Mechanical Characterization of Bituminous Materials J Murali Krishnan Professor Department of Civil engineering Indian Institute of Technology Madras

Lecture No - 27 Viscosity Grading – Part 2

(Refer Slide Time: 00:13)



So now let us take a look at IS73 as it exists and we grow from them. But before that we will also have a general discussion on some of the international specifications that are available okay.

(Refer Slide Time: 00:28)



So first and foremost the thing that I want to show here is the Australian specification. Some of the climatic zones in Australia some of them not all of them are more or less identical to what you see here. And please focus your attention on only these two parameters here. So there is something called class 170, there is something called class 320. And this is written in Pascal second.

So for your convenience if you want to write it in poise, so this is going to be 2600 to 3800 poise for class 320, so this is the viscosity at 60 degree centigrade and the pen at 25 degree centigrade is taken as a minimum of 40 okay. And this is a classical viscosity based specification. Why I am saying that this is a viscosity based specification? Because you can actually see that the viscosity values are bound.

So there is a minimum value there is a maximum value whereas the penetration value is left with the minimum. If it goes above it well and good but at least it should be minimum. In fact it will be very nice if you could have a penetration of 50 for such kind of material. Because 2600 to 3800 is very good bitumen at 60 degree centigrade. And if I could have a penetration of a 50 for this material at a 25 degree centigrade.

In a sence I will also be able to handle the fatigue damage that can happen. Please do not relate try and relate the penetration with the fatigue. What I really wanted to say is a low penetration means a stiff material and a stiff material is likely to have more tendencies for fatigue damage at that temperature okay. So one should not generalize here.

(Refer Slide Time: 02:21)



This is the specification based in USA, ASTM D3381. Now ASTM gives this in two tables there is table one there is a table two here. Please take a moment and look at these two tables and then we will focus our attention on AC30, and as usual on 300 ± 60 which is the viscosity at 60 degree centigrade and penetration at 25 degree centigrade, which is 30. And similarly look at another AC30 which is 300 ± 60 and penetration of 50.

So we will call this as table 2 and we will call this as table 1. In fact if you are a highway engineer and if you are asked to choose bitumen from table 1 or table 2 what is the choice that you will make? So if you actually look at it both the viscosity values are the same but only the penetration values are different. And the table two material has more penetration compared to the table one material and based on the discussion that we had earlier you will always go for table 2 material, because this is a much superior material compared to Table 1.

And in fact in, US if viscosity grading is used in specific places the default table to be used in that tender document is table 2 and not table 1. So table 2 always gives you a superior material if you are going to use penetration and viscosity as the indicator for the expected quality of the material.



European international scenario as far as European Union is concerned, is completely complicated and when European Union was formed there was a harmonization of the specifications that also happened. And because of this there are two sets of specifications that available one set of specifications are compulsory another set of specifications of voluntary. So you could have a penetration based specification.

You can have a viscosity based specification. And depending on the different types of grades that are available each and every country picks the compulsory tests, as well as the voluntary tests to be used. So we would not spend that much time, but I just wanted to indicate that such kind of specifications are in place okay.

(Refer Slide Time: 05:14)

International Scenario – South Africa

Property		Test Method			
	40/50	60/70	80/100	150/200	
	14.00	Requ	uirements		
enetration at 25 °C, 0.1 mm	40-50	60-70	80-100	150-200	ASTM D5-IP49
oftening point, °C	(49-59)	46-56	42-51	36-43	ASTM D36*
scosity at 60 °C, Pa.s	(220-400	120-250	75-150	30-60	ASTM D4402*
scosity at 135 °C, Pa.s	0.27-0.65	0.22-0.45	0.15-0.40	0.12-0.30	ASTM D4402*
eformance when subjected to <u>RTFOT</u> : mass change, % by fraction) max viscosity at 60 °C, % of original, max softening point, ing & ball), °C min increase in softening point, °C max retained pen, % of original, min	0.5 300 52 9 60	0.5 300 48 9 55	0.5 300 44 9 50	0.5 300 37 11 50	ASTM D2872 ASTM D2872 ASTM D4402* ASTM D36* ASTM D36* ASTM D54P49

Now, in my opinion, this is by far, one of the most rigorous specification, one can actually have and this is the South African specification. Now let us take a look at this penetration grade though they call it actually based on the penetration grade everything here is bounded, if you can from the way in which you can see now please take a close look at the penetration at 25 degree centigrade it is 40 to 50, we are familiar with this.

And then look at viscosity at 60, so this is 220 to 400 pascal second or if you use IS73 it is 2200 to 4000 poise what is very interesting here is, not only the viscosity is bounded the penetration is also bound and the softening point is also bound. In addition the viscosity at 135 degree centigrade is also bound. So that means there is a strict quality control that is necessary to produce bitumen to meet the required specifications, if you want to produce it in South Africa.

So that means all the four parameters in unaged conditions have to be bound. So it cannot exceed and goat now the another part is when you subject this to short-term aging. You have already we have already discussed about short-term aging. The mass change values are given viscosity values at 60 degree centigrade is also given. The softening point values are given. The increase in softening point as well as the retain depend is given here. So this is for more stricter and completely bound specification.

(Refer Slide Time: 07:02)

How should technical specification for a product be formulated?

- What is the probability of a random sample to be out of lower/upper limit ?
- What is the probability of a random sample to be not only out of its own limit but trespassing adjacent limits?
- · Are all the properties normally distributed?
- What is the acceptable standard deviation?

NPTEL-MCBM-Viscosity



Now why this is being discussed in detail is for the simple reason that in any refinery production process one needs to clearly understand the variability associated with the refinery production. So if you recall the discussion that we had about refinery production in the earlier lecture, we talked about two types of production process. The first one is the biturox kind of a process or air blowing process. The second one needs about the PDA based process or component blending process.

In one case we reach the viscosity by blowing hot air through it. So we go from let us say 200 poise to 2,000 poise. In another case we have a pitch which has probably a viscosity of 20000 poise and we take a 200 poise vacuum residue to 20000 poise. And then from there we come back to 2,000 poise by addition of heavy extract, HVGO or furfural extract to the material. So these two production process are completely different.

Now in any production process in any industry when, if the design is made to meet one specific parameter it is always likely that the other parameters. For instance, let us take this case here the penetration value may really be go out of range. So that is why when any specification is design the variability associated with the production process is taken into account and we always write a bound for it.

(Refer Slide Time: 08:46)

International Scenario – South Africa



So that means in if you are going to produce bitumen as far as using the South African specification is concerned a South African refinery will be shooting for close to around 3,000 poise for the viscosity and 45 penetration as well as around close to 50 for softening point the idea is you can always shoot for the median value and there will always be the variability in the production process that will in essence take into account the limits and that is how these things are written here.

(Refer Slide Time: 09:18)

How should technical specification for a product be formulated?

- What is the probability of a random sample to be out of lower/upper limit.?
- What is the probability of a random sample to be not only out of its own limit but trespassing adjacent limits?
- Are all the properties normally distributed?
- What is the acceptable standard deviation?

NPTEL-MCBM-Viscosity



it go above the upper limit. What is the probability? The next question that we need to answer is what is the probability of a random sample to be not only out of its own limit trespasses into the other adjacent limit?

So this is what in fact it is all about, so now let us take a look at the American specification here. (**Refer Slide Time: 10:11**)



So, this viscosity is, for instance, this is 200 ± 40 so that comes to 160 to 240 here and this is 300 \pm 60, so this becomes 240 to 360 and here, it is 400 \pm 80 so that goes from 320 to 480. Now there is a possibility that this 320 that is seen here could be also part of this AC30 specification. But then you will also understand immediately that you not only have to meet this specification. You also need to meet this specification plus the penetration value as well as many other parameters here.

(Refer Slide Time: 10:56)

How should technical specification for a product be formulated?

- What is the probability of a random sample to be out of lower/upper limit.?
- What is the probability of a random sample to be not only out of its own limit but trespassing adjacent limits?
- Are all the properties formally distributed?
- What is the acceptable standard deviation?

26

NPTEL-MCBM-Viscosity



So if you are going to be focusing your attention only on viscosity you might think that there is a possible overlap but, in that is not necessarily the correct way of looking at it. Because any product has to meet all the required specifications. So that means one should write the specifications in such a way that, even if one of the values parameters trespasses on goes to the other limit it will automatically not trespass and go into other limits for some other specification parameters.

So that is why what we normally do is we keep the penetration values minimum and we give a range for viscosity, because since it is a minimum value the possibility of the trespassing into the different limits are always not met. And we also need to understand whether this is normally distributed, because this is the basic assumption with which we actually work here and how much is the acceptable standard deviation.

These are some of the questions one should really understand raise discuss before we start writing any specification. This particular slide is given more in terms of making you understand the intricacies related to production process which we have already discussed and the specifications that we are going to discuss as far as bitumen is concerned okay.

(Refer Slide Time: 12:22)



Right, to give a simple example, what you see here is the statistical variations that you are going to see as far as the South African specification is concerned. So you can actually see in x-axis the viscosity at 60 degrees centigrade and they are cleanly spaced out for the different pen values so there is absolutely no trespassing that you are going to see here. And such kind of clear divisions will come only when you are going to provide bounds for each and every parameter but such kind of products are extremely difficult to produce and manufacture.

(Refer Slide Time: 13:01)



So I am going to use this opportunity also to explain to you how IIT Madras wrote the IS73. This was based on a generous funding that was provided by Bureau of Indian Standards, so what we did was.

(Refer Slide Time: 13:21)



We collected samples 300 samples across the country. So these were from construction-site samples refinery samples, samples that were used before modification as well as some of the imported samples that were used here.

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And we ran around 6000 tests on these samples, so that means all the IS73 tests and we also ran tests at 60 and 135 degree centigrade using rotational viscometer in unaged and short-term aged

conditions. Because the original mandate of Bureau of Indian Standards was to try and see whether we could introduce viscosity specification using rotational viscometer, because the actual measurement of viscosity that is currently stipulated is based on vacuum capillary viscometer.

Kinematic viscosity at 135 degree centigrade we also carried out the test at short-term aged conditions and the penetration again we looked at it at the short-term aged conditions. Softening point in addition to it, we did the PG testing for 100 samples and this was carried out not only at IIT Madras to check the reproducibility of the results this additional testing was carried out at Chennai Petroleum Corporation Limited.

In fact we are very very grateful to them for the cooperation extended during that research project and to find out what is the rheological behavior of the material, we carried out frequency sweep for 100 samples in unaged and short-term and conditions. By this time you should be familiar with what is a frequency sweep experiment? What is a master curve? and what is a thermo rheologically simple behavior?

So if you are able to have two materials whose master curve lie on top of each other you can safely say that the temperature susceptibility of the material are more or less identical okay, so that is the take home message here.

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Aging	Grade	Source	Statistic	Mean	P- Value	Std.	Significance level 0.02	Distribution
condition						Deviation	Critical value	\bigcap
	VG10	Refinery	0.1077	83.607	0.34879	14.578	0.124	Normal
		Site	0.1508	87.6	0.9888	1.759	0.3815	Normal
Danad	VG30	Refinery	0.1162	62.95	0.2502	11.47	0.1225	Normal
Chageo		Site	0.1983	61.81	0.1646	9.202	0.2702	Normal
	60/70	Site	0.1983	61.81	0.1646	9.202	0.2702	Normal
	80/100	Site	0.2332	94.70	0.6951	12.35	0.50654	Normal
	VG10	Refinery	0.104	50.31	0.3906	12.83	0.1241	Normal
		Site	0.2586	44.02	0.6482	8.779	0.3815	Normal
Short term	120000	Refinery	0.1373	38.62	0.1252	9.326	0.1774	Normal
aged	1 650	Site	0.1021	38.81	0.5684	6.934	0.1404	Normal
	60/70	Site	0.3667	44.2	0.2376	8.379	0.3815	Normal
	80/100	Site	0.15647	53.71	0.97231	11.90	0.50654	Normal

So we did lot of statistical analysis as we mentioned we wanted to check whether it is normally distributed

(Refer Slide Time: 15:41)



Similarly viscosity at 60 degree centigrade whether it is normally distributed (**Refer Slide Time: 15:47**)



And based on all these things and doing lot of structural simulations which we may not necessarily be able to discuss at this point of time. Because there is a considerable pavement engineering background that is needed payment design background is needed. Originally we came out with two sets of classification. The first classification is based on high volume traffic and low volume traffic.

And in fact we kept the penetration values different for high and low volume traffic but we kept the viscosity constant irrespective of it. Why did we do that? If you recollect the discussion that we had about fatigue it is not only because of having a stiff binder it is also related to traffic. So when we are talking about traffic we are talking in terms of axle load as well as the number of repetitions.

So we wanted to keep for low volume traffic a slightly lower pen and for high volume traffic we wanted to keep a slightly higher pen. Again please do not try to relate if you have a high penetration it is a better material for fatigue response. No that is not the idea, the idea is a higher penetration material is soft at 25 degree centigrade. And the tendency to crack is likely to be less. So this is basically you consistency based parameter.

And we came out with divisions more or less identical to ASTM D3381 the rest of the parameters are more or less standard as far as high what we meant as high is more than 50

million standard axles in 15 years. Ok that is what we meant as high and low is less than 50 million standard axles. But when this specification was submitted and basically due to operational reasons it was felt the refineries may not really be able to have eight grades of bitumen available. Because for each and every grade that is a high-volume traffic material that is also a low volume traffic material.

(Refer Slide Time: 18:15)



So finally, we came out with a different strategy but before that we need to also understand how to use this specification? So the first and foremost thing that we need to understand is when you want to choose any binder for any specific location, the first thing that you need to know is what is the air temperature? So let us say you are constructing your road in 2020 you want to really find out and let us say you are designing it for ten years you need to find out what is the expected air temperature distribution from let us say 2020-2030.

Now this is a big thing, fortunately there are algorithms that are available that can do the job. Once you know the air temperature, what you now need to do is to find out what is the pavement temperature. So we need to have air temperature to pavement temperature. So if you are take let us say 365 days, 24 hours. At least if you are looking for one year you are going to have this many number of hours of pavement temperature distribution.

And from this we now want to pick what is the maximum and the intermediate. So why maximum? Maximum is related to rutting, intermediate is related to fatigue. Okay. This is what we will normally call as uncorrected temperature. But what can really happen is due to the speed, okay. The actual intensity of the distress that you are going to see here can be considerably more. To give an example, let us assume that there are two locations Chennai, 60 degree centigrade, and maximum pavement temperature during summer. Another location let us say Jaipur maximum pavement temperature is 70 degree centigrade. A truck going at 40 kilometer per hour in both these locations having the same weight will cause more damage in Jaipur compared to what you are going at Chennai. So if you really want to do that then what we really need to do is we also need to make some small corrections as far as the temperature traffic is concerned we will discuss this in a little more clearer way in the PG testing that we will do PG.

Then what we did next was we prepared a contour map because we are not really expecting that an Indian highway engineer will go collect the air temperature, data then compute what is the pavement temperature and then correct for this traffic. So we prepared the contour map and from this the binder selection will be done. We also need to do analysis of the binder from the different refineries.

The situation in India is slightly different. We choose a binder based on the geographical proximity of the refinery nearby and we need not we do not necessarily choose a binder based on which is the refinery that makes the best bitumen or the one that makes it through a specific process so we have not actually reached that kind of a stage okay.

(Refer Slide Time: 22:06)



So this is based on the work that was carried out by Dr. Nivitha, as part of her M Tech dissertation and in fact she has actually given a series of lectures on chemical composition aging and polymer modification. So what you see here is the maximum pavement a temperature contour. So we were able to see that there are at least five different climatic zones that exist in our country and you can actually see each of them here. And the maximum pavement a temperature a temperature of course this is uncorrected for traffic, it varies from 52 to 68 degree centigrade.

(Refer Slide Time: 22:47)



And when we look into the minimum pavement temperature it goes all the way from -10 to 22 degree centigrade. So and again there are at least six temperature zones. So if you take the case of Chennai to give an example you see that it is 60 degree centigrade. So the maximum

pavement temperature in Chennai can be 60 degree centigrade and the minimum pavement temperature is 16 degree centigrade, this is +16 degree centigrade.

So if you are designing a pavement at Chennai you should be ensure that you will pick a grade of bitumen that will give you the rocket rut resistance at 60 degree centigrade, as well as the fatigue resistance at + 16 degree centigrade. In PG we even go to the lowest temperature is taken but this is on the lowest temperature that we are going to see in this country. You are going to see substantial low temperature near Jammu and Kashmir where as it is it can go up to -10 degree centigrade and, again this is unconnected for traffic.

(Refer Slide Time: 23:59)



So what was the original way in which we wanted to do so you select a location, you do the find out the traffic volume so when we are talking about traffic volume we are talking in terms of the axle load as well as the 24 by 7 count. Then if the traffic is less than thousand five hundred commercial vehicles per day, say that you pick a low traffic material. But if it is more than that we wanted you to use a high traffic material.

And in fact this is basically based on extensive calculations that were carried out and in fact we have published all these things as part of the research report to Bureau of Indian Standards. (Refer Slide Time: 24:45)



So how do you choose the temperature? You first use the contour map given in the report are placed here for the selected reliability, reliability plays a critical role so that means, what is the reliability that you want 90 percent or 95 percent?, after correcting for traffic as well as speed and peak pavement temperature. So if it is less than or equal to 65 degree centigrade use a VG-10 material, 65 to 70 VG 20, VG 30 is 70 to 75 degree centigrade.

This is after correction for temperature and traffic. One important point that I need to mention here so this is something to do with VG 40, so when we wrote our specifications the number of VG 40 samples that were available as part of the refinery production was very minimal nevertheless we included them based on the international data that was available. And later we have been also working with the few oil companies and we were able to find that those which is 40 parameters that we have written are indeed achievable within the country.

There are lot of issues related to this particular grade of bitumen and that we will not discuss as far as this course is concerned if you want anything you can always get in touch with one of us. (Refer Slide Time: 26:18)

	This is our ne	w IS7	73-20	013!	!!		
				2	018	4	
s	Characteristics		Paving	Grades		-	
No.	(2)	VG10 (3)	VG20 (4)	VG30	VG40	.)	
0	Penetration at 25°C, 100 g, 5 s, 0.1 mm, Min	80	60	151	35	-	
ii)	Absolute viscosity at 60°C, Poises	800-1 200	1 600-2400	2 400-3 600	3 200 4 8	00 2	
iii)	Kinematic viscosity at 135°C, cSt, Min	250	300	350	400	3	
iv) 1	Flash point (Cleveland open cup), °C, Min	220	220	220	220		1100
V) :	Solubility in trichloroethylene, percent, Min	99.0	99.0	99.0	99.0	100	10 A 10
VI) :	Softening point (R&B), °C, Min	40	45	47	50	4	Contraction of the local division of the loc
vii)	Vieweite educate from rolling thin film oven test:	4.0	10	10	10	r .	CI manual
1	 b) Ductility at 25°C cm. Min 	75	50	40	25	2	Contraction of the

So right now this is the IS73-2013 and this has been revised when reissued in 2018 and you can actually see that the penetration is kept a minimum value of 45. There is the viscosity is given as 2400 to 3600 and rest of the values. So as you can see there is a no overlap here, there is no overlap here. But there seems to be some overlap here, but you should also see that the penetration minimum values are completely different.

So, and in the same way the kinematic viscosities are also completely different. So if you are going to measure in terms of look in terms of only viscosity you might think that there is a overlap here. But actually there is no overlap, because you have to meet all this specification parameters here okay. And in fact I will come straightaway here to this and these parameters have to be met and only then you can classify a binder as based on VG40 specification right? (Refer Slide Time: 27:32)

IS73 – What you should read carefully!



So this is something that you should read very carefully, so what it means is we have given it in terms of maximum air temperature suitable for seven-day average maximum air temperature. So you will see that this is the seven-day average maximum air temperature for a period not less than five years from the start of the design period. So at least this much of design data that you should be able to handle for you to make a statement right.

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You also need to understand that whatever is specified here, is based on refining of crude petroleum by suitable method. So that means everything should be a refinery product. This specification prescribes only bitumen produced in a refinery, whatever is the parameters that are mentioned here need not necessarily work for any material that is produced outside the refinery.

(Refer Slide Time: 28:37)



So what are the important things that you should know one should know how to measure the viscosity at 60 degree centigrade this is a non-trivial task, it is not easy so what you really need to do first and foremost is to read the appropriate standard very carefully. Measurement of viscosity as far as bitumen is concerned at 60 degree centigrade is very tricky the bitumen should be heated to an appropriate pouring temperature conditioned at a specific temperature in the vacuum capillary viscometer for a specific time period.

And then used here in an oil bath at 60-degree centigrade again for a specific time period. As part of this course we will be sharing you some of the ASTM standards related to measurement of viscosity and you should be able to precisely read and find out what is that temperature and the time duration here? What is the temperature and time duration? The most important part here is the digital vacuum regulator.

ASTM prescribes 300 mm of Hg vacuum to be maintained. And there is also a deviation that is prescribed here you should be able to find out how much the deviation that is allowable is. So that means when the vacuum is connected and when it is switched on, it should not vary between a specific ranges. If it varies the measurement of viscosity here will be erroneous and you will have problems okay.

(Refer Slide Time: 30:19)

Read ASTM D2170 very carefully!!!



NPTEL-MCBM-Viscosity

Standard Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer¹

Do you have all the accessories for performing this test?

So this is the standard that you should be using and you should have all the accessories for performing this test. And for a period of time in the last 15 years since we have been testing bitumen at IIT Madras, we have found out that this is probably one of the trickiest tests as far as measurement of viscosity is concerned. If you deviate from any one procedure any one step you are likely to get erroneous results. So you have to be very very careful right?

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35:12/35: et us summarise! Rutting, Faligue. (Indian) Susceptility 4) Indian 5) 6) 1) roull -NORMAL IITM -6) TS 73-10) NPTEL-MCBM-Visi

So let us summarize what we discussed here. So the first we talked about the pavement design cross-section okay. We mentioned that the pavement has two bituminous layers that is going to be distressed due to rutting, there is also distress due to fatigue. And then we mentioned that rutting happens at high temperature and fatigue happens at, what I should call it as intermediate

temperature not necessarily low temperature, if I follow the world terminology but what I would use within the Indian context the low temperature.

Because in most of the locations in this country even at the lowest temperature we are likely to have only fatigue damage and not the low temperature cracking right? Then we also understood what is really called as temperature susceptibility? That means how rapid is the variation in the consistency parameter as a function of temperature so if you are going to have bitumen that is soft at 60 degree centigrade, you are going to have problem due to rutting.

So if you want to going to make it hard then at 25 degree centigrade it is going to become even more harder getting into this issues related to fatigue. If you want to use a bitumen so that there will not be any fatigue damage and if you use a soft binder 25 degree centigrade, at 60 degree centigrade you are going to have issues related to rutting.

So you really want your bitumen whose temperature susceptibility is minimal in the sense that the variability associated with temperature of any rheological parameter will not be drastic, so we need to understand that. Then we looked at we identified viscosity at 60, pen at 25 degree centigrade in the Indian context and we were looking for general specifications. We looked at international specifications.

And we discussed in detail the USA specification for the simple reason that it had this temperature susceptibility clearly taken into account in the table 2. So when we are going to balance bitumen may produce bitumen with more than 6 or 7 parameters, the production process plays a critical role, so we also need to understand the variability associated with production process.

So you want to know whether the distribution is normal. Then we discussed in detail how IS73 was written in IIT Madras with funding from Bureau of Indian Standards. Then we showed you the temperature contours and also the traffic details. And then finally a roadmap on how to choose bitumen for a given location was shown here, okay. So this is the crux of this particular lecture. Thank you.