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Recent trends in bitumen characterization in India

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ABSTRACT

This paper reviews the history of bitumen testing and recent trends in bitumen characterization in India. India currently follows a viscosity grading system as given in IS73, 2012 for paving bitumen. Viscosity grading for paving bitumen is based on single point measurements such as viscosity at 60 and 135 ºC and penetration value at 25 ºC. Due to the complex nature of bitumen, material processed from different refineries while meeting the IS code requirements, can have different temperature, shear susceptibility and durability characteristics. Manifestation of the rheological properties of bituminous binders over a wide a range of temperature in real life pavements can be attributed to various factors viz. the crude source and processing methods, the effect of aging and chemical composition of bitumen. There is an urgent need to shift towards a performance based specification for binders from the existing viscosity grading specification for bitumen in India. A clear understanding of the fundamental rheological properties and their inter-relationship with the chemical composition is important to address the issues related to binder characterization. This would lead to development of a rational binder specification and this paper discusses the relevant issues in detail.

KEY WORDS: Bitumen, Rheology, Chemistry, Viscosity, Aging, Viscoelasticty

INTRODUCTION

India is currently constructing the largest ever bituminous based national highway project. The entire cost of this construction is expected to be more than Rupees 480,000 Crore and is likely to be completed in the next 10 years (Ministry of External Affairs, ITP Division, Government of India, 2013). While the national highways in India constitute less than 2% of the total road network of 42.4 lakhs kilometers, it carries more than 40% of the total road traffic. Also about 65% of freight and 80 % of passenger traffic are carried through roads in India (NHAI, 2013). Rational bituminous binder and mixture specifications and quality control measures are needed today so that the maintenance cost of these superior quality roads can be minimized in the future. The quality of bitumen used as a binder material has a major impact on the performance and durability of these roads which are vital for Indian economy.

Bitumen is a byproduct produced from the vacuum residue during processing of petroleum crude in a refinery. Depending on the crude source and the processing method, bitumen undergoes considerable changes in its physical properties and chemical composition (Rajan, 2010). The influence of temperature on the behavior of bitumen is one of the most important aspects to be understood by a highway engineer. While most construction materials do not exhibit such drastic changes in mechanical response during service, bitumen displays unusual sensitivity to changes in temperature. The influence of temperature on the behavior of bitumen is evident from the time of pavement construction to its final design life. When bituminous mixture is laid and compacted, the binder is subjected to temperatures around 150 °C. During service, the temperature of the bituminous layers can vary from a very high temperature of +70 °C to a very low temperature of – 10 °C in India. These high variations in temperature in the pavement structure attributes to most of the stresses and subsequent failures of pavements. For instance, rutting in pavement occurs at high temperatures and temperature induced cracking occurs at low temperatures. Interestingly, the demands of a pavement engineer with respect to binder are quite conflicting. For instance, at high temperatures rutting must be avoided and the binder is expected to be 'stiff', but being 'too stiff' at low temperatures result in cracking. In order to understand how such requirements can be met, one has to trace the path of the bitumen from the time it is produced in the refinery and used as a binder material in pavement till its useful life time (Padmarekha and Murali Krishnan, 2011).

This paper outlines the complexities in understanding the rheological behavior of bitumen. With a brief outline about history of bitumen testing, this paper further discusses on the recent improvements on bitumen characterization in India, and the urgent requirement for a performance based binder specification and the tasks involved in it. Significant understanding on the influence of crude source, various processing methods, aging characteristics and chemical composition of bitumen on its rheological behavior is vital to establish a reasonable connectivity between the specification test parameters and field performance.

History of bitumen testing and Indian specifications: Originally, the quality of bitumen was determined by chewing (Halstead and Wellborn, 1974). A sample of bituminous binder was literally chewed to subjectively determine its degree of softness. This method is no longer in use today for obvious reasons. Apart from chewing, the penetration test is the oldest specification test for bitumen used throughout the world. The penetration machine was invented by Bowen of the Barber Asphalt paving company in 1888 (Halstead and Welborn, 1974). The basic principle of the penetration test is to determine the depth to which a truncated No. 2 sewing needle penetrated a bitumen sample under specified conditions of load, time and temperature. In 1915, ASTM even went as far as specifying the brand of needle (R.J. Roberts Parabola Sharps No. 2) (Halstead and Wellborn, 1974). Indian Standards (IS) 73 penetration graded specification for bitumen was first formulated in the year 1950 and was called as ‘Specification for asphaltic bitumen and fluxed native asphalt for road making purpose’. Five grades viz. S35, S45, S65, S90 and S200 were included in that specification. In 1961, this specification was revised to incorporate the methods of test as per IS 1201 to IS 1220: 1958 ‘Methods for testing tar and bitumen’. In this revision, paving grades were included deleting the fluxed native asphalt grades. In 1992, again there was a revision in the specifications taking into account of binders having high wax content. Additional specifications related to penetration ratio, paraffin wax content, viscosity at 60°C and 135°C and retained penetration after thin film oven test were incorporated in this revision. Two types of technical specification namely type 1 paving bitumen from non-waxy crude and type 2 paving bitumen from waxy crude were proposed. Type 1 paving bitumen had six grades classified based on penetration value and type 2 bitumen was classified into four grades. Subsequent amendments to this specification were carried out in 2002.

Understanding the limitations of the existing penetration grading system in characterizing paving bitumen, a revised Viscosity Graded (VG) specification was incorporated during 2006 in India in line with ASTM D3381 (2005) (Table 1) specifications for Asphalt binders below with only four paving grades viz. VG10, VG20, VG30 and VG40 of bitumen. Empirical tests or parameters such as penetration ratio, paraffin wax content and Fraass breaking point were eliminated in this revision. For each grade, a minimum value of 60 °C viscosity and a penetration range was provided in this revision. This was further revised in 2012 giving greater emphasis to the viscosity measurement at 60 °C providing viscosity ranges for all the four paving grades of bitumen. Also, a minimum value of penetration at 25°C is stipulated instead of a range for penetration as specified in the earlier revision. This revision has also rationalized the binder selection process by categorizing the binder grade based on design maximum air temperature. Hence, the choice of the grade depends upon the design maximum air temperature of the location where the binder has to be used. For each grade of bitumen, the range of viscosity values and minimum penetration value at 25°C are specified. Ductility test is no longer mandatory for specification compliance. Table 2 below presents the specification as per IS73 (2012). With all this recent improvements in standards for bitumen testing in India one has to understand the fact that all these tests are empirical in nature and are single point measurements taken at a discrete set of temperatures. The mechanical behavior of the binder over a wide range of temperatures cannot be cleary characterized by these tests. Also, the relationship between the index properties of bitumen and field performance of the binders is quite questionable. In an attempt to address these issues, during 1987, the United States (US) congress initiated a five year $150 million product driven research effort called SUPERPAVE (SUperior PERforming PAVEments) under the Strategic Highway Research Program (SHRP) which includes development of performance based specification for binders. On similar lines, India needs to develop performance linked specifications for binders in the near future (Sairam and Krishnan, 2009).

Table.1.Requirements for Asphalt Cement, Viscosity Graded at 140°F (60°C) (ASTM D3381, 2005)

Test	Viscosity Grade					
	AC-2.5	AC-5	AC-10	AC-20	AC-30	AC-40
Viscosity, 140°F (60°C), P	250 ± 50	500 ± 100	1000± 200	2000 ± 400	3000 ± 600	4000 ± 800
Viscosity, 275°F (135°C), min, cSt	125	175	250	300	350	400
Penetration, 77°F (25°C), 100 g, 5 s, min	220	140	80	60	50	40
Flash point, Cleveland open cup, min,° F (°C)	325 (163)	350 (177)	425 (219)	450 (232)	450 (232)	450 (232)
Solubility in trichloroethylene, min, %	99.0	99.0	99.0	99.0	99.0	99.0
Tests on residue from thin-film oven test:						
Viscosity, 140°F (60°C), max, P	1250	2500	5000	10 000	15 000	20 000
Ductility, 77°F (25°C), 5 cm/min, min, cm	100[A]	100	75	50	40	25

Table.2.Requirements for Paving Bitumen (IS73, 2012)

Table 1 Requirements for Paving Bitumen

(*Clause* 6.2)

Sl. No.	Characteristics	Paving Grades				Methods of Test, Ref. to IS No.
		VG10	VG20	VG30	VG40	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Penetration at 25°C, 100 g, 5 s, 0.1 mm, *Min*	80	60	45	35	IS 1203
ii)	Absolute viscosity at 60°C, Poises	800 - 1200	1600 -2400	2400 -3600	3200 - 4800	IS 1206 (Part 2)
iii)	Kinematic viscosity at 135°C, cSt, *Min*	250	300	350	400	IS 1206 (Part 3)
iv)	Flash point, (Cleveland open cup), °C, *Min*	220	220	220	220	IS 1448 (Part 69)
v)	Solubility in trichloroethylene, percent, *Min*	99.0	99.0	99.0	99.0	IS 1216
vi)	Softening point (R&B), °C, *Min*	40	45	47	50	IS 1205
vii)	Tests on residue from rolling thin film oven test					
	Viscosity ratio at 60°C, *Max*	4.0	4.0	4.0	4.0	IS 1206 (Part 2)
	Ductility at 25°C, cm, *Min*	75	50	40	25	IS 1208

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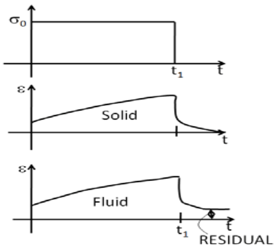
RHEOLOGY OF BITUMEN

Rheology is the study of the flow and deformation of all forms of matter (Bingham 1944 and Reiner 1964). Bituminous binder demonstrates different rheological behavior at different temperatures (-40 ºC to 160 ºC) say from a glassy solid - viscoelastic solid - viscoelastic fluid - non–Newtonian fluid -Newtonian fluid during pavement construction and in-service (Krishnan and Rajagopal, 2005). Under normal pavement temperatures and traffic loadings, the binder acts like a viscoelastic fluid exhibiting both elastic and viscous characteristics. With the gradual change in temperature during daily variations, the binder response changes from viscoelastic fluid to viscoelastic solid and vice-versa. The response of the pavement to traffic loading depends on the temperature and the rate of loading (speed of traffic) due to the viscoelastic nature. At high temperatures, a slow moving vehicle can damage the pavement due to rutting. This rutting consists of two parts, densification and shear flow. While the densification is believed to be influenced by the reduction of air voids in the material, the shear flow is attributed to the influence of the flow of the binder at high temperatures. One can expect considerable accumulation of irrecoverable deformation leading to rutting at high temperatures where the binder exhibits viscoelastic fluid behavior. Figure 1(a) & (b) shows the relationship between rutting seen in a pavement and its creep and recovery. Figure 1(a) & (b)clearly shows the densification (depressions in the wheel path) and shear flow (minor humps in the sides of the wheel path) and Figure 1 (b)shows the schematic figure of strain accumulation when the material is a viscoelastic fluid.

Understanding damage accumulation in bituminous mixtures is difficult. This makes developing a relationship between fatigue cracking of the pavement and rheological properties of the binder more complex. Most of the fatigue failure-related specifications are therefore ad-hoc. For instance, it is not clear whether the fatigue crack propagates from the top or bottom of a pavement. If one uses linearized elastic theories, the maximum tensile strain leading to crack initiation and propagation can occur only at the bottom of the bituminous layers. However, linearized viscoelastic theories show that considerable tensile strains are induced at the top. The role of the thickness of the bituminous layer is also important in cracking failures. Hence it is not uncommon to see different specification criteria for quantifying the fatigue failure for different pavement layer thickness. Figure 2(a) shows the fatigue cracking of a typical bituminous pavement. In order to relate the rheological property of the binder to the fatigue property of the mixture, a small amplitude oscillatory shear is conducted on a binder under aged conditions. The variation of linear viscoelastic properties such as dynamic modulus is taken to quantify the damage accumulation. Figure 2 (b) shows the dynamic modulus of binder at different temperature. Here, the point of drastic decrease in dynamic modulus indicates the cracking in bitumen. Hence, this figure indicates that the sample fails in cracking earlier at lower temperatures. At lower temperatures, viscoelastic response dominates and to quantify damage accumulation in the bituminous pavement, an understanding on the viscoelastic solid/fluid behavior of bitumen is essential (Padmarekha and Murali Krishnan, 2011).

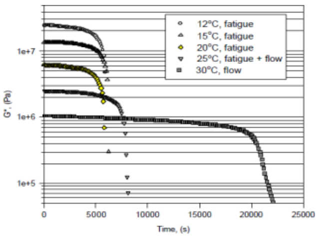
(a) Rutting (Pavement Interactive Guide, 2013)



(b) Creep and Recovery (Wineman and Rajagopal, 2000)

Figure.1.Rutting of Pavement and Relations to Creep and Recovery

(a) Fatigue Cracking (FHWA, 2013)



b) Time Sweep at Different Temperatures (Soenen and Eckmann, 1999)

Figure.2.Fatigue Cracking and Small Amplitude Oscillatory Shear

Bitumen characterization challenges: Bitumen is a very complex chemical mixture whose compositions vary widely depending primarily on the crude source and the manufacturing process. Low API gravity crudes contain large proportion of bitumen whereas high API gravity crude contain minor fraction of bitumen (Rajan, 2008). The traditional methods for processing bitumen from crude are: vacuum distillation, atmospheric distillation, solvent refining and air blowing as shown in Figure 3 (Corbett, 1984). Bitumen can be straight away reduced to the required grade depending on the properties of the vacuum residue and the grade of bitumen required. Also through the Propane De Asphalting (PDA) process (shown as Ppt. bitumen) production of bitumen can be carried out through blending. Light gas oils are used as a blending component if the viscosity of the vacuum residue is found to be very high. It can also be blended with low viscous vacuum residue. In some refineries a PDA pitch of low penetration value (approximately 10 pen) is blended with heavy oil to obtain the required grade of bitumen. The proportion of PDA pitch and heavy extract for blending depends on the required grade of bitumen. Generally, 90% of pitch is blended with 10% of heavy extract to obtain VG30 grade of bitumen (Rajan, 2008). Alternately, the low viscous vacuum residue can be air blown to the required grade. The air blowing process converts high 'penetration' vacuum residue to low 'penetration' bitumen. In the blending process, the low 'penetration' pitch is converted to high 'penetration' bitumen. The air blown and blended bitumen undergo completely different thermal histories during processing and hence exhibit different thermo-rheological behavior during service. At a specific temperature, the rheological properties and aging characteristic of air blown and blended bitumen of same viscosity grade exhibit mechanical response that are of different orders of magnitude (Rajan, 2008).

In India, bitumen is produced from crudes procured from a number of sources using at least two different manufacturing processes (Rajan, 2008). It is not known to the highway engineers which crude is used and what processing method is followed. One can clearly understand the influence of crude source, the processing method and cut point temperature on the specification parameters from Figure 4 below. It is seen that all the products manufactured from sources A, C and E (shown in Figure 4) while meeting the same specification parameters are completely different in their chemical composition. Clearly this is the contribution of the nature of the crude sources and the magnitude of cut point used in reducing the material to such grades.

The widely used chemical characterization technique for bitumen is contributed by Corbett (1969). Corbett used a fractionation procedure to separate bitumen into four generic fractions in the order of their increasing molecular polarity as saturates, naphthene aromatics, polar aromatics, and asphaltenes. Since this underlying chemical composition demonstrates the rheological behavior of the material, it is necessary that over and above such specification parameters viz. viscosity, penetration etc., some constraints in possible chemical composition is also introduced during production of bitumen. For instance, knowing how the asphaltene content dictates the Newtonian viscosity of the binder can help to provide upper and lower bounds for asphaltenes.

Also, the effect of aging on the evolution of the internal structure of bitumen has to be understood. Aging of binders during construction and during performance is well known to pavement engineers (Petersen, 1993 and 2009, Robertson, 1991). Aging understandably makes the binder material much stiffer which results in increase in viscosity. Currently, a Rolling Thin Film Oven Procedure (RTFO) is the standard durability test used throughout the world simulating the short term aging occurring during construction. Laboratory simulation of long term aging of the pavement during its performance is also necessary and this is normally carried out by Pressure Aging Vessel (PAV). It is important to note that Indian standards for bitumen do not have any specification inbuilt to address this situation (Sairam and Krishnan, 2009).

Figure 5 below illustrates the relationship between asphaltene content and apparent viscosity at 60 ^{o}C for a blended bitumen from Basrah Light Crude at three different blend proportions say 90:10, 85:15 and 75:25 of PDA pitch : Heavy Extract at three aging conditions unaged, RTFO aged and PAV aged respectively. From the figure, one can observe significant variations in the viscosity values per unit asphaltenes given within the parenthesis for different blend proportions of bitumen at unaged, short term aged (RTFO) and long term aged (PAV) conditions. Also one can understand that only blends with a lower proportion of PDA pitch (75:25) shows the least change in viscosity per unit change in asphaltene as an effect of aging. As asphaltenes are produced in oxidative aging of bitumen, the addition of more asphaltene (through more PDA pitch) essentially creates bitumen that is likely to fail much earlier due to cracking. On the other hand, blends with higher PDA pitch content (90:10) (more asphaltene content) can withstand the plastic deformation normally expected in the initial life of the pavement. But from this one cannot conclude that asphaltenes are fully responsible for the viscoelastic behavior of bitumen. Considering the fact that bitumen is made up of different component fractions as discussed above from the literature, one can argue that it is the association of asphaltenes with the remaining component fractions that holds the key and such associations have not been understood fully until now (Rajan, 2010).

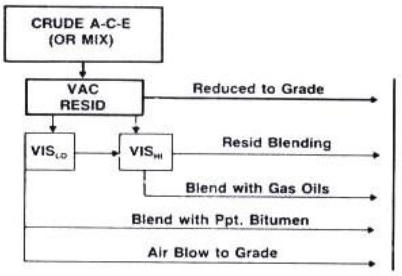


Figure.3.Possible Ways of Manufacturing Bitumen (Corbett, 1984)

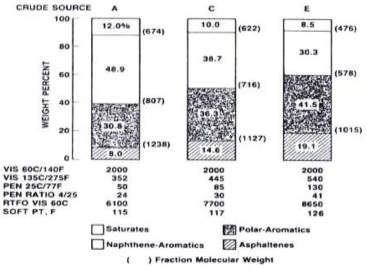


Figure.4.Similar Specification Parameters–Different Chemical Composition (Corbett, 1984)

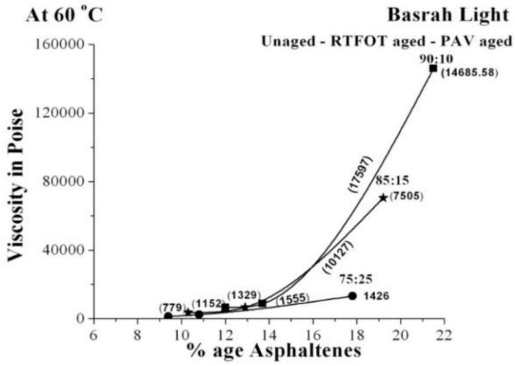


Figure.5.Percentage asphaltenes vs. viscosity at 60°C: Basrah Light Crude (Rajan, 2010)

Performance based specifications: Very little work has been done in India to establish a connection between field performance and the index properties of bitumen. This task includes collection of significant database related to different rheological properties of bitumen including the information about the crude source, processing method, chemical composition at the unaged condition as a first step. As discussed earlier as such the specification parameters currently followed in India are measurements carried out at discrete sets of temperatures with little connectivity to the field requirements. These grading systems such as penetration or viscosity can be met by changing the proportion of pitch to heavy extract (blending) or blowing temperature, air pressure, circulation rate and use of catalyst (air blowing). By this one can produce the same penetration/viscosity grade of bitumen from a wide variety of crude sources but cannot produce similar performance grade properties from such crudes without changes in the production process.

Figure 6 below shows the performance grade properties of the same grade of binder produced from different crude sources (Sairam and Krishnan 2009). They concluded that the penetration or viscosity grading system cannot differentiate between the different performance characteristics of the processed binders. In India, bitumen is produced in most refineries by adjusting the process parameters to meet penetration grade at 25 °C and other related specification parameters at specified discrete temperatures. When the same binders are checked for performance grade properties as per ASTM - D6373 (2007), they exhibit interesting trends. One must understand the underlying rheological behavior over a range of temperatures before attempting to produce bitumen fulfilling its performance requirements (Sairam and Krishnan, 2009).

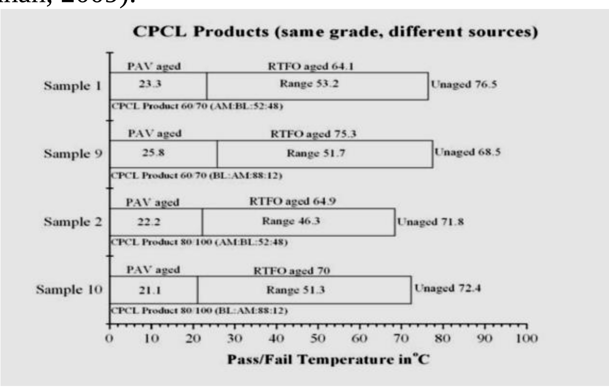


Figure.6.Performance Grade Properties –Same Grade, Different Sources (Sairam and Krishnan, 2009)

SUMMARY

Indian specification for paving bitumen IS 73:2012 includes only empirical tests such as penetration and viscosity which are measured at a discrete set of temperatures. Very little work has been done till date in India to establish a connectivity between field performance (durability) and the index properties of bitumen. Rheological behavior of bitumen influences the performance of real life pavements. Due to a wide range of temperatures existing in India, bituminous binders used in pavements demonstrate different rheological properties. While a viscoelastic fluid like behavior of the binders at high temperatures is attributed to rutting; a viscoelastic solid like behavior at very low temperatures is attributed to fatigue cracking. Development of a performance based specification is urgently needed for rational binder characterization in India. This task includes systematic collection of significant database of different rheological properties of bitumen including the information about different crude sources, processing methods, chemical composition at the unaged condition as a first step. Also a detailed study on the effect of aging on binder rheology and the inter-relationship between the rheology and chemical composition of bitumen has to be carried out.

REFERENCES

ASTM D3381-05, Standard Specification for Viscosity-Graded Asphalt Cement for Use in Pavement Construction, Annual Book of ASTM Standards, 04.03, West Conshohocken, Pennsylvania, USA, 2005.

ASTM D6373-07, Standard Specification for Performance Graded Asphalt Binder, Annual Book of ASTM Standards, 04.03, West Conshohocken, Pennsylvania, USA, 2007.

Asphalt Institute, Superpave Mix Designs, Superpave series no. 2. Report no. SP-2, Asphalt Institute, Lexington, KY, USA, 2003.

Bingham EC, The History of the Society of Rheology from 1924-1944, January 1944.

Corbett LW, Refinery processing of asphalt cement, Transportation Research Record, 999, 1–6, 1984.

FHWA, http://www.fhwa.dot.gov/pavement/recycling/98042/03.cfm," Accessed on December 2013.

Halstead WJ and Wellborn JY, History of the Development of Asphalt Testing Apparatus and Asphalt Specifications, Asphalt Paving Technology Proceedings, 43A, Association of Asphalt Paving Technologists Historical Session, 26 February 1974, Williamsburg, 89-120, 1974.

India Road Network in Ministry of External Affairs, Investment and Technology Promotion (ITP) Division Government of India, http:/www.indiainbusiness.nic.in/industry-infrastructure/infrastructure/road.htm, Accessed on December 31st, 2013

Indian Standards IS 73, Paving Bitumen Specifications (Third Revision), Indian Standards Institution, New Delhi, 2006.

Krishnan JM and Rajagopal KR, On the Mechanical Behavior of Asphalt, Mechanics of Materials, 37, 2005, 1085–1100.

National Highway Authority of India (NHAI), Indian Road Network in National Highways Authority of India. http://www.nhai.org/roadnetwork. htm, Accessed on December 31st, 2013

Padmarekha A and Murali Krishnan J, Experimental Investigation on High Temperature Transition of Bitumen", Construction and Building Materials, 25(11), 2011, 4221-4231.

Petersen JC, Asphalt Oxidation – An Overview Including a New Model for Oxidation Proposing that Physiochemical Factors Dominate the Oxidation Kinetics, Fuel Science and Technology International, 11(1), 1993, 57-87.

Petersen JC, A Review of the Fundamentals of Asphalt Oxidation: Chemical, Physicochemical, Physical Property and Durability Relationships, Technical Research Circular EC140, Transportation Research Board, Washington DC, 2009.

Pavement Interactive Guide, http://www.pavementinteractive.org/article/rutting/, Accessed on December 2013.

Reiner M, The Deborah number, Physics Today, 62, January 1964.

Rostler FS and Sternberg HW, Compounding Rubber with Petroleum Products Based on Asphalt Composition Using Precipitation Methods, Proceedings of Quality Control and Acceptance Specifications, Vol.2, Asphalt Technology, 1965.

Robertson RE, Branthaver JF, Plancher H, Duvall JJ, Ensle EK, Harnsberger PM, and Petersen JC, Chemical Properties of Asphalts and Their Relationships to Pavement Performance, Proceedings of the Association of Asphalt Paving Technologists, 60, 1991, 413-436.

Rajan NK, Selvavathi V, Sairam B and Murali Krishnan J, Rheological Characterization of Blended Paving Asphalt", Road Materials and Pavement Design, 9(SI), 2008, 67-86.

Rajan NK, Selvavathi V, Sairam B and Murali Krishnan J, Influence of Asphaltenes on the Rheological Properties of Blended Paving Asphalts, Petroleum Science and Technology, 28, 2010, 331–350.

Sairam B and Krishnan JM, Assessment of the Performance Grade Properties of Binders Processed at CPCL, Chennai Petroleum Corporation Limited, Chennai, 2009.

Soenen H and Eckmann B, Binder-Related Fatigue Properties Studied by Rheology, Proceedings of the Durable and Safe Road Pavements, Transportation Research Board, 1, 1999, 189–97.

Wineman AS and Rajagopal KR, Mechanical Response of Polymers: An Introduction, Cambridge University Press, United Kingdom, 2000.