Mechanical Characterization of Bituminous Materials
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Module No # 05
Lecture No # 24
Aging of Bituminous Binders and Mixtures – Part III

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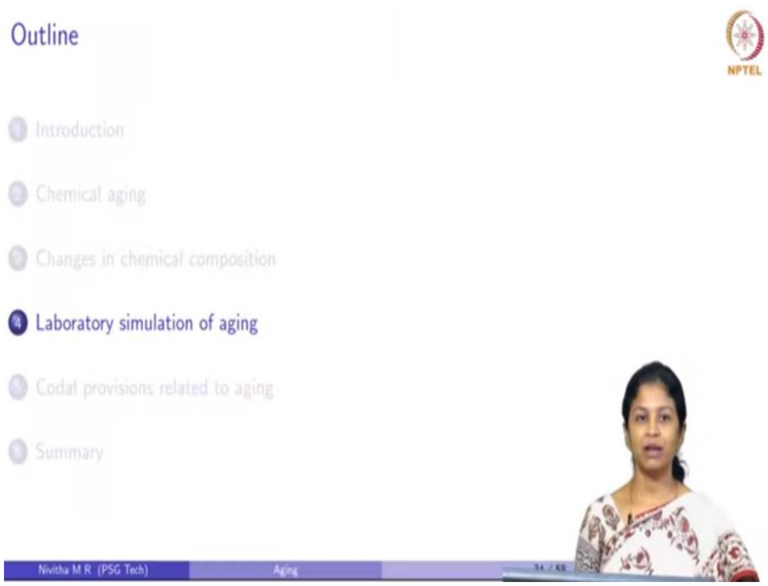

In today's class we will continue the second half of the aging. So in the previous class we have defined what aging is? We have seen two types of hardening in bitumen: the reversible hardening and irreversible hardening. So we said irreversible hardening is generally defined as aging or chemical aging. And then we have seen what chemical aging is? It occurs in two stages which is classified as short term aging and long term aging.

Then we moved on to the changes in chemical composition that occurs in bitumen on aging. So we said the formation of carbonyl and sulfoxide compounds are the predominant reasons for aging in bitumen. And the formation of these two compounds are influenced by temperature and the availability of oxygen. So in today's class we will see the methods available for laboratory simulation of aging. Aging in bituminous binders and aging in bituminous mixtures.

So why do we need this laboratory simulation of aging. So as we have defined earlier, aging is a process which happens over a period of time. So if we want to quantify the effect of long term aging we have to wait for about 7 to 8 years. So it is not practically possible to wait for 7 to 8 years, collect the data and then evaluate the performance. That is also needed but we need to do some accelerated testing in the laboratory and quantify what is the effect of aging on bitumen.

So for that purpose we have laboratory simulation of aging and we do it separately on binders and mixtures. So first we will see what is the procedure available to simulate the short term aging in bituminous binders.

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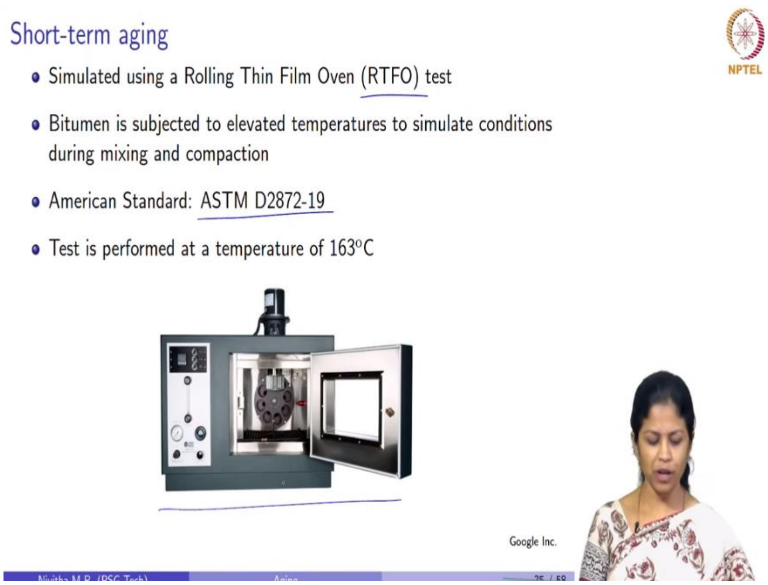

So this short term aging is simulated using a rolling thin film oven. So this is in RTFO. So previously when I showed you some results on elemental composition and molecular structure, we had seen RTFO and PAV. So this RTFO is a test which is used to simulate the short term aging condition in a binder. So in this procedure bitumen is subjected to elevated temperatures and the presence of oxygen and aging is induced in this material.

So this test is done as per American standard ASTM D2872. And this test is performed at a fixed temperature of 163 degree Celsius. So this equipment what we see here is a RTFO equipment. So I will explain you how this equipment works.

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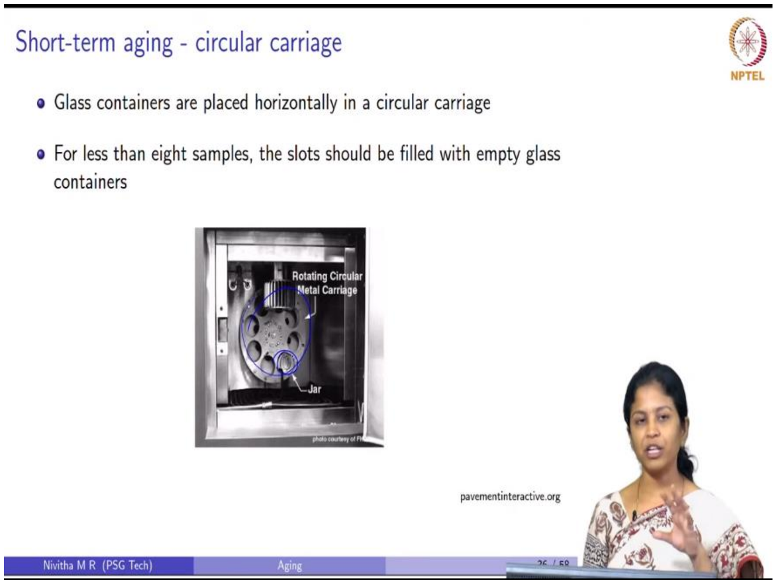

So there is a circular carriage which is present here which is capable of rotating. The samples are placed inside this circular carriage in glass containers. You can see a jar here it is an empty jar. So this jar has to be filled with bitumen and it has to be placed in this circular carriage. And this RTFO equipment is a temperature controlled oven. So once we have some temperature control sensors inside we will be able to maintain the test temperature throughout the test period.

We also have an air flow vent which will supply oxygen into this oven. So we have an apparatus where we can control both temperature and the supply of oxygen. So here in this circular carriage bitumen is placed in these glass jars. So there are some 8 slots we can see here and bitumen filled in the glass jars are placed in all these slots. So this circular carriage is capable of rotating.

So why do we have this rotating circular carriage? So previously when we were discussing about the chemical aging we said when we have bitumen spread in a tray only the surface of the bitumen will be subjected to oxidation and it forms kind of a skin which prevents the entry of oxygen molecules to the lower layers. So for this purpose we need to avoid this formation of skin. So we have this circular carriage which is rotating which will kind of move this material continuously and provide a chance for homogenous oxidation in this material.

So for less than 8 samples here these slots should be filled with empty containers because the oxidation happens at some specific temperature. And that temperature distribution inside this oven depends upon the contents in this oven. So if there is empty place here the temperature distribution will be disturbed. So for even distribution of temperature in the oven we need to place empty jars where sample is not placed.

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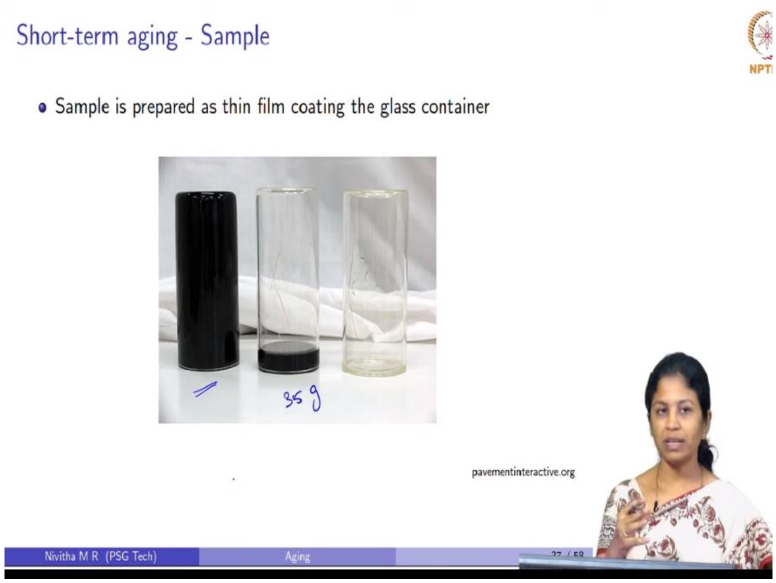

So how do we prepare this glass jar coated with bitumen. So we need to take this glass jar fill it with 35 grams of bitumen and then we need to continuously roll this glass jar so that it is completely coated with bitumen as shown here. So this glass jar has now to be placed in the slots which is provided in the circular carriage.

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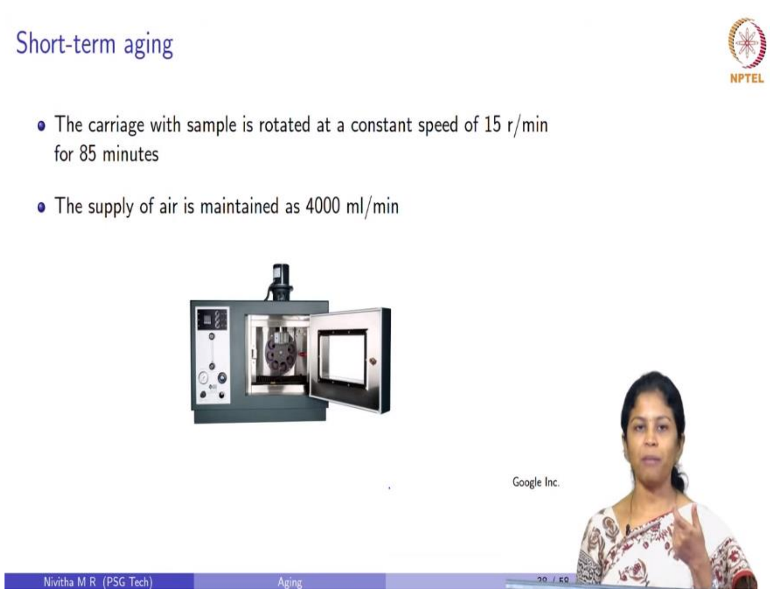

And this carriage with the sample right, so in all these 8 slots here we fill it with glass jar coated with bitumen. So this carriage is rotated at a constant speed of 15 revolutions per minute for 85 minutes. And the air supply is maintained as 4000 millimeter per minute. ASTM provides specifications related to the variability allowed in case of the temperature of testing, the air supply that is provided to this equipment and the number of revolutions. So we need to ensure that any type of equipment used to simulate the short term aging is operating within this allowed variability.

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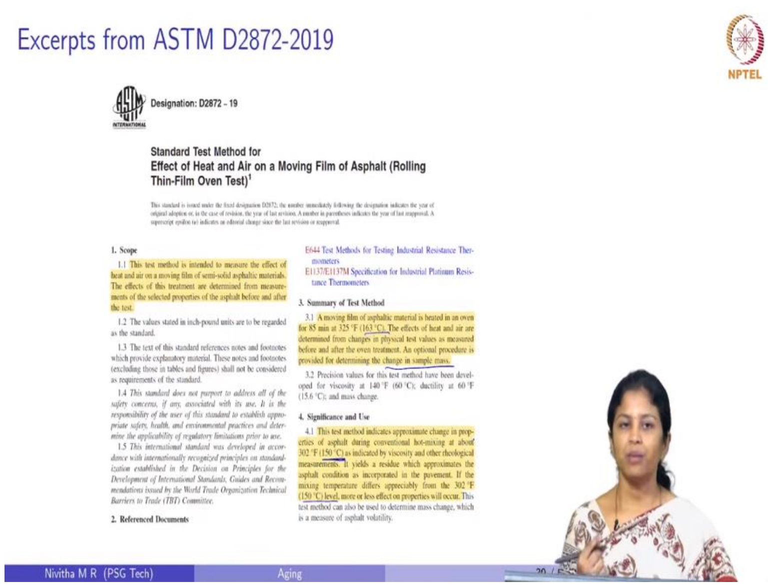

So I have shown you some excerpts from ASTM D2872 to show some important factors related to this simulation of short term aging. So first let us look into the scope. I will read the text: This test method is intended to measure the effect of heat and air on a moving film of semi solid asphaltic materials. The effects of this treatment are determined from measurements of selected properties of asphalt before and after the test.

So this is only a treatment which is applied to this material. So at the end of short term aging we have to measure the properties of this material and then compare it before and after short term aging. The next thing is the summary of this test method. So this says in 1 or 2 lines about what this test is? A moving film of asphaltic material is heated in an oven for 85 minutes at 163 degree Celsius. The effects of heat and air are determined from changes in physical test values as measured before and after the oven treatment.

And there is also an optional procedure provided for determining the change in sample mass. So we initially place some x amount materials in the containers; after it has been subjected to short term aging we take out the material and measure the loss or gain in mass. So the loss in mass will tell how much is the volatiles that has been evaporated from this material. The next portion says the significance and use of this method.

So this test method indicates approximate change in property of asphalt during conventional hot mixing at about 160 150 degree Celsius as indicated by viscosity and other rheological measurements. It yields a residue which approximates the asphalt condition as incorporated in the pavement. If the mixing temperature differs appreciably from 150 degree Celsius level more or less effect on the properties will occur.

So we need to make sure that this is to simulate the mixing which happens at about 150 degree Celsius. So if we use higher mixing temperatures then we need to ensure that this method will give us only a lower value or a higher value for the oxidation products. So in the case of modified bitumen generally a higher mixing temperature is suggested. If it is 165 degree for the base bitumen, about 180 degree Celsius is suggested for certain class of modified binders.

So in such cases this particular method will not simulate the aging which happens at the end of short term aging process. So we need to make sure that for modified binders we need to use some other kind of aging process. But as of now there is no alternative which is available for modified bitumen. So even for modified bitumen we are now using this short term aging procedure only.

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The next one is that after removing the residue from each of the glass containers it is stirred gently and it is collected in a container to homogenize the residue from different containers. And it should be done without introducing air into it. This residue what we get from this short term aging procedure has to be tested within 72 hours of performing the RTFO test. So once we get the residue we have to test it within the test period and if this 72 hours is expired we have to prepare fresh sample for testing the physical properties.

And the report here is we need to report the results from RTFO test in terms of the change in physical properties. I will show you some codal provisions where they say this is the property that is desired after short term aging of this material. So that is what is the result that we get from short term aging. If we measure the change in mass loss this change in mass loss is an optional procedure. If we measure the change in mass loss, then we can report that and you should see that it should be reported to the nearest 0.001%.

So that is the precision to which the change in mass loss has to be reported in this case. And if we have a mass loss it should be reported as a negative number and if there is a mass gain it should be reported as positive number. So this is a summary of the short term aging procedure.

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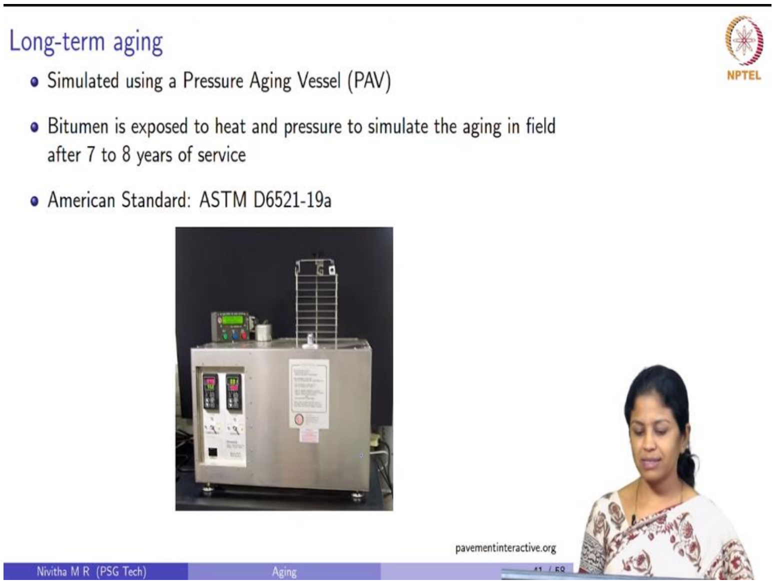

Next we will move on to the long term aging procedure. This long term aging is simulated using a pressure aging vessel. So here bitumen is exposed to heat and pressure to simulate the aging in field which happens at the end of 7 to 8 years. And this test is based on the American standard ASTM D6521 the 2019 version. This equipment which is shown here is a PAV vessel. I will tell you how the sample is prepared for this particular process.

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So the long term aging simulates the aging after it has been laid and compacted in field to some 7 or 8 years after service. So we have to use the short term aged samples to long term age them. So whatever the residue we get from the short term aging procedure, the RTFO procedure, that sample is used for subsequent long term aging. The sample has to be poured in trays so this is a tray which is of a specified dimension. Again the ASTM code has lot of details related to this sample tray, what are its dimensions and how is the approximate thickness of the sample in the sample tray and what is the pressure, temperature, lot of details the code has.

So this sample has to be placed in this aluminum tray and this tray is stacked here inside this particular container and that is placed inside the PAV vessel. So we can see here there is a slot here. So this tray is placed inside this slot here and we have to maintain a constant temperature and a constant pressure inside this vessel. So this constant pressure is maintained to ensure diffusion of oxygen molecules throughout the sample.

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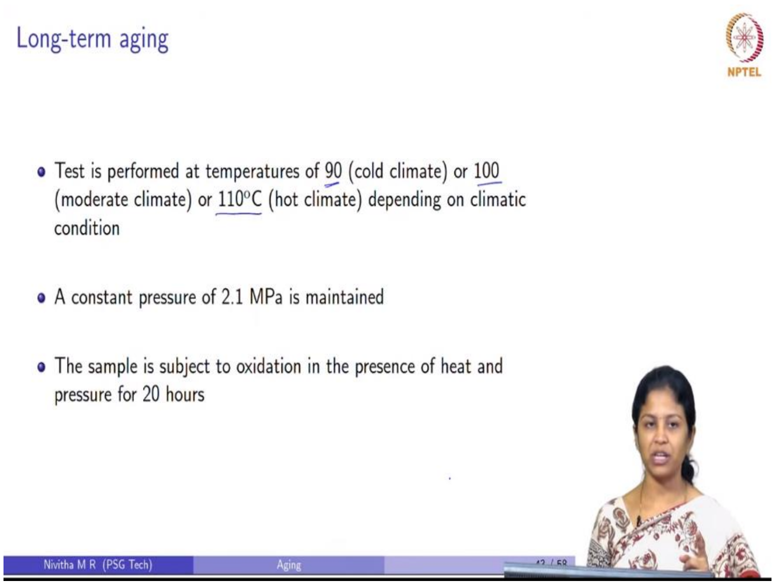

And this test has to be performed at a temperature of 90, 100 or 110 degree Celsius. The climatic conditions of the pavement where the material is going to be used determines the test temperature. If the climate is very cold, a test temperature of 90 degree Celsius has to be used. If it is a moderate climate 100 degree has to be used and for very hot climate 110 degrees Celsius has to be used.

In the previous short term aging procedure we saw that it is done at a constant temperature of 163 degree Celsius; because for most of the bitumen we use 165 as the common mixing and compaction temperature at least in the case of unmodified bitumen. Whereas when the mixes laid, it is subjected to environmental conditions and the environmental conditions are going to be varying depending upon the location where the mix is used. So to take that into account we are using different temperatures depending upon different climatic condition. So a constant pressure of 2.1 mega Pascal is used and the sample is subjected to the effect of temperature and pressure for a duration of 20 hours.

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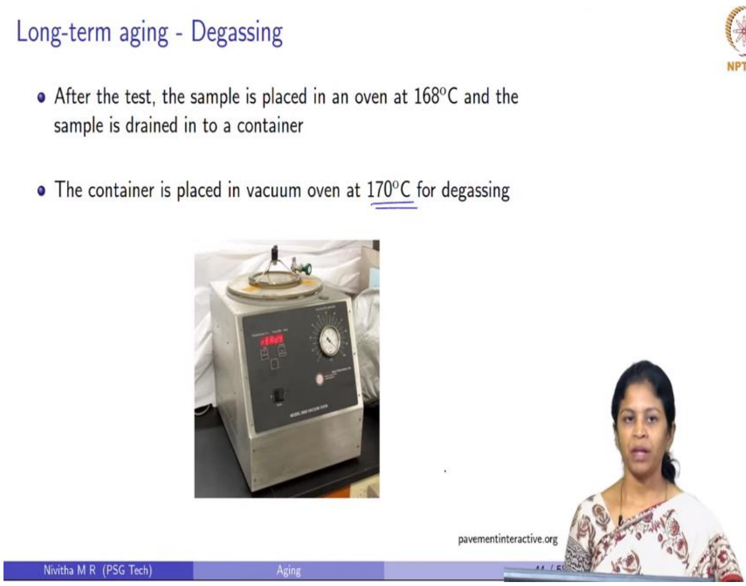

At the end of 20 hours the sample is removed, placed in an oven at 168 degree Celsius and it is drained into a container. This sample is then placed inside a vacuum oven at 170 degree Celsius for degassing because when we perform the long term aging we subjected it to a pressure. So for the degassing purposes we place it inside a vacuum oven. So at the end of this process, the sample is now ready and it is used to represent the condition of bitumen after it has been laid in the pavement for about 7 to 8 years.

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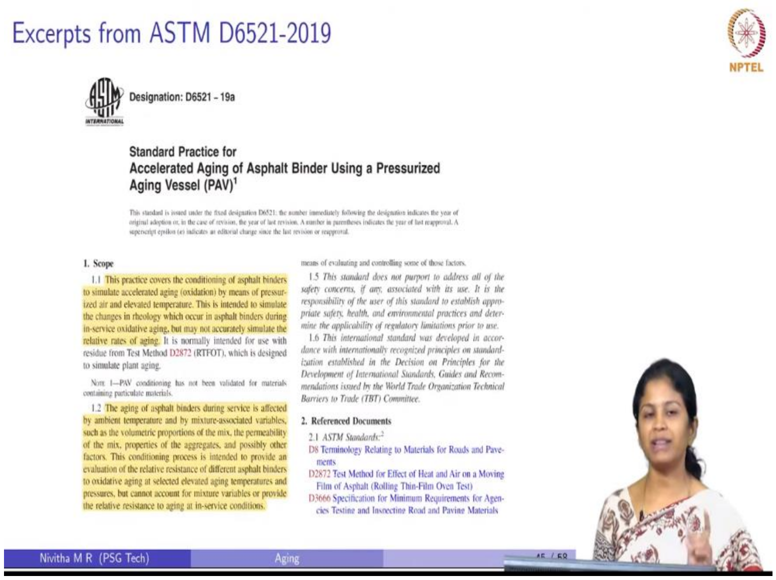

Now I will show you some excerpts from ASTM D6521; this particular code has lot of if's and but's. So we need to be aware of these facts before subjecting the material to long term aging based on this particular code. Again the scope: this practice covers the conditioning of asphalt binders to simulate accelerated aging; they are specific only due to oxidation; by means of pressurized air and elevated temperature. This is intended to simulate the changes in rheology which occur in asphalt binders during the in service oxidative aging. But may not accurately simulate the relative rate of aging.

So what they say is at the end of the 7 or 8 years it will gives us what is the extent of aging in the material. But it will not give us any idea about how the material is aging over the 7 or 8 years period. If we read the next one the aging of asphalt binders during service is affected by ambient temperature and by mixture associated variables such as the volumetric proportions of the mix, the permeability of the mix, properties of the aggregates and possibly other factors.

So they say that the aging is not only because of the availability of oxygen alone as we had seen earlier. But in the mix, it is going to depend upon lot of other parameters like the volumetric proportions of the mix; permeability which is nothing but the air void and the properties of aggregates and other factors. So this conditioning process is intended to provide an evaluation of the relative resistance of different asphalt binders to oxidative aging at selected elevated temperatures and pressures.

Let me pause here so what they say here is this method can only simulate the relative oxidative aging. So if I have a binder A and a binder B, this will tell me whether binder B will age more compared to binder A. So that is what they mean by relative rate of aging. But at the end of 7 or 8 years if I get some stiffness I cannot say that, that will be the precisely the stiffness that will happen in field.

Because in field they will say there is lot of variability associated with the mix in addition to the binder. So let us continue: but cannot account for mixture variables or provide the relative resistance to aging at in-service conditions. So it will only take into the account of aging in the binder but the influence of mixture and other parameters are not considered here.

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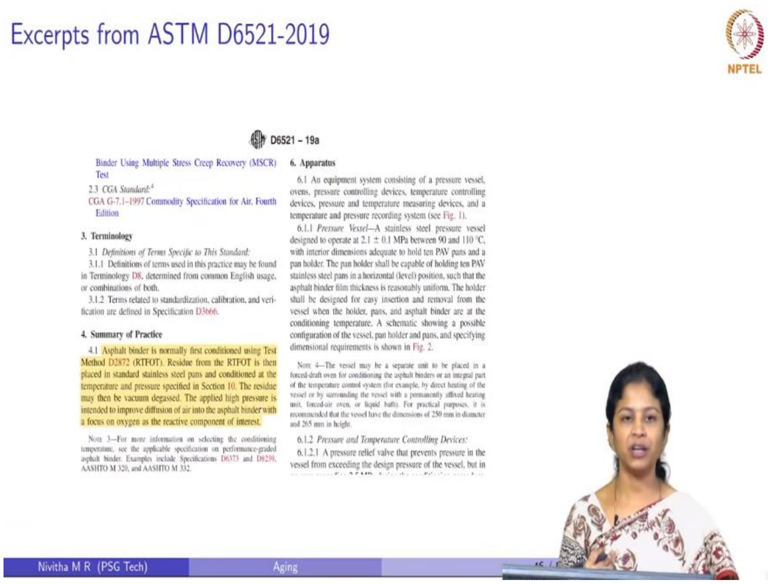

So the summary of this practice is this asphalt binder is normally first conditioned using test method given in ASTM D2872 which is nothing but the short term aging procedure. The residue from RTFOT is then placed in stainless steel pans and conditioned at temperature and pressure specified in section 10. So the temperature depends upon the location where the binder has to be used and the pressure is a constant 2.1 mega Pascals.

The residue may then be vacuumed degassed. So we have to subject the residue to vacuum degassing and the applied high pressure to intended to improve diffusion of air into the asphalt binder with the focus on oxygen as the reactive component of interest. So we want to ensure that most of the molecules in bitumen receive oxygen for the oxidation to occur. So to make that happen we are used a high pressure here.

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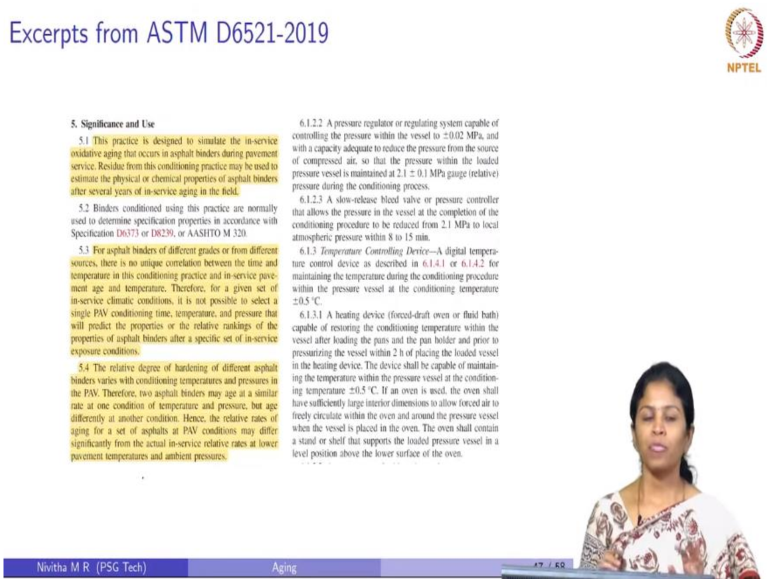

So this is something very interesting: this practice is designed to simulate the in-service oxidative aging that occurs in asphalt binders during pavement service. Residue from this conditioning practice may be used to estimate the physical or chemical properties of asphalt binders after several years of in-service aging in field. So we can measure the physical or chemical properties. So we can measure the increase in stiffness or the functionalities that are formed on aging.

For asphalt binders of different grades or from different sources, there is no unique correlation between the time and temperature in this conditioning practice and in service pavement age and temperature. So what they say here is if I have a temperature at 90 degree Celsius in the laboratory simulation, it will not correlate to a specific temperature range in the pavement right. So there is no unique correlation between time and temperature.

So how much amount of time for which we condition the sample and the temperature will not correlate to the same factors which is observed in field. Therefore for given set of in-service climatic condition it is not possible to select a single PAV conditioning time, temperature and pressure that will predict the properties or the relative ranking of the properties of asphalt binders after a specific set of in-service exposure conditions.

Now if you say me that your pavement temperature is 50 to 55 degree Celsius that is the maximum pavement temperature is 50 to 55 degree Celsius and if the pavement has been there for 5 years, if you want me to give a specific time for which I need to condition it in PAV and a specific temperature, it is not possible to establish this correlation. So that is what they are trying to convey here.

And the relative degree of hardening of different asphalt binders varies with conditioning temperatures and pressures in PAV. So how the material hardens depends upon the temperature and pressure. Therefore two2 asphalt binders may age at a similar rate at one condition of temperature and pressure but age differently at another condition. So if I age binder A and B at 100 degree Celsius they might age in the same manner. So at the end of PAV period I may get the same stiffness or the same amount of oxidation products in both of them.

But if I age them at 120 degree Celsius or 80 degree Celsius I might see a difference in the stiffness values of these two binders or a different value for the oxidation products in both of them. So that is what they are saying: hence the relative rates of aging for a set of asphalt at PAV condition may differ significantly from the actual in-service relative rates at lower pavement temperatures and ambient pressures.

So what we simulate in laboratory is under a specific set of conditions: time, temperature and pressure. But in field depending upon the location all three of them are going to vary. But we cannot establish a correspondence between the laboratory aging and field aging. So under all these circumstances only we are using this particular code to simulate the long term aging performance of the binder.

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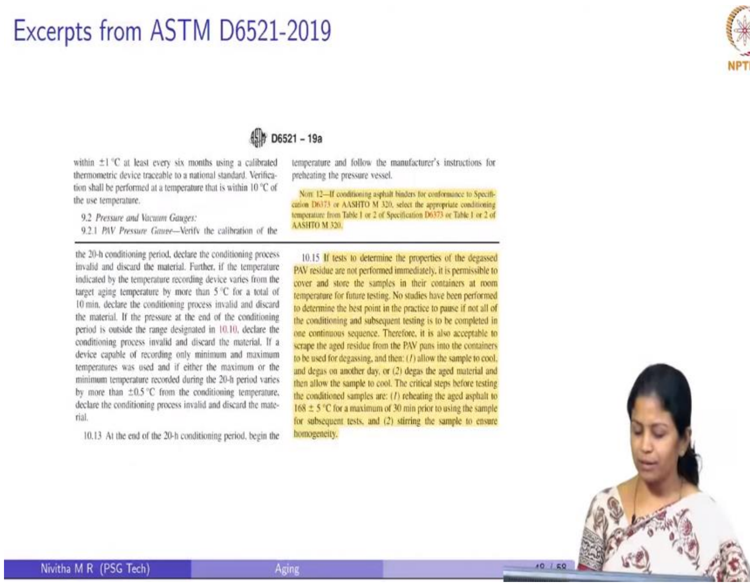

This note says if the conditioning of asphalt binders for conformance to specification D6373 or AASHTO M320, select the appropriate conditioning temperature from table 1 or 2 of specification D6373 or table 1 or 2 of AASHTO M320. So they saying this particular code for PG grading of binders ASTM D6373 or AASHTO M320 tells us what is the PAV aging temperature that should be used for different types of binders.

And this one says what is the condition of the residue. If test to determine the properties of degassed PAV residue are not performed immediately, it is permissible to cover and store the samples in their containers at room temperature for further testing. No studies have been performed to determine the best point in the practice to pause if not all of the conditioning and subsequent testing is to be completed in one continuous sequence.

So in the case of short term aging we said right the entire testing has to be completed within 72 hours, if not we have to discard that prepare a fresh sample. But for the case of PAV aging there is no condition that is specified here. So therefore it is also acceptable to scrape the aged residue from the PAV pans into the containers to be used for degassing and then allow the sample to use and degas on another day or degas the aged material and then allow the sample to use.

So we can perform degassing on some other day or do that immediately also. The critical steps before testing the conditioned samples are reheating the aged asphalt to 168 degree Celsius for a maximum of 30 minutes prior to using the sample for subsequent test. So they say before you test the sample we need to heat it to 163 degree Celsius and mix it for homogeneity in the sample and stirring the sample to ensure homogeneity. So this is the only condition that we have to do before testing the long term aged sample and there is no stipulated time in which this testing has to be completed.

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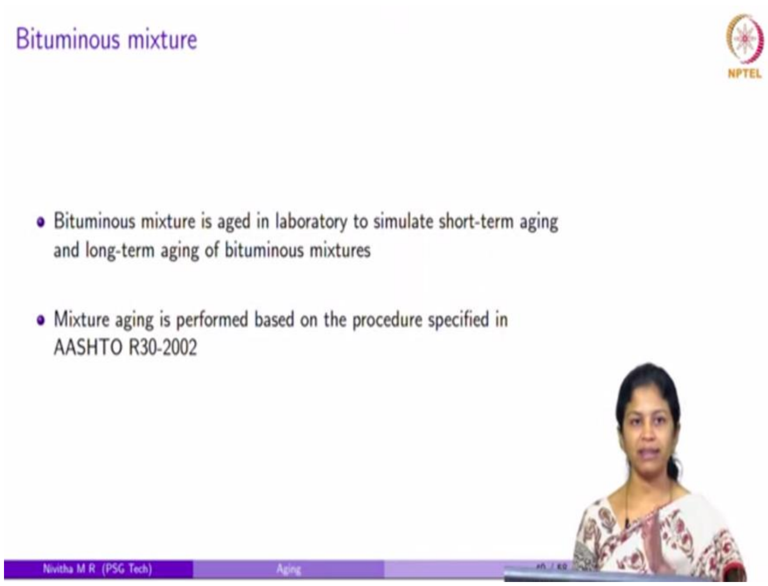

Next we will move on to the aging laboratory aging for bituminous mixtures. So bituminous mixtures is aged in laboratory to simulate again the short term and long term aging. So previously we have seen how binders are aged to simulate these two aging conditions. So here we are going to see how mixtures are aged simulate these aging conditions. So this mixture aging is performed based on the procedure specified in AASHTO**.**

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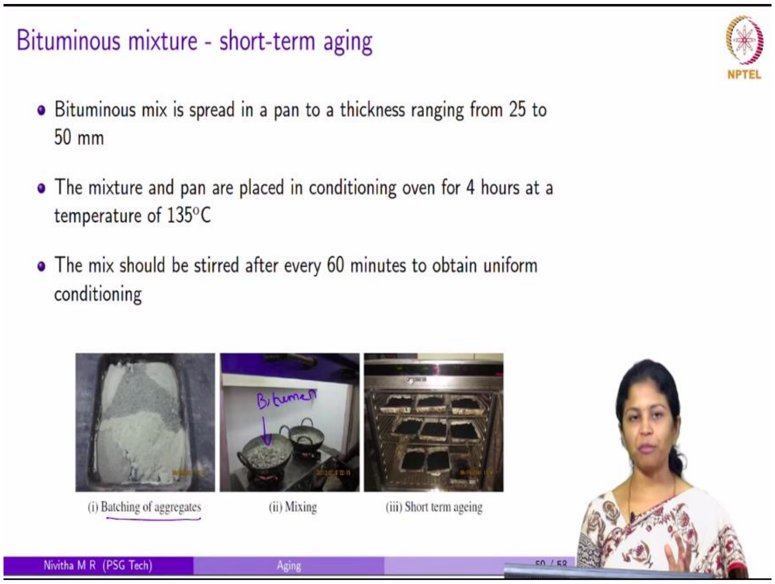

So for short term aging how is this bituminous mixture aged. The bituminous mix right, mix means we have taken a specific gradation of aggregates, we have added the required bitumen content to it and we have mixed them nicely. So after the end of mixing process the bituminous mixed is spread in a pan to a thickness ranging from 25 to 50 mm. So the thickness should not exceed greater than 50 mm.

So we know the reason here right, if the thickness is exceeding 50 mm then the particles lying on the lower surface will not get sufficient access to oxygen. The mixture and pan are placed in conditioning oven for 4 hours at a temperature of 135 degree Celsius. So the mix here should be stirred after every 60 minutes to obtain uniform conditioning. So this image here shows the stages in the short term aging process.

So the first image shows the batching of aggregates; we have taken aggregates of specific gradation and then we have mixed them; we have added the required bitumen content to this and then we have mixed them. After mixing we have spread them in trays like this and then it is placed inside a conditioning oven. So we have now seen the short term aging. This short aging is performed on loose mix. So what happens during the mixing and compaction process is, the mix is, the aggregate and bitumen are mixed and then they are transported. So in that case, the mix is in a loose condition. So this short term aging is performed on loose mix.

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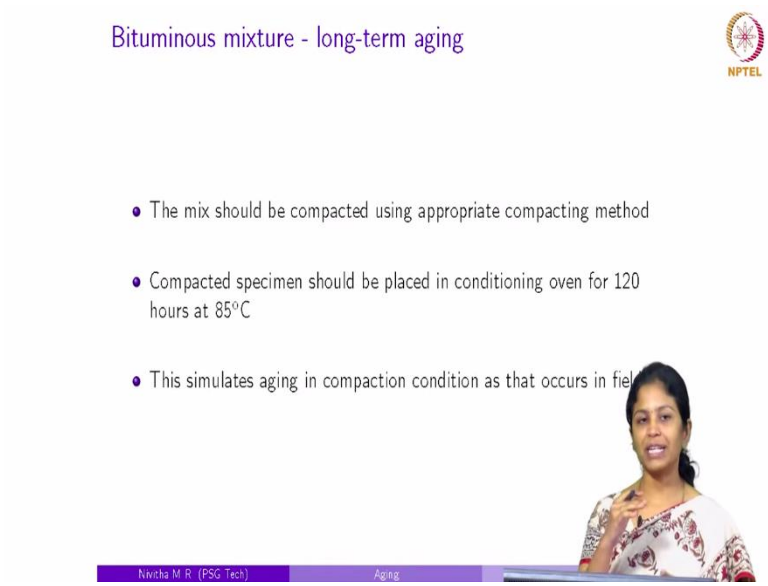

But the long term aging procedure is performed on a compacted specimen. So what happens in field is after laying the mix is compacted and when it is subjected to in-service conditions, it is in a compacted form. To simulate that particular case we have prepared compacted specimen. So the mix can be compacted using any of the standard compacting procedures.

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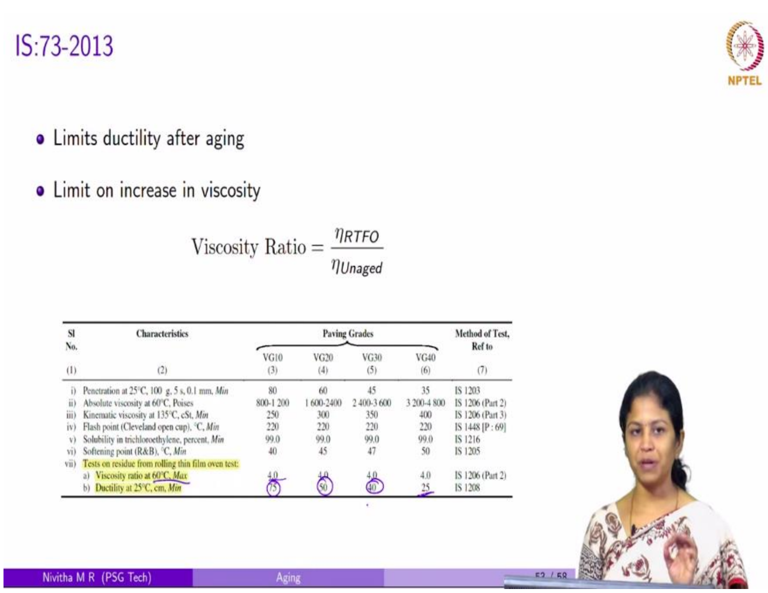

This standard is based on so we can see here the ductility at 25 degree Celsius should have a minimum value of 75 in the case of VG10 bitumen, 50 in the case of VG20 bitumen, 40 in the case of VG 30 and 25 in the case of VG 40 bitumen. As we go to higher grade the requirement the ductility value reduces here. This is the minimum value which is specified here. The other parameter is viscosity ratio at 60 degrees. So we know what this viscosity ratio is which is the viscosity in RTFO case to the viscosity in unaged condition. So this value should be limited to a maximum of 4. So what this means is at the end of short term aging procedure the increase in viscosity of the binder should be limited to a maximum of four times compared to its unaged condition.

And there is no specification in this particular standard related to the long term aging procedure. They only give specifications to test the properties of the binder in unaged and short term aged condition.

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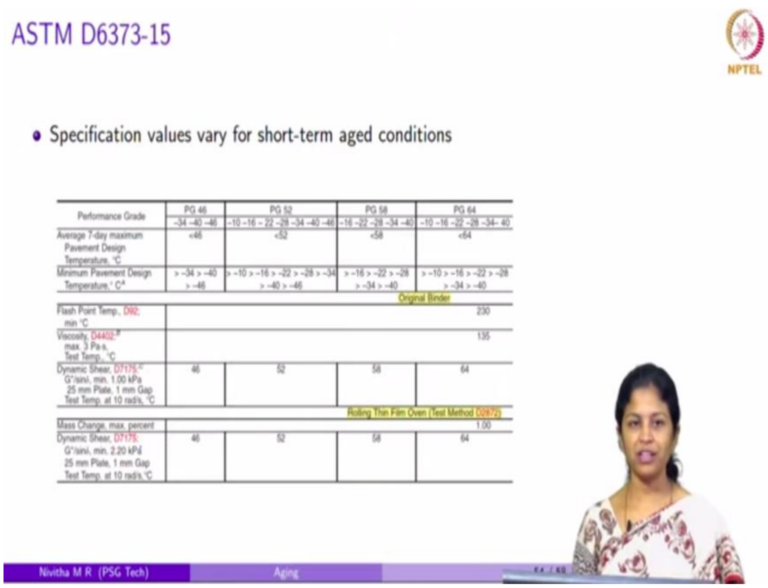

The next one is the ASTM standard which gives specifications for unaged, short term aged and long term aged conditions. So we can see here this table is extracted from ASTM D6373 so there are some tests which are specified on the original binder. So when you were discussing the PG grades for binder, you have seen what does this G*/sinδ and it is specified to have a minimum value of 1 kilo pascal in the unaged condition.

In the short term aged condition which is given as rolling thin film oven residue, this value G*/sinδ has to be a minimum of 2.2 kilo Pascal. So in the previous case in IS code we had a ratio but in this case they have given different specification values in unaged condition and in short term aged condition. So in unaged condition this G*/sinδ has to have a minimum value 1 kilo Pascal whereas in short term aged condition it is sufficient to have a minimum value of 2.2 kilo Pascals.

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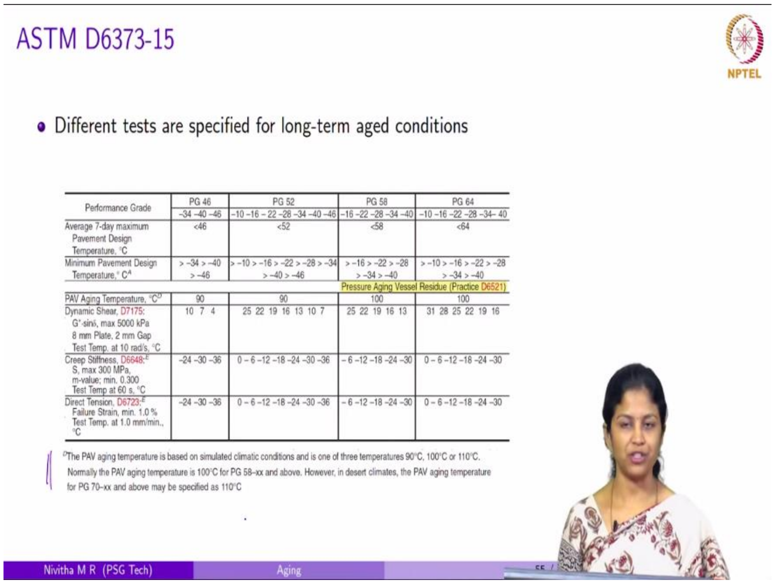

Next we will move on to the long term aged conditions. So this long term aging is simulated using a pressure aging vessel. So in the long term aged condition there are three parameters that are specified here. One is $G^* \times \sin\delta$ which has a maximum value of 5000 kilo Pascals and then creep stiffness and direct tension test is required to be performed on long term aged specimen. So as we know that at long term aging the stiffness increases so at that case we need to check the increase in stiffness of this material.

So we limit the increase in stiffness using these three parameters. Now is this increase in stiffness completely detrimental? No it is not so. It has also a small positive effect in the initial stages. In the initial stages after the pavement is laid we know that rutting is the predominant distress. Rutting happens because the stiffness of the binder and the mix is insufficient to take the wheel load. So there is some stiffness increment that is desired in the initial stages.

So we have seen that there is about 4 fold increase or a maximum of 4 times increase in viscosity after the material has been subjected to short term aging. So then at the end of short term aging the binder has gained some amount of stiffness which will help to reduce rutting initially after the mix is laid. After that it kinds of hardens, there is densification of the mix, lot of other things which you will be seeing when you discuss the mix portion. But the aging of the binder is also one of the factors which is contributing to this increase in stiffness.

So in the initial stages we want aging so that we will minimize rutting but in the later stages we do not want aging to increase the stiffness beyond a particular value such that the fatigue cracking and low temperature cracking will become critical. And here in this case you can see the note related to PAV aging. So they have said that the PAV aging temperature is based on simulated climatic condition and is one of the three temperatures 90, 100 or 110 degree Celsius.

So normally the PAV aging temperature is 100 degree Celsius for PG 58 and above. However in the desert climates PAV aging temperature for PG 70 and above may be specified as 110 degree Celsius. This is the note related to the test temperature from this particular specification.

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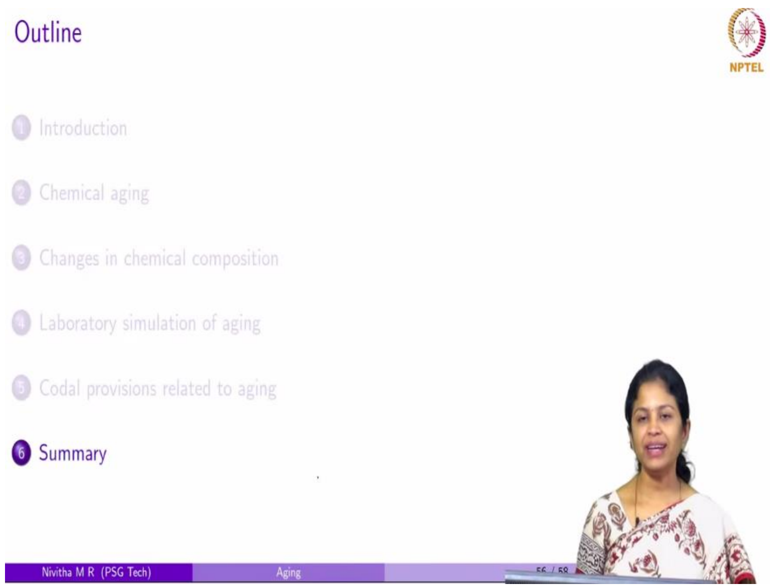

Finally we will summarize our understanding related to the aging of bituminous binders and mixtures.

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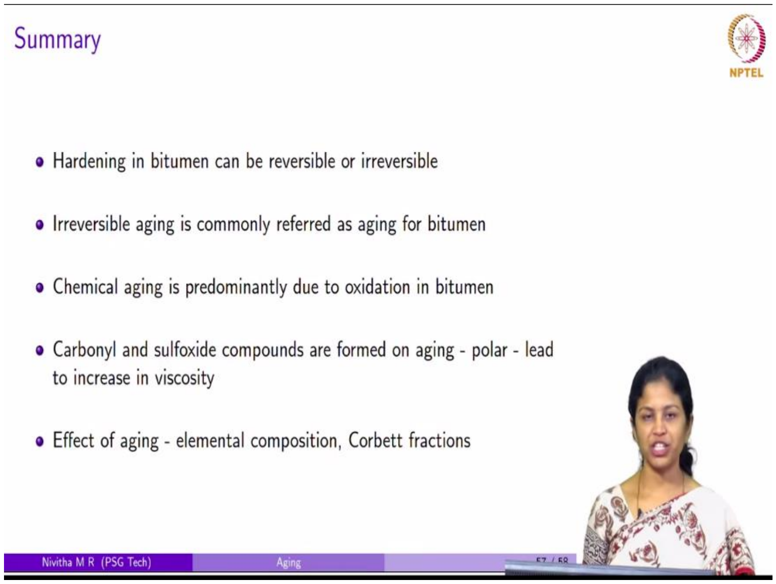

So the first thing is we talked about hardening and bitumen. There is some increase in hardness of bitumen. So this increase in hardness can be reversible or irreversible. The reversible hardening again occurs under two conditions: one is low temperature physical hardening which occurs when material is stored at isothermal temperatures below its glass transition temperatures. The second one is steric hardening which occurs when bituminous is stored at room temperatures for a long period of time. Both of these effects in bitumen can be reversed.

The low temperature physical hardening can be reversed by heating bitumen to a temperature which is above its glass transition temperature. The effect of steric hardening can also be removed by heating it to temperatures greater than 70 degree Celsius. So this is with regarding to the reversible hardening. There is also an irreversible hardening which is called as chemical aging or simply aging in bitumen.

So when we refer to aging we by default mention the irreversible hardening that happens in bitumen. So this irreversible hardening results as a consequence of the oxidation which happens in bitumen. There are a lot of other factors but oxidation is the predominant reason for chemical aging that happens in bitumen. And as a result of this oxidation, there is formation of carbonyl and sulfoxide compounds in bitumen.

So these compounds are polar in nature. So because of their polarity they try to form strong association which leads to an increase in viscosity of the system. And the effect of aging we have seen on the elemental composition and on the Corbett fractions. So we saw that there is no variation in the carbon, hydrogen, nitrogen component of bitumen. But only in the oxygen fraction we saw an increase in the proportion.

And on the carbon fractions we saw that there is no variation in the saturates fraction of the bitumen but on the naphthene aromatics, polar aromatics and asphaltene fraction we saw a variation. There is a conversion of naphthene aromatics to polar aromatics and polar aromatics to asphaltenes. So there is a net increase of asphaltene fraction in bitumen as a result of aging.

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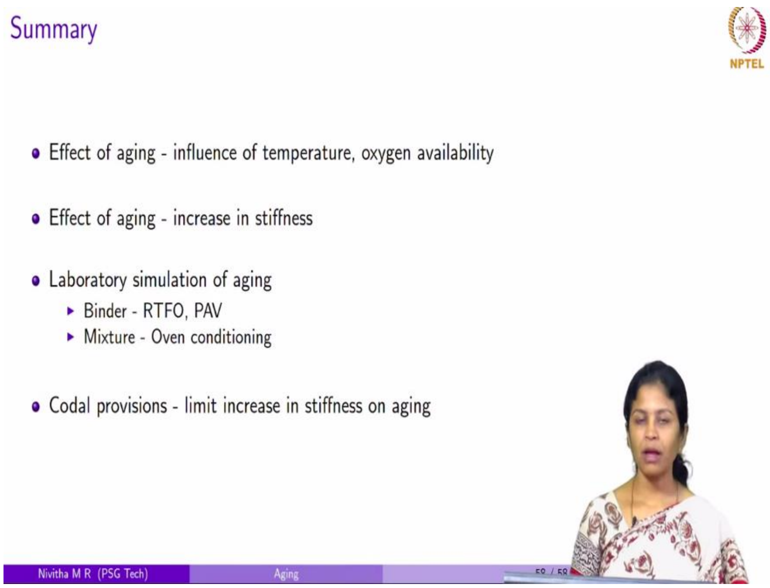

We also saw what is the effect of temperature and availability of oxygen on aging. So at low temperature the material is in a gel like form so the molecules are associated with each other and there is less availability of oxygen to the molecules in inner core for oxidation. Whereas at high temperatures it is in a sol state so there is more availability of oxygen to many of the molecules which is present in bitumen.

And so the oxidation at low temperature proceed at a lower rate. Whereas the oxidation at high temperatures proceed at a relatively faster rate and the effect of aging is an increase in stiffness. So we saw that during short term aging most of the increase in stiffness because of the binder occurs and during long term aging there is gradual increase in stiffness of this material. And we also saw two procedures to simulate the short term aging and the long term aging in the binder.

So the short term aging is simulated using a rolling thin film oven and the long term aging is simulated using a pressure aging vessel. We also saw the respective ASTM standards and the concerns which were addressed in these standards especially related to the long term aging. We also saw the aging procedure for bituminous mixtures. For short term aging we aged a loose mixture whereas for long term aging we prepared a compacted specimen and then subjected it to aging.

And finally the codal provisions related to the aging was also discussed. We saw IS73 which provided limits on ductility and viscosity ratio and in ASTM D6373 which is the performance grade for binders, we saw different specification parameters for the binders in unaged, short term aged and long term aged conditions. So with this we will wind up the topic on aging in bituminous binders and mixtures. So I thank you.