

Rheological Study Of Bituminous Modified Pavement Mixes

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ABSTRACT

The empirical tests such as penetration, softening point and ductility have been used for characterization of bitumen in India. The rheological characterization of bituminous binders based on the properties such as penetration, softening point, ductility and absolute viscosity are not satisfactory to characterize the bituminous binders. In the present study, dynamic shear rheometer was used for characterizing the bituminous binders which measures rheological properties directly. The rheological properties of modified binders were analyzed using Dynamic Shear Rheometer (SR5 Ashphalt Rheometer) and testing was performed in temperatures from 46°C to 82° C in increments of 6°C at a frequency of 10 rad/s. A comparison has been made between empirical tests results and the rheological properties obtained from Dynamic Shear Rheometer like complex modulus (G^*) , phase angle (δ) and dynamic viscosity (η ') were discussed in this paper. The results from the present study indicate that the rheological properties increases as the modifier concentration increases. It is observed that the complex modulus increases with decrease in penetration values but phase angle increases with increase in penetration values.

AIMS AND STRUCTURE OF THE THESIS

Correlations of chemical and thermal properties with low-temperature rheological characteristics of bitumen

An effort will be made to correlate the molecular weight and aromatic properties of bitumen with its low-temperature rheological behavior. Potentially, the results of such analysis could provide insight into the relation between the structural and lowtemperature dynamic properties of bitumen. In addition, possible correlations between the glass transition temperatures (Tg), as measured by differential scanning calorimetry (DSC), and lowtemperature rheological properties of bitumen will be investigated. The aim of this analysis is to study whether and how the low-temperature thermodynamic and dynamic properties are related in bitumen.

Physical aging of bitumen as studied by the 4-mm DSR technique

It will be investigated whether physical aging in bitumen can be effectively quantified by the 4-mm DSR method. Further, the applicability of timeresolved mechanical spectroscopy (TRMS) and timeaging time superposition principle in the analysis of physical aging data will be tested.

chapter 1 gives an introduction to the experimental section of this thesis. The basic physical and thermal properties of the investigated bitumen samples are presented, followed by the description of the characterization techniques used in the laboratory **INTRODUCTION**

The rheological characterization of bituminous binders is generally based on two properties, penetration and softening point (Ring and Ball). The penetration indicates the distance; a needle will penetrate into the bituminous binder under specified conditions of loading and time, in tenths of a millimeter. The softening point is the temperature at which the consistency of the binder is such that a steel ball placed on a disk of binder in a brass ring will cause a certain deformation of the disk under specified conditions. Bitumen's are viscoelastic materials and their mechanical behavior is dependent on both the temperature and the loading times. At low temperatures and short loading times they behave as elastic solids, while at high temperatures and long times of loading they behave as viscous liquids. The specifications for bituminous binders typically based on measurements in empirical tests are not adequate for fully describing the linear viscoelastic properties. The Strategic Highway Research Program (SHRP) developed the new specifications for rheological characterization of binders. The rheological properties of the binders as per SHRP are determined by using Dynamic Shear Rheometer in terms of complex modulus, G* and phase angle, δ (Khalid et al. 1998).



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In order to characterize Polymer Modified Asphalts (PMA), it has been shown that traditional tests (Penetration, softening point, absolute viscosity) are usually inadequate to illustrate the increase in performance brought by polymer addition to the (Lenoble and Nahas, 1994). The bitumen performance can be characterized by means of fundamental rheological characterization of the binders. Test methods and apparatus for this type of fundamental characterization have improved enormously over the past two decades. Due to the relative newness of these tests and high initial cost for the acquisition of the testing apparatus, the binder industry is reluctant to use them (Ven and Jenkins, 2003). The conventional tests methods are still used to investigate the properties of the binders in many countries including India. Therefore, the relationship between empirical tests results and the rheological properties obtained from Dynamic Shear Rheometer like complex modulus, phase angle and dynamic viscosity is discussed in this paper. The rheological behavior of bitumen is very complex phenomenon, varying from purely viscous to elastic, depending on loading time and temperature. As a viscoelastic material, bitumen plays a prominent role in determining many aspects of road performance. For example, a bituminous mixture (asphalt) needs to be flexible enough at low service temperatures to prevent pavement cracking and to be stiff enough at high service temperatures to prevent rutting. These functional properties are required to enable pavements to accommodate increasing traffic loadings in varying climatic environments [1]. Unfortunately, due to the increased performance related requirements on asphalt pavements, the asphalt containing conventional bitumen does not always perform as expected. Therefore, in order to improve the asphalt properties modified bitumen has been adopted and widely used in the industry as commercial product. The uses of virgin polymers in bitumen to improve the characteristic COPPER SLAG(CS) of resulting polymer modified bitumen have been accomplished for many years. Recently there is interest in the substitution of commercial virgin material by recycled polymers. When recycled polymers are used as bitumen modifying agents, the resulting mixture may show similar performance to those containing virgin polymers [5]. Casey et al. [6] stated that in general, recycled plastic COPPER SLAG(CS) have poorer mechanical properties than the virgin ones; they concluded that although the recycled polymer modified binder did not perform to the same high levels as the available proprietary commercial binder, it did however demonstrate enhanced performance when compared with unmodified binders. These results suggest that the recycled polymer modified binder has great promise. From an environmental and economic standpoint, the use of recycled instead of virgin materials help easing landfill pressures and reducing demands of extraction.

LITERATURE REVIEW: Bituminous roads form the major part of road network in India. The high intensity of traffic in terms of commercial vehicles and the over loading of trucks have been responsible for early development of distress symptoms like undulations, rutting, cracking and potholing of pavement. The factors, which are of serious concern, are the varying climatic conditions prevalent in India and the inadequate quality control during the construction of the roads. Poor performance of conventional bituminous mixtures under severe exposure condition and high traffic has led to the increased use and development of modified binders. Addition of modifiers into conventional bitumen results in the improvement of performance characteristic COPPER SLAG(CS) of bituminous mixes used in road construction (Burger et.al (2001) and Ismail (2007)).

The principle modes of failure of bituminous courses are fatigue and rutting which are influenced by temperature. Bitumen being a viscoelastic material, factors such as temperature, rate of loading and number of repetition of loads have a significant effect on the performance. Ageing of bitumen is a very complex process leading to hardening of bitumen. Due to ageing there is decrease in penetration and increase in softening point, higher the softening point lower the temperature susceptibility and higher resistance to deformation (**Mohammad Alawi** (2005)).



The durability properties in terms of resistance to ageing of the bitumen binder, is the key factor for the binder characterization in asphaltic mixes and hence pavement performance. The bitumen in the asphalt pavement will undergo two distinct hardening phenomena. The first is the short-term hardening, which occurs during mixing production and construction, it is the initial stage, due to these losses (Asphaltene increases, agglomeration of resins into bigger molecules and evaporation of lighter fractions of maltenes) and oxidation takes place (Charles Gloveret.al (2002) and White oak (1990)).

Second is the long-term hardening, which occurs during in-servicepavement life over long time and with continuous degradation due to varying temperature and climatic conditions. Bituminous materials exhibit viscoelastic response and the performance of flexible pavements depends on the rate of loading and temperatures. The performance of bituminous binders can be measured by means of rheological parameters (complex modulus, storage modulus, loss modulus and phase angle) over a wide range of temperatures and frequency (Abdelaziz Mahrez and Mohamed Rehan Karim(2003)).

In the present paper, the results of rheological properties of base and modified binders are reported for a wide range of temperature from 50 to 80°C at a frequency of 10Hz for virgin and aged bitumens and Marshall properties (stability and flow) for base and modified bitumens and how the conjectures made on the basis of rheological properties correlated with the Marshall properties are reported.

Pundhir et al (2005) studied that the copper slag (CS) was used as a fine aggregate (up to 30%) in the design of bituminousmixes like bituminous macadam, dense bituminous macadam, bituminous concrete and semi-dense bituminous concrete. Marshall Method of mix Design is adopted in which CS and stone dust as fine Aggregate and hydrated lime as a filler. OBC for different mixes was found as follows BM, 3.5, DBM, 5.3, BC, 5.9, and SDBC, 5.9%. Addition of CS a fine aggregate in various bituminous mixes provides good interlocking and eventually improves volumetric and mechanical

properties of bituminous mixes. Because of improved property by the incorporation of copper slag it can be used as a fine aggregate in bituminous mixes as the substitute of crusher dust as fine aggregate.

Havanagi et al (2012) The waste like copper slag, zinc slag, steel slag and pond ash were investigated for their suitability inroad embankment and sub grade layers, while copper slag and zinc slag may be used as a partial replacement of fine aggregate for the construction of sub base, base and bituminous layers. The specific gravity of copper slag, zinc slag and steel slag varied in the range of 2.75 - 3.6, while pond ash exhibited low value of specific gravity of 2.27. The bulk density of Marshall Samples varied in the range 24.4 kN/m3 to 25.6 kN/m3, Marshall stability value in the range 8.7 kN to 14.7 kN, Flow value in the range 3 mm to 4 mm, % air voids value in the range 4.13 to 7.19 and optimum bitumen content varied in the range 3.4 % to 7.19 %. Copper slag and zinc slag materials can be used as a replacement of fine aggregate in sub base, base (WMM) and bituminous layers of road pavement. The amount of replacement varies from 20 to 30 % in WMM mix and 10 - 25 % in the bituminous mixes.

Kajal et al (2007) Study present the use of waste plastic and copper slag in hot bituminous mix to enhance pavementperformance, bituminous mixes were prepared by mixing of graded mineral aggregate and increasing percentage of binder content 5.0,5.5,6.0,6.5% by wt. of mineral aggregate. Optimum binder content is achieved at 5.2% by weight of mineral aggregate specimens are also prepared with composition of CS(10,15,and 20%) replacing stone dust ,CS(15%)has been found best.

Debashish Kar et al (2014) Were Investigated the influence of fly-ash as a filler in bituminous mixes ,For comparison, controlmixes with cement and stone dust have also been considered. Marshall test has been considered for the purpose of mix design as well as evaluation of paving mixes. Marshall stability and unit weight increase with bitumen content up to 5% after which these two parameters decrease. At any bitumen content the stability value and unit weight are highest for mixes with cement as filler followed



by that with stone dust and fly ash. flow value increases with bitumen content. However, the flow value is lowest for mixes with cement as filler compared to stone dust and fly ash. In the similar manner the air void decreases with increase in bitumen content. However it is to be highlighted that the fly ash causes maximum reduction of air voids compared to the other two fillers. Hence, it is generally concluded that the fly ash can effectively be used as filler in paving mixes in place of most commonly used fillers such as ordinary Portland cement and stone dust.

METHODOLOGY

The viscoelastic behavior of bitumen is exceptionally complex to depict by basic traditional experiments of consistency, for example, penetration tests and softening point tests. Hence, the assessment of bitumen attributes ought to be focused around its performance regarding fatigue and rutting safety. Hence, new test instruments like the Dynamic Shear Rheometer (DSR), Brookfield Viscometer have been created to give rheological properties of bitumen over an extensive variety of loading and encompassing conditions. The DSR might be acknowledged as the most compelling and complex instrument for characterization of the bitumen flow properties. It is additionally really vital to comprehend the chemical progressions of bitumen that has been made throughout change by sulphur . To study the chemical compound arrangement framing, thermal and morphological investigation of modified bitumen, a few tests have been led utilizing new innovation instruments, for example, FESEM, TGA, DTA and FTIR Spectroscopy individually.

Determination of rheological properties of bitumen: Rheological properties are utilized as execution parameter has favorable circumstances and disadvantage. The point is that it permits estimation of physical properties with wide temperature range at high and low recurrence, which is prone to be accomplished in the field because of movement. Dynamic shear rheometer need qualified individual with high encounter to work the element tests and additionally to get great rheological results. In this section a concise representation of the element shear rheometer (DSR) device and in addition the geometry and example creation and example measurement will be exhibited. In this section additionally a point of interest description of all rheological test methods





Specimen geometry: The DSR geometry was picked as indicated by the test condition and particular. The 25mm diameter geometry with specimen thickness (1mm) utilized for high temperature test to spare the sample from dissolving. At intermediate road temperature the sample ought to have little diameter (8mm) with specimen thickness (2mm) to keep it from fatigue failure. The DSR geometry has been shown in [figure 3.5].The bitumen specimen is sandwiched between two parallel plates. The upper plate geometry is permitted to pivot about its own particular hub while base plate stays settled throughout testing.



Phase Angle: For an applied stress shifting sinusoidal with time, a viscoelastic material will additionally react with a sinusoidal strain for low amplitudes of stress. The sinusoidal variety in time is typically portrayed as a rate. Phase angle is defined as the lag between the applied stress and resulting strain of a body when subjected to a sinusoidal shear



stress. The phase angle is a critical parameter to portray the viscoelastic behavior of bitumen, which is a yield result from the dynamic mechanical examination through DSR.



Stress Level, Strain Amplitude and Frequency of Oscillation: A viscoelastic material, bitumen does not carry on straightly as far as their stiffness as a function of stress or strain. Accordingly the dynamic shear modulus and phase angle rely on the size of the shear strain with both expanding and diminishing shear strain. A linear region is characterized as at little strains where the complex shear modulus is autonomous of shear strain The point of confinement of the straight viscoelastic behavior is characterized as the measured quality of G* reductions to 95% of its zero strain esteem. The rheological tests ought to be performed with in the straight viscoelastic area of bitumen performance. So a strain range test was led at 60°c as demonstrated for VG-30 and sulfur modified bitumen.



EXPERIMENTAL **PROGRAM:** The work demonstrated in this section has been partitioned into four zones. The primary zone of this study comprises the type of material used, their standard properties and sample preparation for testing. The secondary region of study depicts the operational confinements of the Viscometry and DSR in wording of connected stress levels and recoverable strain levels along with the testing conditions of samples. The third range of study examined the impact of different temperatures on the physical properties tests. The fourth zone of this study summarized with chemical, morphological and thermal analysis with testing conditions of the

samples. In this study the rheological, physical, storage stability, chemical, thermal and morphological properties of both unmodified and modified bitumen, their working standards have been briefly discussed.

Material :It is known from the studies that the level of modification relies on upon the neat bitumen type and modifier type. Different studies have been carried out in the field of sulfur modification and there are a few descriptions for the need of utilizing modifier within bitumen industry. There are different explanations behind utilizing bitumen modifier within bitumen industry began with expansion the service life of the pavement, enhance its performance, meet the overwhelming traffic demands and at last saving the expense of maintenance. In this test project viscosity grade bitumen VG-30 has been utilized. The physical properties of VG-30 bitumen were given in table underneath.





At first the Rheological properties of sulfur extended bitumen have been tested to know about the progressions in the viscoelastic properties of modified specimen. To discover a good structure for a good sulphur modified bitumen , four steps are carried out under which a few sets of tests are to be directed with viscometry and DSR instrument to evaluate the rheological properties of sulphur extended bitumen to evaluate the optimum sulfur content and ideal condition for proper modification and are explained below.

Softening point Test: In this test bitumen sample in fluid condition poured in a brass ring, levelled and kept for 30 minutes at room temperature. Glass beaker containing distilled water kept at B.O.D. incubator at 5°C for 30 minutes. A steel ball of



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weight 3.5 g is placed on a bitumen sample contained in a brass ring that is suspended inside a water bath, maintaining bath temperature to be raised at 5°C per minute. The softening point of bitumen was determined as per IS: 1205-1978.Temperature at which the bitumen sample touches the lower plate is reported as softening point of that sample.

Ductility Test: The test has been carried out at a temperature 27^{0} C and a rate of pull of 50mm/min. The experimental procedure has been followed according to ASTM D113 –The distance from the starting point of bitumen thread formed to the broken point of bitumen thread was reported as ductility value.

Elastic Recovery Test: Sample preparation up to attachment of mould with sample and briquette in ductility testing machine is same as ductility testing of sample. The experimental procedure has been followed according to ASTM D6084. After that elongation of sample was done up to a distance of 10 cm at speed of 50 mm/min. Sample at that condition was for 1 hour at 15 °C. After that the two broken sample was made to come closer and the distance was recorded. Using formula as given below in equation 4.1, the percentage elastic recovery was reported

RHEOLOGICAL INVESTIGATION

The rheological behavior of sulfur modified binder at different level of modification was compared with that of the base binder i.e. VG 10 used in the study. The viscoelastic behavior of bitumen can be characterized using Dynamic Mechanical Analysis (DMA) with the help of Dynamic Shear Rheometer (DSR) having parallel plate geometry. It can be operated in stress as well as strain controlled modes. Controlled strain mode is normally used to determine dynamic mechanical properties of bitumen. A sinusoidal stress or strain amplitude is applied to a sample sandwiched between two plates with the lower plate fixed and the upper plate applying the oscillatory load.For a controlled strain mode the following defines the evaluation for the viscoelastic parameters

γ=γ₀ sin ω

 $\omega = 2\pi f$

Where, $\Upsilon 0 = \text{peak}$ oscillatory strain

 ω = angular/rotational frequency, defined as

Where, f= frequency, Hz

The corresponding stress response is given by the equation

$\sigma = \sigma_0 \sin(\omega t + \delta)$

It is the phase difference between the stress and strain and is also called the loss angle or phase lag (Fig.2). For a purely elastic material the value of δ is 0° and for a purely viscous material it will be 90°. It is thus a very important parameter in describing the viscoelastic property of bitumen.



Rheological Characterization

To study rheological properties of each sample, the Rotational Viscometer (RV) tests were conducted utilizing a Brookfield DV-III Ultra viscometer equipped with thermosel. The basic RV test measures torque required to maintain a constant rotational speed of a specified spindle (in this study, spindle SC27 was used) submerged in liquid asphalt at a constant temperature. The data is then used to calculate viscosity from a measured torque. The viscosity of non-aged samples in the presence of various amounts of silica fume (2, 4 and 8%) was measured by viscometer, following the ASTMD4402 specification. Viscosity was measured at four different temperatures (105, 120, 135 and 150°C) and six different speeds (5, 10, 20, 25, 50 and 75 RPM). At each temperature, the reading was recorded after every 30 min.



REASEARCH RESULTS AND THEIR ANALYSIS: This section portrays the rheological properties in terms of fatigue and rutting behavior results for sulphur modified bitumen as well as short dissection of test information. The skeleton of testing covers was chosen keeping in mind the end goal to research that impact of sulfur in the bitumen subjected properties to distinctive loading parameters. The rheological properties of the different binders were portrayed utilizing dynamic shear rheometer over wide ranges of temperatures and frequencies. In this study both VG 30 bitumen and its modification with sulphur was tried and a summary of all results introduced underneath in tables and graphical structure.

Effect on penetration values with different modifiers: The penetration values are decreasing significantly for 80/100 bitumen mixed with CR and SBS. It is observed that the penetration value decreases as the concentration of modifier increases. Further, the bitumen modified with SBS seems to be more effective in the penetration values as compared to CR modifiers for 80/100 grades of bitumen. As per IRC: SP: 53-2002, the 80/100 bitumen modified with CR from 3% to 9% is found to be of CRMB 50 grade. The 80/100 bitumen modified with 7% and 9% of SBS show the characteristics similar to 30/40 grade bitumen (PMB 40). Hence this type of modified binders can be used for hot climate areas and heavy traffic conditions.

Effect on softening point values with different modifiers: It is observed that the softening point increases with increase in percentage of modifiers. The effect of SBS on softening point is much more than that of CR. Further, there is not much effect on softening point on CR mixed bitumen. The softening point for 80/100 bitumen increases from 47 to 54°C when 9% CR is added. The softening point increases more than 70°C by addition of 5%,7% and 9% SBS. As per IRC: SP: 53-2002 and IS 15462: 2004, binders modified with 3% to 9% CR was falling in CRMB 50 grade and it can be used for cold climatic areas. The softening point increases as the bitumen becomes increasingly viscous DSR test results of modified binders: There are several major distresses of road pavement related to bitumen properties. Since bitumen is a viscoelastic material, its rheological properties are very sensitive to temperature as well as the rate of loading. The relationship between the rheological characteristics of bituminous binder and field performance illustrates the importance of understanding the rheological bahaviour of bitumens (Navarro et al., 2002). The rheological properties such as Complex modulus (G*), Shear modulus (G*/sin\delta), Loss modulus (G"), Storage modulus (G') and Phase angle (δ) are evaluated by using Dynamic Shear Rheometer (DSR).



Correlation between phase angle at different temperatures and penetration value: The phase angle of binders increases with increase in penetration value. The phase angle of binders modified with CR and SBS are lower as compared to neat bitumen as shown in Figures 5and 6. It is found that the regression coefficients (R^2) of modified binders are varied at different temperatures. The regression coefficient (R^2) for 80/100 bitumen modified with CR is less as compared to modified with SBS. The SBS modified binders ($R^2 = 0.9887$) indicates better relationship than CR modified binder $(\mathbf{R}^2 = 0.7698)$ at the same temperature and penetration values as shown in Figures 5 and 6. From the regression equations, phase angle can be determined at any penetration value. The phase angle of binders modified with SBS is lower than binders modified with CR. The penetration values of the modified binders decrease as the percentage of modifier increases. Therefore, the phase angles of



modified binders decrease as the percentage of modifier increases. Decrease in phase angle indicates lower viscous flow and higher elastic behaviour of the binders.



CONCLUSIONS

The empirical tests such as penetration, softening point and elastic recovery are improved with addition of the SBS and CR. SBS modified binder gives higher softening point and better elastic recovery than CR modified binders. The complex modulus increases with increase in percentage of modifier and decreases with increase in temperature. However, phase angle decreases with increase in percentage of

modifier and increases with increase in temperature. The increase in complex modulus and decrease in phase angle of the modified binder indicate higher resistance to deformation as compared to neat bitumen. The SBS modified binder exhibit a higher stiffness and elasticity than CR modified binders at high temperatures. There is significant improvement in the rheological properties when the SBS content is increased from 3% to 7% by weight. The improved rheological properties of the binders are not directly proportional to the modifier content. The rheological properties show good curve with empirical binder properties. Shear modulus ($G^*/\sin \delta$) is found to be in important roles to characterize the rheological properties and predict performance of modified bitumens at high temperature.Bitumen (80/100) modified with 3 % and 5 % crumb rubber are meeting the specification requirements of PG 58 and PG 64 respectively. However, SBS modified binder with the same percentage above are satisfying the PG 64 and PG 70 requirements. It is clearly seen that same grades modified bitumen specified by IRC falls in different performance grade (PG) specified in SHRP. Therefore, it is suggested that rheological property such as $G^*/\sin \delta$ should be used in India also for development of the performance grades as per SHRP specifications.

FUTURE WORK

Future work should also further investigate and model the relaxation time spectrum of bitumen in the vicinity of the glass transition. It is expected that the extremely broad distribution of molecular size and composition in bitumen averages relaxation processes in a way that will generate basic patterns which might be universal to all types of polydisperse glasses. This is remarkable since the glasses investigated and modeled so far in the rheological literature have been almost exclusively monodisperse in nature. Also, the modeling of the low-temperature rheological data of bitumen with the 1S2P1D model is a fascinating topic for future research.In the future, it would be also interesting to study the low-temperature rheological properties of bitumens modified with other additives than SBS polymer. For example, chemical modification with thiourea and some



reactive prepolymers has shown great promise in improving the low-temperature performance of bitumen [233-237]. Moreover, some lightweight oil products have been shown to improve the thermal cracking resistance of bitumen [238]. The author feels that it would be intriguing to investigate especially modified bitumens of the aforementioned types with the 4-mm DSR technique. In addition, it would be interesting to try to extend the 4-mm DSR technique to the low-temperature rheological characterization of mastics.

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