and that is done by this covalent cross linking.

So, the question was that why does it happen is because you are able to set some of the deformations or resist further deformation by putting inter molecular cross links.

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The process....? Cotton Viscose Crosslinking Ply twist – set – detwist (past neutral ply twist) • Thermo-chemo mechanical texturing ?

So, theoretically they thought that well why not, we can do this process on cotton and viscose and produce a textured yarn. What it meant were that you are supposed to put in introduced cross links intermolecular cross links with in the fibre so that one can get the twist which is a deformation mechanism set in a better manner.

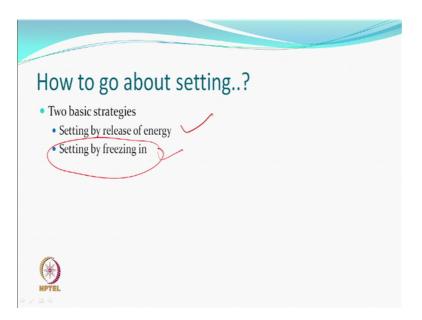
So, the general process would be as we discussed last time also instead of one single yarn you will take two yarns and give them a ply twist and then you do the setting and the detwisting past the neutral ply twist, what it means is that you have done x ply twist in z direction. So, after setting you will give x plus delta x in the z direction.

So, that in the final textured yarn because there are only two yarns they should not absolutely separate out a certain amount of twist which is two to three turns per centimetre or less could be there, so that the yarn actually behave like one rather than two different yarns, so it is a bundle which is important. So, people give slightly more than that detwisting is slightly more than the twisting.

So, that some twist ray you remember even the multi filament yarns we said that either twist or air entanglement may be required just to make sure that they just behave like one body. So, we also said that this process in some way that is the cross linking process in some way, represents thermo chemo mechanical texturing. So, you may be heating to get the reaction done you are using a chemical.

So, it is a chemo and mechanical because you are swirl twisting and detwisting. So, whole process therefore, would relate to this type of a texturing process thermo chemo mechanical texturing.

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So, setting as we learned before also there are two ways in which you do the setting release of energy or freezing in. So, the cross linking is in this category where you are not releasing the energy, you are freezing; that means, after twisting where the internal energy would rise because you are twisted you have put the mechanical energy and you do not allow it to release it stays there as it is fixed frozen in by additional covalent cross links. So, one important thing must remember always that you are not releasing the energy in this process.

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Same and Sam	
Setting by crosslinking	
Freezing-in principle	
Crosslinking agents?	

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Crosslinking agents • Bifunctional agents • Can these be multifunctional?	covalent bond F-R-(F)
NPTEL	c ellulos

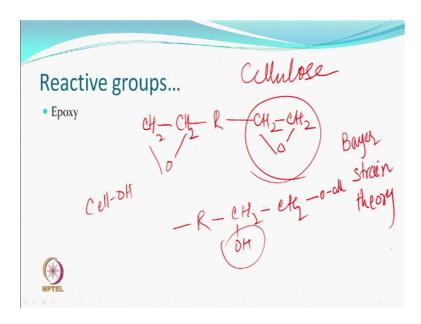
So, again setting is by freezing in principle by using various kinds of cross linking agents. So, what are the cross linking agents can be? A cross linking agent a remember we are talking about the one which will finally, make a covalent bond. The minimum requirement will be that this is a molecule which has got some length or some segment which has got two functional groups at least two functional groups. These functional groups are supposed to react with let us say cellulose and so if they react at two points it makes a cross linked.

So, you cannot obviously, think of making a cross linked by using a by a mono functional agent right, it will only get like a reactive dye makes a one single link, so it does not cross link, but a by functional reactive dye can actually produce some cross links as well because it has two functional groups.

Can these be multifunctional? Yes, it can be multifunctional. You can have more than two functional groups people generally may not prefer in a situation where flexibility is the hallmark. Because, if you have tri functional hexa functional compounds they can make network and so the body or this whole thing become rigid, like your phenol formaldehyde type of situations or melamine formaldehyde.

Melamine formaldehyde can fell is a multifunctional group and it can make very rigid sheets like melamine aware that you have unbreakable plastics what you call them thermo set material, but that will not be good for anything called texturing you need flexibility stretch bulk. So, we should not use multi functional agents as a cross linking agent.

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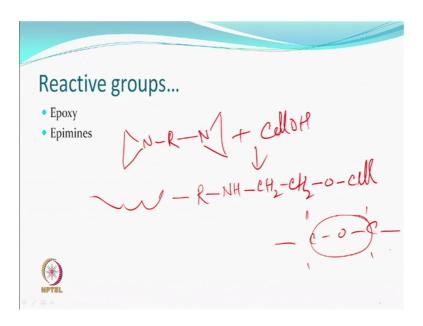
So, some of the groups which can easily react with let us say cellulose we are not discussing cellulose right we are discussing how to texturize cellulose spun yarns. So, some of the reactive groups which can react with cellulose can be listed like this epoxy right, epoxy groups you must have seen a product called erudite right.

So, if you have their epoxy based reasons they can cross link, they can make hard things also, but if you ensure that only reactions are limited and not in a three dimension things you will get flexible things also some these epoxies may look like this. So, you may have R and you can have another epoxy. So, as a try membered ring you know if you have heard of Bayer strain theory.

They are very reactive, they are more unstable it is because 3 membered rings are generally very unstable and they are therefore, reactive they can react, what will happen; obviously, is this 3 member ring is going to break up and theoretically can react with cellulose, if there is a cellulose somewhere which we can represent as cellulose and one hydroxyl group then.

So, this type of a link can be formed and this somehow leaves the hydroxyl group behind as well. So, in one way one can say well it blocks one of the hydroxyl groups of cellulose, but simultaneously creates an other hydroxyl group, so hydrophilicity may not be affected right. So, overall hydroxyl groups could be there.

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So, the other similar type of a group are in the series which we call as epimines; epimines are again 3 membered ring which a nitrogenous compounds. Here again is the three membered ring and therefore, it can break under like epoxy generally would be more reactive in alkaline medium.

Epimines can react in acidic medium and one will be able to react and get some link of this type, if cellulose is reacted and of course, on this side also something similar would be there. So, you can get a cross link which in a way you can consider the ether link. So, this is a ether link, this can be also generated. So, one can produce cross links in this manner.

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Reactive groups	
• Epoxy • Epimines • N-methylol • Acid chlorides • Isocyanates O = C = N - R - N = C = D	

The other interesting group which have been very effective and commercially actually the textile people use them relate to a class of called the N methylol compounds. So, you have again a nitrogenous compound. So, this is the N methylol group, so you can attach with n e r and have two such groups, then they can also react with cellulose to make cross links.

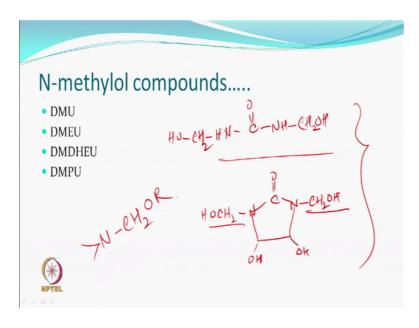
Acid chloride there also if remember sometimes we talked about very reactive compound which can go for interfacial polymerization also with any type of acid alcohols and so on so forth, they can also react, but people have tried isocyanates also. So, one can have type of groups which can also make cross links they are very reactive compounds and they can form there are other compounds also which have been used to produce cross links.

But as I mentioned this group has been commercially more successful because the conditions for generating cross links suit the normal textile process where the time and temperatures are in your ranges save your work. Epoxies epimines etcetera probably will

be working through liquids and solutions you may or may not be very happy in doing cross linking solution will take more time.

Acids and isocyanates, acid chlorides isocyanates are dangerous in some sense I have got to be very different very careful when you are using them, but they are generally relatively much more safer of course, there are other than these compounds also people have used for cross linking purposes.

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So, N methylol compounds, the first such compound which somebody said produced was from a urea. So, you are simple urea, so you have die methylol urea which meant molecule like this; this is urea with 2, but if you add CH 2 OH here and CH 2. So, compounds like this we generated which were acid catalyzed.

Another series of compound which came be called the DMEW which is dimethylol ethylene urea, another compound which again became quite commercial success was called the DMDHEU which is dimethyl, dihydroxy, ethylene urea the compound something like that. So, this compound and the previous compound the difference is this is linear and this is a cyclic compound

So, this by itself is a five member ring, so relatively more stable it does not break the reactions are going to be taking place with these N methylol because nitrogen's got lot of lone pair and therefore, it is easy for reactions to take place with the hydroxyl groups of

cellulose. Well similarly people went on to say instead of ethylene you have a propylene urea, propylene hydroxide urea not only that instead of methylol they talks of compound which are N CH 2 instead of OH some other alkyl group, so alkylol N alkylol compounds could also have been use. It is basically to balance what they call as a reactivity versus stability.

So, in some cases reactivity may be high stability may not be so high, stability means basically hydrolytic stability. All such material the after being applied will have to be all textiles will be washed many times over. So, if the cross link breaks then; obviously, is not effective hydrolytic stability is one which people like to work round.

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Reaction mechanism with cellulose... Acid catalyzed reaction $> N-CH_2OH + H^+ \longrightarrow N-CH_2OH$ $\int -H_3$ N-CH2-0-Cell -H+ N-C92

So, the general mechanism of reaction with cellulose we are now looking at the N methylol compound which I said they became relatively more commercially successful compounds and they are still. So, acid catalyzed if you have a reaction something similar. So, this proton acts and adds itself and later in the reaction a water molecule goes you get a carbonium ion which is reactive, this can react in the presence of cellulose to give something like this, a link and in the process releases the proton back.

Therefore, is catalytic, so you are not consuming the catalyst as such it goes and come back and you get the reaction. So, this is what in the meantime you; obviously, are removing one water molecule every time, so there is a substitution reaction and again forming an ether link. Ether link is relatively more stable in alkaline conditions and therefore, when you wash you would have better hydrolytic stability compared to an ester link because most are washings our soap detergents are alkaline and so in this can.

Cyclic crosslinking agents... • Why? ~ - c'-NH-enz-v-cell

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So, the cyclic compounds became more popular than the linear compounds because although the reactivity was reduced, but hydrolytic stability improved and so a large number of them are going to be the cyclic compounds. And the linear compound also had another problem which means that if we look at urea for example, formaldehyde if this is the kind of reaction that happens.

So, this part which is got a hydrogen on the nitrogen this is called a labile hydrogen, in a cyclic compound this labile hydrogen it is not there. Labile hydrogen nitrogen having lot of lone pair of electrons, this can easily get replaced by if you have a bleaching solution somewhere it will become NCL instead of NH.

And will keep coming out whenever you do ironing or any another thing HCL can come out and then degrade yellow the fabric, so this was called a chlorine retention problem. So, whenever you have a labile hydrogen on nitrogen you may have a chlorine retention problem and cyclic compound therefore, do not have such issues and so a good number of such agents therefore, have been used. (Refer Slide Time: 25:46)

• Pad - dry - cu • Wash?	ing Process	∑ 120-150 e
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So, the cross linking process is for setting pat dry cure this is the process which is used for the fabric and if you have to use for texturizing also the same process has to be. Padding means you have a solution of a cross linking agent and a catalyst also and they have to be applied on to textile, one of the easiest way applying is padding and then you dry and then a cure, purpose of drying is to remove water and purpose of curing is to generate a cross linked.

So; that means, these things are going to be done at two different temperature ranges, this drying may be better less than 100 degree centigrade aim is to remove water. And the curing; obviously, will be somewhere 120 to 150 degree centigrade where we expect in general reaction may take place. So, do we wash? We have to wash.

So, this is therefore, different than the normal texturing process for thermo mechanical texturing of thermoplastics because in every reaction you can have unreacted compound, then you have added a catalyst which; obviously, is just a catalyst for reaction after that has no purpose to serve. So, you will be washing drying.

So, additional steps may have to be taken if you want to complete this setting process and then you will do the detwisting. So, you have the twisting part then a setting which is a long process which is messy in the sense that it is got liquid, water drying washing all those kind of things and then detwisting. So, if you do all those then you are likely to get textured yarn; so, just some word about the catalysts for the same type of compound which are the N methylol compounds.

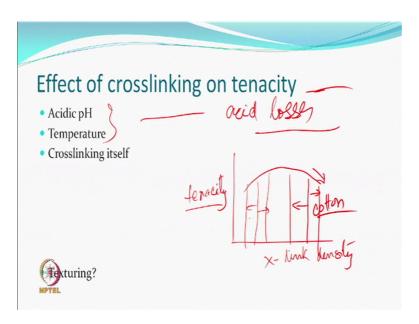
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So, acids can be used because they give proton when relay acids can be used HCL S 2 SO 4 can be use nitric acid people do not use too strong; organic acids like citric acid, glycolic acid and such compounds can be used for catalysis purposes, the only thing is that they are active; obviously, they are ready to give proton.

And so some strength lost due to the acid itself can happen and so people would like to not use the acids directly for catalysis will use something else then catalyst strength loss, I am sure you know the cellulose can be dissolved or at least there is a test method in the sulfuric acid, so actually it can degrade. So, it has to be used as a catalyst and when you go for higher temperatures also strength loss can occur.

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So, one may like to remember that whenever you do cross linking in a manner in which we have just described, the tenacity is going to reduce because of the acid, so we call them acid losses. We are talking in terms of tenacity, no talking on the mass. So, cellulose is sensitive to acid all ether links are sensitive to acid.

Then temperature we are saying curing temperature is going to be high, so because of acid plus temperature again the loss can take place, the time room temperature the difference it will be different and higher temperature it is different. You see when what happens is when you dry whatever concentration of acid that you are taken in the beginning on the fabric as the water goes out the concentration of acid keeps increasing.

So, acid can become concentrated on the substrate and then can do, but if they become concentrated only at the time when cross linking is required, then they are participating in reaction if it happens before then it can right. For example, if you do not use the cross link agent just have acid and temperature you will get the loss, higher than when you have a cross linking agent. The cross linking itself is supposed to bring some loss, but that depends on which fiber we are talking about right.

So, this is just by cross linking even if there is no acid loss, if there is no loss due to temperature or higher temperature just by cross linking you can get something like this particularly in cotton you observe this. This is a hypothesis proposed by a Ribbon field and what it says is that if you have less cross link density if you keep on increasing cross

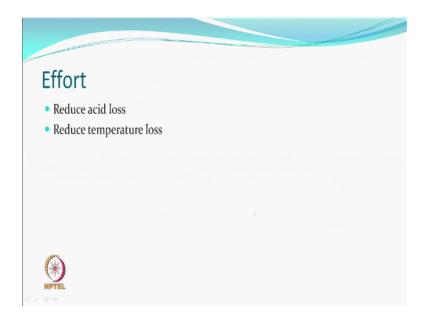
links, the tenacity of let us say the yarn or the fabric will increase after a certain point it will start decreasing and as they say the cotton actually lies here.

So, when you increase cross link, so you are going in this direction. So, you will lose just because of this, it is reverse happens when you decrease; when you decrease let us say by wetting. So, hydrogen bonds so this cross link is not only chemical crossing is representing everything intermolecular all kinds of bondings like hydrogen bonds reduce, so the strength increases. So, you see the cotton strength in wet state is higher than the dry state, so this is one.

On the other hand if you have this course which actually has a low crystallinity and so on so forth overall cross link density less in this case reverse happens that if you do the increase the cross linking the strength can increase, if these two have been controlled if you wet the wet strength of this course is low right. So, you can; obviously, have this in addition the texturing. So, what is texturing doing?

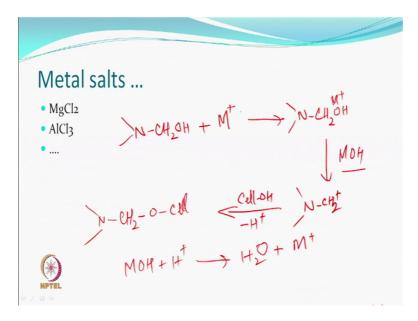
It is twisting first, it is not like the fabric or a fiber or a yarn in a relaxed state it is actually not in a relaxed state you are twisted and you are going to be cross linking it twisted yarn because that is what the texturing has to be used for this is how the texturing will be done. So, what it means is a material has been subjected to stresses and the stresses are being stored in and so this material will become weaker in that sense also who likes to be under stress.

Any material under stress can fracture in fact and so tenacity after cross linking or let us say after texturing by this method is likely to be less and it is nothing to do with what we were talking about was disorientation you know that is another thing here intermolecular changes are not taking place there as it is, but whatever you are doing is also not very helpful effort tenacity is concerned. (Refer Slide Time: 34:06)



So, effort therefore, is what to do? You can reduce the loss due to the acidic environment or reduce the temperature of cross linking and therefore, you can do, but cross linking by itself whatever it does it will do, you may not be able to change that part.

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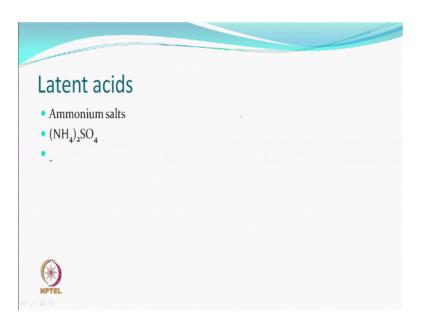


So, instead of acids people suggested using metal salt metal salt becomes effective because of the metal, the higher is the oxidation number of the metal higher is its activity. So, people do not use aluminium chloride generally because it is very active and you may have more degradation because of this activity, but settling down people settle down at magnesium chloride, zinc chloride etcetera.

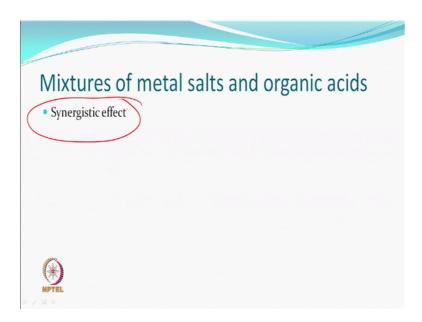
Zinc is relatively a heavier metal, so magnesium is not so heavy metal this is one of those popular catalysts which has been used and it works in the same manner as the proton base system say a metal with whatever oxidation number that you have, if you have more it will work like that. Metal hydroxide we come out and you get again a carbonium ion after that is the same reaction get the have proton back you.

So, they also become considered to be more active because this MOH can react with the generated proton and can produce H 2 O plus metal ion which can go like this or the proton itself can catalyze. So, therefore, the probability becomes of effectiveness is good, but it is not more degrading because this process starts only after a certain temperature, it does not you can sort at room temperature not much thing will happen, but if you source sulfuric acid based fabric at room temperature things will keep happening at room temperature is well. So, that way it reduces the strength loss and still effective.

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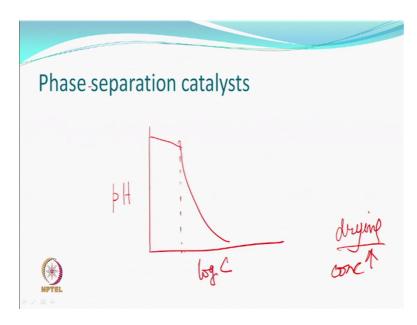
Similar compound like, latent acids which ammonium salts they at room temperature are not very effective, but at higher temperature ammonia will be deliberated and suddenly they will become effective. (Refer Slide Time: 37:55)



So, people also you know try to use metal salts along with organic acids; organic acid by themselves catalyze metal salts can catalyze, but when they are present together, then the complex can be formed where the metal ion is handled by the organic acid or organic acid remain complexed along with the metal.

And at a later stage both of them are available and what people have seen with this type of thing is the temperature of the curing can be reduced from 150 degrees to 120 degrees 110 degrees which means less loss in strength people look concerned about the loss in strength. L is one part of it, but if it becomes yellow also you know then you see it, so you may not like to that.

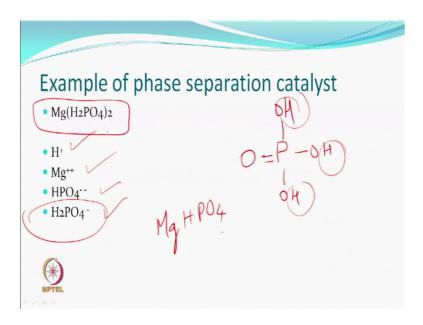
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Another very interesting class of catalyst is called a phase separation catalyst phase the separation; that means, during this process of curing, the catalyst separates in two phases and the pH of the solution or wherever it is; obviously, as you do the drying if you do drying that is the moisture is going up the concentration keeps on increasing.

So, pH goes down, but at some point it falls rapidly and that is the point which is a critical concentration where phase separates and so activity starts increasing and so this again to reduce the loss due to acids.

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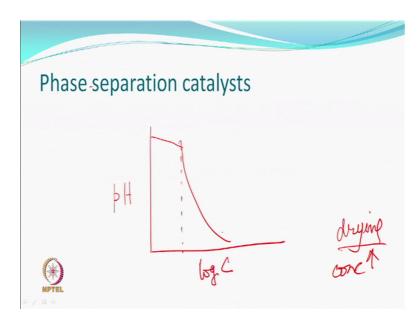


One of the example of a suppression catalyst is hydrogen phosphates, so magnesium hydrogen phosphate, aluminium hydrogen phosphate they are called the phase separation catalysts it is because of the phosphoric acid being such a nice acid that hydrogen can come from here; can come from here; can come from here if the phosphor case is concerned, but we react with let us say a magnesium ammonium and so on so forth, these activities are subdued.

So, if you have a magnesium dihydrogen phosphate which is an example given. So, in a solution where this has been dissolved one can have hydrogen ion which is interesting, you can have magnesium ion which is a metal ion so which is interesting, you may have hydrogen phosphate ion or you may have a dihydrogen phosphate ion all of them are present depending upon dissociation that keeps on happening at any given point of time.

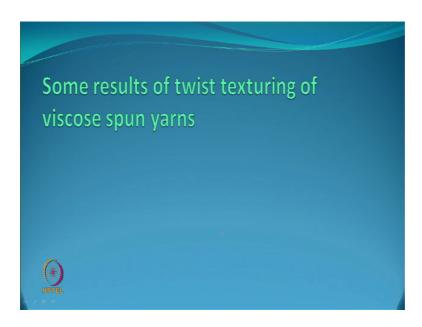
It so happens; that means, you have a magnesium, hydrogen phosphate as well as magnesium dihydrogen phosphate available in the solution as the dissociation is concerned and as you dry at some stage the solubility of the magnesium hydrogen phosphate is less, in water it precipitates and the other one becomes more active. In the beginning most of this is because of this and so it works like a weak acid.

The moment the other compound which is magnesium hydrogen phosphate which is from here which is, this has a low solubility it precipitates and therefore, equilibrium shifts and more of more of protons are produced and we will get a phase separated and a pH change which would look something like this. (Refer Slide Time: 42:22)



This type of compound has been also found to be a very good as for a less stable under less degradation and is otherwise quite stable to the what we call as a temperature if it was a ammonium phosphate dihydrogen (Refer Time: 42:43) with ammonia would get liberated magnesium does not get liberated in that manner and one would see good cross linking.

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and the second s		
The process		
•		
 Ply twist- set-detwist 	(past the neutral plytwist)	
 Setting 		
0		
 Pad – dry – cure –was 	h –dry	
 Typical recipe 		
 Resin (DMDHEU) 	20% (w/v)	
 Softener 	1-2 % (w/v) .	
 Catalyst 	1-1.5 % (w/v)	
Wetting agent	0.1-0.2 % (w/v)	
NPTEL		

So, we just see some of the work that has been done in this. So, the process if this experiment was done is a ply twist set detwist past the neutral ply, so setting was done by pad dry cure wash dry method I am just trying to tell you why we do drying we go drying means low temperature removal of water you can dry at high temperature also. But that is what is the standard one some reason which could be DMDHEU any other reason, some softener because invariably people have found that some stiffness occur.

So, softener will do the reverse of it a catalyst whatever catalysts that you may like to use some wetting agents everything it is surface tension reduced and so on so forth. That type of a thing can is a starting point, so you do the twist and the twisted yarn is parrot right and then dried cure washed before detwisting this is typical change the resin, change the catalyst concentration is that everything will change so any kind of a portion where treatment something like this one would see.

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Crosslinking conditions • Curing temperature • High temperature curing • Flash curing P-D-C
NPTEL

So, then comes the curing conditions now we are just trying to emphasize based our need of texturing also. So, normal curing conditions after drying could be let us say 120 to 150 degree centigrade. Now, here general time requires are could be 4 minutes to 5 minutes or 3 minutes to 5 minutes. While we were discussing times of the order of point 1.2.3 seconds.

Now, you are talking about minutes right time here is in minutes. So, you may have 3 to 5 minutes as a time. So, that people thought if we have this therefore, it has to be a batch process then nothing else can be done and will be more and more you know unattractive. So, people also have what we call as a high temperature curing; high temperature curing means that you can go beyond 200 degree centigrade.

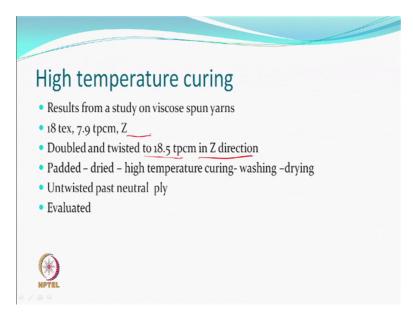
Considering the time temperature issues; that means, at high temperature time would be reduced if the time comes in seconds, then there is a possibility that you may actually have continuous process also in a minutes. And can think of continuous process with it is like a standard you know doing all kinds of things there is a long sighs look at this texturing machine one simple heater and going through the whole thing versus a whole standard business which people use for the textile fabric.

So, high temperature curing is actually believe this is pad dry cure this also is pad dry cure, but at higher temperatures. The another term which is called a flash curing which pad cure, their aim was that well if you are going to higher temperature drying will also

take place right. But people would prefer drying step in between because if a higher temperature rate of drying is very high in some sense it is good, but because of that high rate some of the chemicals can migrate from inside to the surface as they come in, so migration in the non direction.

So, you have a non uniform cross linking, if you have a non uniform cross linking you can understand what will happen to the properties. So, cross linking is a bulk change, property change in bulk like thermo mechanical also change in the whole bulk, so you cannot have only surface being treated or treated more than the rest. So, people may not prefer flash curing as such, but it may go for high temperature curing; that means, you dry initially at a low temperature and then cure at a high temperatures that you can go faster.

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So, one example which I am just listing here is some of the viscose yarn which was some 18 tex, 7.9 twists per centimetre twisted in Z direction, they see on was doubled and twisted to some twist level like 18.5 twists per centimetre again in Z direction this is an important thing you know. So, even if you do ply twist you are not supposed to do the ply in the reverse direction you may do a bad job some of the yarns may start opening up.

So, Z on Z right, so you do Z on Z; obviously, when you will do the reverse twisting it will be in s direction that is it. So, you pad dry high temperature curing; that means you

are going for padding, drying and higher temperature curing washing and drying untwist past the ply and then see some of the properties.

Result	S				
Catalyst	Conc (m mole %)	Nitrogen (%)	Breaking stress (K- Nm/kg)	Extensio n at break (%)	Crimp rigidity (%)
MDP	15	1.12	109.5	9.4	10.7
Control	-	-	120.0	12.7	-
NPTEL			Curing tem Time 2.5 se		10 °C

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So, some result here curing temperature of 210, time was 2.5 seconds; 2.5 seconds, the catalyst here is magnesium dihydrogen phosphate which means this is phase separation catalyst all right. Some concentration measurement of nitrogen has been done because when the cross linking agent gets added you do have a nitrogen also getting added, so we can measure nitrogen.

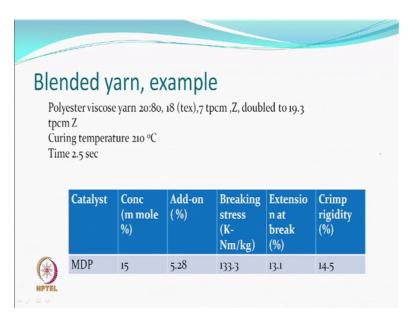
So, some strength loss you can see from a control 120 209, extension at break also reduces and some crimp rigidity has been obtained; that means, this material is now a textured yarn which; obviously, has got helical structure and you can do stretch and bulk both.

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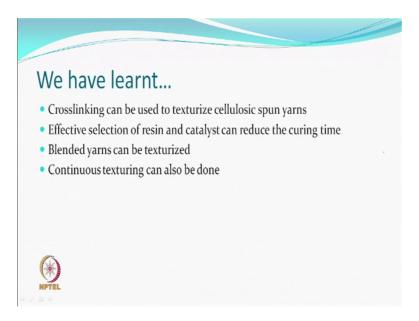
So, if you have this process coming up to this level, then can use a blended yarn also like polyester viscous polyester cotton, if times are less than you may think of false twist texturing, if possible you can think of texturing and dying together because there is a process called procion resin process were in acidic medium the reactive dyes can react through the resin and die also. So, one can think of you go for a high temperature curing you can go for a mixture of dye and resin and theoretically you can do continuous dyeing and texturing.

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So, one example here is where less polyester has been taken more viscose, but if you change the percentage of polyester; obviously, thermo mechanical setting of the polyester will help, if it is more of viscose you have to have more of cross linking agent. Similarly done 210, time 2.5 same catalyst it is a blend, so you can do texturing as well.

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So, what we learnt is that cross linking can be used to texturize cellulosic spun yarns, effective selection of resin and catalyst can reduce curing time, blended yarn can be texturized, continuous texturing can also be done; so some conclusion from that point of view.

Thank you.