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EFFECT OF ANTIOXIDANTS PHENOL AND AMINE ON RHEOLOGICAL PROPERTIES OF BITUMEN

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***Abstract*—This paper deals with the laboratory investigations on antioxidants namely Phenol and Diethyl amine modified bitumen. Conventional physical tests like penetration, softening point and viscosity were performed as per Indian standard codes guidelines using modifier content of 0-5%, with increment of 1% by weight of penetration grade 80/100 bitumen. Rheological properties such as Complex Modulus and Phase Angle, Permanent deformation resistance ($G^*/Sin\delta$) and Fatigue resistance parameter ($G^*.Sin\delta$) were also performed with modifier content of 0-5%, with increment of 1% using Dynamic Shear Rheometer (DSR). The effect of phenol on penetration reveals that with increase of phenol content penetration increases whereas with increase of diethyl amine penetration decreases. Softening point in case of phenol decreases whereas in case of diethyl amine increases. Viscosity value was represented with increase of temperature and found its trend, decreases with increase of temperature in both the modifiers. Complex modules and phase angle varies with increase of temperature from 40^{o}C to 90^{o}C for both the cases and shows same nature of variations. With increase of modifier content Permanent deformation resistance is increasing. The modified bitumen has higher complex modulus than plain bitumen which means that it is more stable. This means that using modifiers with the plain bitumen increases the binder elasticity at high temperatures and improves the flexibility at low temperature thereby lessening both rutting and fatigue cracking. Comparison of the rutting parameter ($G^*/Sin\delta$) shows that modification can improve the rutting resistance of bitumen. So as far as the analysis of fatigue resistance parameter ($G^*.Sin\delta$) is concerned, it was shown that the modification reduces the fatigue life at the medium temperature ranges.**

Keywords— ***Phnol & Amin, Viscosity, Rutting resistance***

I. INTRODUCTION

In India, the methods for rheological characteristics of bituminous binders are inadequate to characterize the bitumen. Hence a complete rheological study and characterization of bitumen using Dynamic Shear Rheometer (DSR) would be helpful. Bitumen is an important binder used in flexible pavement construction. About eighty percent of the produced bitumen is used for pavement construction. The oxidative behaviour of bitumen is thought to be one of the critical factors contributing to the performance of hot-mix. Oxidation of asphalt is a major cause of pavement failure owing to hardening of the asphalt binder with accompanying changes in viscosity, separation of components, embrittlement and loss of cohesion and adhesion of the asphalt in the mix. The major concern for cracking of road surface is overloading of commercial vehicles and increases in traffic density and the reasons behind it, is the loss of cohesion and heavy stiffness or hardness of bitumen film and failure of adhesive bond between aggregates and bitumen. With the use of antioxidants in bitumen binders, age-hardening can be reduced. The Rheological properties such as Complex Modulus and Phase Angle of bitumen needs to be enhanced with the use of antioxidants in bitumen binders, in order to provide more durability to pavement under extreme climatic condition and increased traffic density.

Cubuk M.et al (2014) studied on influences of phenol formaldehyde resin on the rheological properties of petroleum bitumen. Different amount of phenol formaldehyde were doped into the bitumen of 50/70 penetration grade and variations in viscosity as a function of temperature and additive concentration were determined. The effects of the phenol formaldehyde additive were examined by conventional and super pave test methods. *Dessouky S. et al* (2013) used hindered phenol to improve the rheological characteristics of base (unmodified) and polymer-modified bitumen. Rheological properties were measured using a rotational viscometer, a dynamic shear rheometer and a bending beam rheometer. Stiffening indices and complex modulus indices were used to assess the effectiveness of the additive in the bitumen and its thermal stability respectively.it was found that the additives were capable of increasing the stiffness at high temperature and reducing the hardening propensity of aged bitumen at intermediate and low temperatures. *Iskender E. et al* (2012) studied to analyse effects of SBS polymer and fatty amine anti-stripping agent additives for asphalt mixtures. Three types of moisture conditioning were selected. Regimes were based on water immersion, freeze-thaw cycles and superposition of these. Repeated creep tests and indirect tensile tests were applied.

The aim of this study is to investigate the effect of antioxidants on conventional properties of bitumen and rheological properties of bitumen with and without antioxidants (phenol and diethyl amine).

II. EXPERIMENTAL PROGRAMME

A. Material

Materials used in this study includes 80/100 penetration grade bitumen obtained from road construction department, Government of Bihar, Patna. Two types of antioxidants are used like Phenol and Diethyl amine to modify bitumen of 80/100 penetration grade bitumen. Both antioxidants were obtained from local market at Patna. An aromatic compound contain -C_6H_5 (phenyl group) and whose molecular formula is C_6H_5OH is recognised as Carbolic acid. It has bonding with -OH group. Carbolic acid is volatile in nature and a white crystalline solid. The molecular structure of Diethyl amine is $CH_3CH_2NHCH_2CH_3$ and also written as $C_4H_{11}N$.It is powerful and flammable alkaline liquid.

B. Sample Preparation

Samples were prepared using mix blending method. The bitumen about 500gm was heated on the hot plate heater and phenol was slowly added. Mix was stirred slowly and temperature was kept between 100^0C to 120 ^{0}C. In the same way diethyl amine based mix was prepared. Similarly different mix was obtained at different proportion of antioxidants like 1%, 2%, 3%, 4% and 5% by weight of bitumen. The modified bitumen was then sealed in containers covered with aluminium foil and stored for further testing.

C. Laboratory Testing

Test on the prepared samples were conducted according to the Indian Standard codes, ASTM and AASHO test guidelines. Effect of Antioxidants (Phenol & Amine) on Rheological Properties of Bitumen was investigated along with conventional physical test like Penetration, Softening point and Viscosity.

D. Penetration

Penetration test is performed according to the IS: 1203-1978 guidelines. The standard 100g, 25^0C and 5 sec penetration test was performed. Test result on plain bitumen and both antioxidants modified bitumen at different percentage is presented in Table1.

E. Softening point

Ring and ball is the standard test as per IS: 1205-1978 to determine the consistency of the bitumen, which represents the temperature at which a change of phase from solid to liquid occurs. It is the temperature at which standard 3/8 inch steel ball weighing 3.55gm fall and touches the base plate which is 2.5mm away. The results are shown in Table 1.

Table-1: Result of penetration and softening point

Percentage of antioxidant used	Penetration at 25^{o}C, 0.1mm, 100g, 5sec (Average of three samples)		Softening point (R &B), ^{o}C (Average of three samples)	
	Method of test IS No. 1203		Method of test IS No. 1205	
	Phenol	Amine	Phenol	Amine
0	81	84	49	49
1	94.2	79.5	48.75	51
2	97.6	73	48	53.5
3	109.6	71	47.25	54
4	118.6	67.5	46.5	55.5
5	123.8	62	45.75	57

F. Viscosity (ASTM D4402)

Viscosity test was conducted using rotational visco-2000 viscometer. The visco 2000 viscometer is designed to perform ASTM D4402 high temperature testing of asphalt binder. The viscometer is a constant speed motor with a torque detection system. The sample is placed in the gap between upper and lower measuring system. Lower measuring system is a 27.5mm wide cup in which the sample is placed. This fits inside the heater casing. Upper measuring system is a bob (concentric cylinder) which is lowered into the cup. The bob has a conical base. The inner cylinder diameter is 25mm.The instrument uses a controlled shear rate. That is, it applies a shear rate (rotational speed) and measures the resultant shear stress (torque) needed to maintain shear rate.

G. Rheology (AASHTO T315-05)

A dynamic shear rheometer is commonly known as DSR. The DSR is used to characterize the viscous and elastic behaviour of asphalt binders at medium to high temperatures. This characterization is used in the Superpave PG asphalt binder specification. As with other Superpave binder tests, the actual temperatures anticipated in the area where the asphalt binder will be placed determine the test temperatures used. The basic DSR test uses a thin asphalt binder sample sandwiched between two circular plates. The lower plate is fixed while the upper plate oscillates back and forth across the sample at 10 rad/sec (1.59 Hz) to create a shearing action. The test is largely software controlled. The DSR measures a specimen's

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complex shear modulus (G*) and phase angle (δ). The complex shear modulus (G*) can be considered the sample's total resistance to deformation when repeatedly sheared, while the phase angle (δ), is the lag between the applied shear stress and the resulting shear strain. The larger the phase angle (δ) means the more viscous the material. Phase angle (δ) limiting values are δ = 0 degrees for purely elastic material and δ = 90 degrees for purely viscous material.

III. RESULT AND DISCUSSION

As per the penetration test conducted on plain bitumen, the penetration lies 80 to 100, hence it is graded as 80/100 bitumen. Test result shows that the penetration value of the binder increases as the amount of phenol increases. This indicated that the bitumen get softer as increasing percentage of phenol. Result shows that the antioxidant reduces the oxidation of the binder which makes the binder softer. This can help in reducing the cracking of the road surface. The penetration test result for both modifiers is shown in the Fig. 1.Whereas with addition of amine penetration decreases and become harder. Therefore, amine modified bitumen can be used in hot climatic region and phenol modified bitumen can be used in cold climatic region.

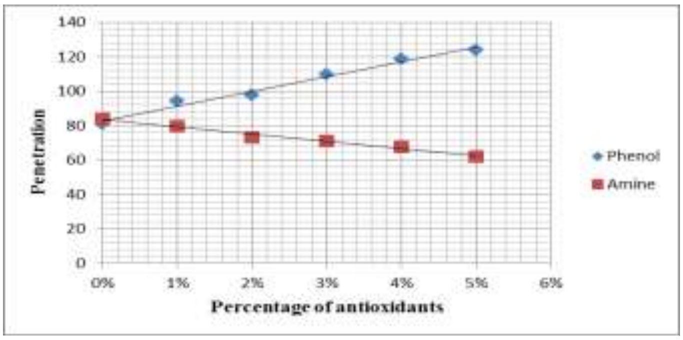

Fig. 1. Effect of antioxidants on penetration

The test result shows that the softening point of the modified bitumen decreases as the amount of phenol increases whereas softening point increases with increase of amine from 1 % to 5%. This shows that increasing amount of phenol makes bitumen less temperature susceptible and increasing amount of amine makes bitumen more temperature susceptible. The test result is shown in the Fig. 2.

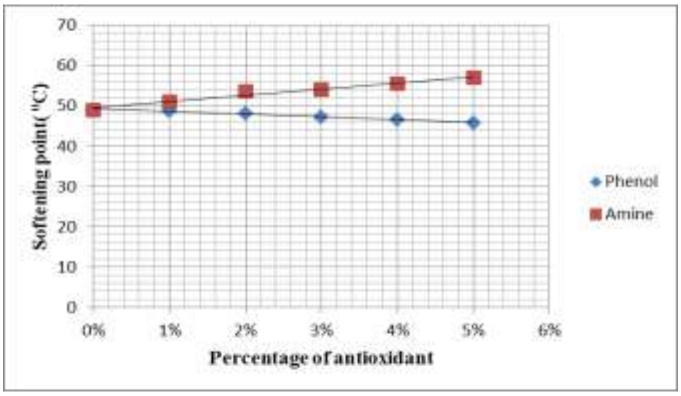

Fig. 2. Effect of antioxidants on softening point

Viscosity test result shows that with increase of temperature viscosity is decreasing. Viscosity increases with increasing amount of both antioxidants content as shown in Fig. 3. & Fig. 4. There is difference in viscosity of almost 1Pas as increase of phenol content but less than 1Pas at low temperature and more at high temperature in case of amine. This trend and observation may improve the property of complex modulus and make the material more viscous.

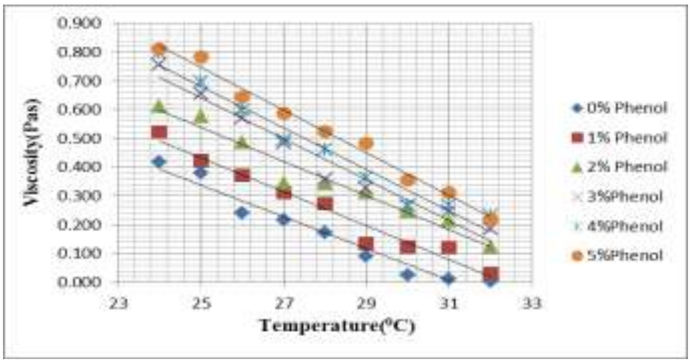

Fig. 3. Effect of Phenol on viscosity with rise of temperature

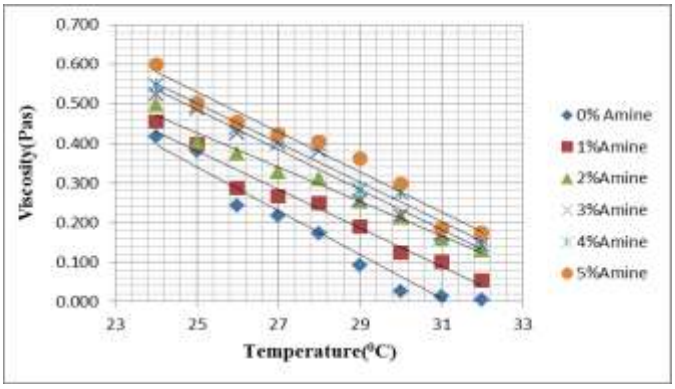

Fig. 4. Effect of amine on viscosity with rise of temperature

Phase angle of the phenol modified bitumen increases with increasing amount of phenol up to temperature 75^0C and beyond this slightly decreases as shown in Fig.5.. Phase angle of amine modified bitumen increases with increasing amount of amine as shown in Fig. 6.The increasing value of phase angle shows that the softness of bitumen increases. Result shows that the both modified bitumen inclined towards the viscous properties of bitumen.

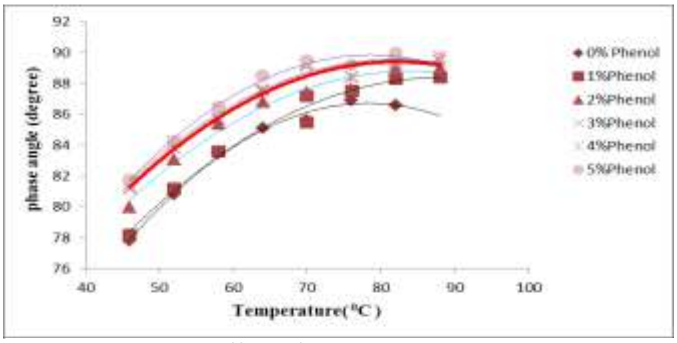

Fig. 5. Effect of Phenol on phase angle

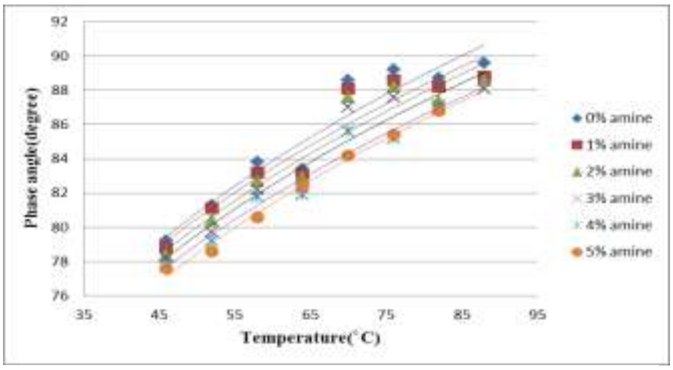

Fig. 6. Effect of amine on phase angle

Complex modulus of phenol modified bitumen in comparing to unmodified bitumen decreases with increase of temperature whereas Complex modulus of amine modified bitumen in comparing to plain bitumen increases with increase of temperature as shown in Fig. 7-8 respectively. Decreasing complex modulus reduces the cracking behaviour. Decreasing value of complex modulus shows the reduction in hardness and increase in softness properties of bitumen binder.

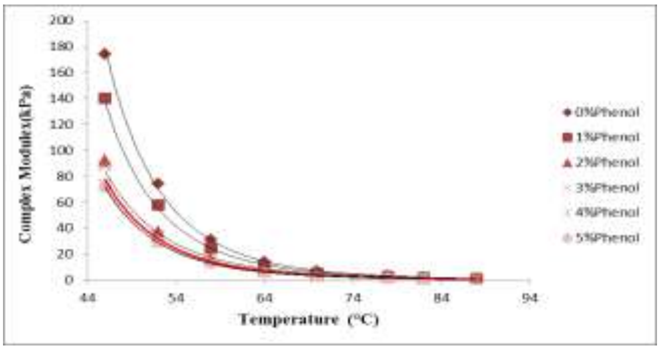

Fig. 7. Effect of phenol on complex modulus

It is observed that the plain bitumen fails at temperature 78^{0}C but phenol and amine modified bitumen are stable. But whereas phenol and amine fails at 88 ^{0}C this clearly indicates that antioxidants used are less susceptible to temperature changes when compared to unmodified bitumen.

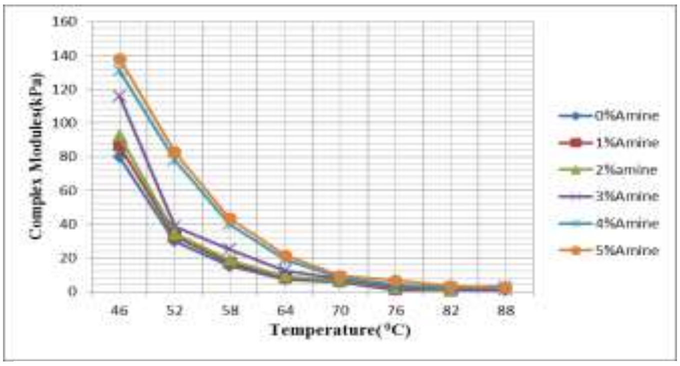

Fig. 8. Effect of amine on complex modulus

The value of $G^{x}/\sin\delta$ increases with increasing amount of phenol and amine as shown in Fig. 9-10 respectively. This reduces the rutting behaviour of the bitumen. Due to the heavy axel load the chances of cracking and permanently deformation increases in the case of stiffer bitumen. These results, therefore, prove that the amine modified bitumen performs much better than the unmodified bitumen in rutting performance.

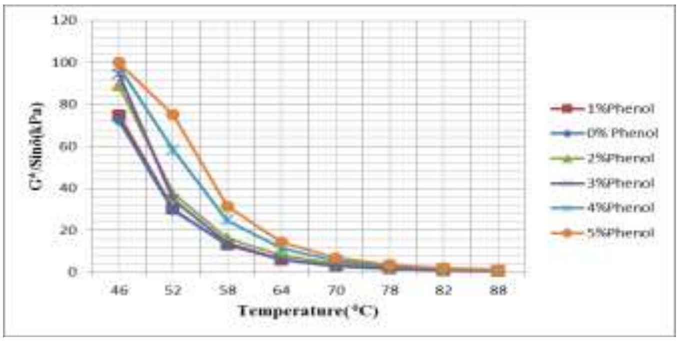

Fig. 9. Effect of phenol on rutting resistance parameter

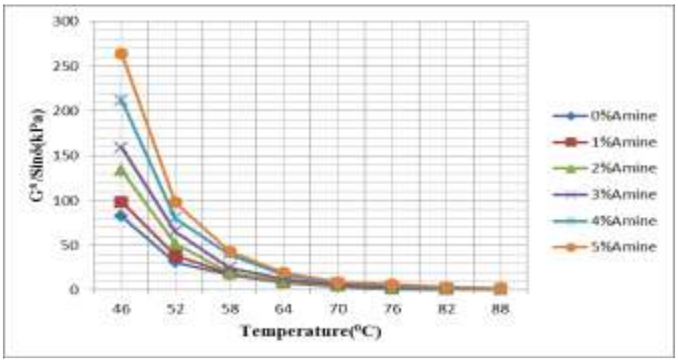

Fig. 10. Effect of amine on rutting resistance parameter

G*Sinδ is known as Fatigue resistance parameter. Effect of phenol and amine on fatigue resistance parameter is shown in Fig.11-12 respectively. With increase of percentage of phenol and amine fatigue resistance parameter is increasing. In case of phenol the value of G*Sinδ is 150 kPas for 5% of phenol modification but in case of amine the value of G*Sins is 138kPas for 5% of amine modification. Thus the observation shows that the phenol modification reduces the fatigue life of bitumen at the medium temperature ranges.

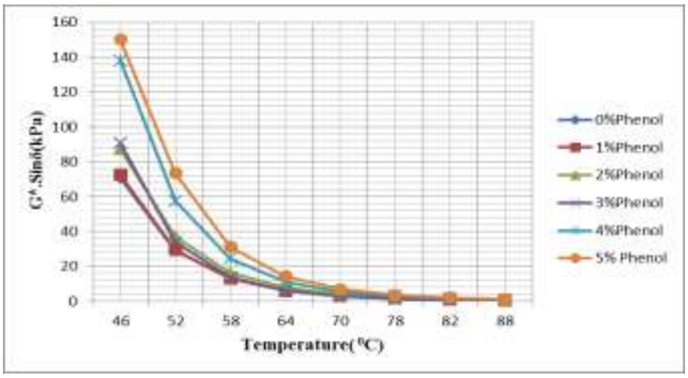

Fig. 11. Effect of phenol on fatigue resistance parameter

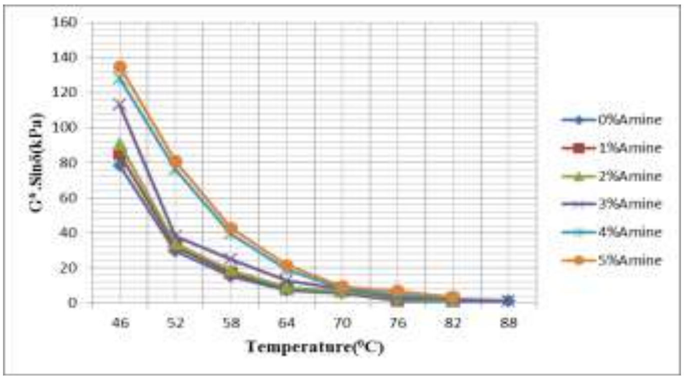

Fig. 12. Effect of amine on fatigue resistance parameter

IV.CONCLUSION

The addition of phenol to the bitumen results in the improvements in the basic properties like penetration and viscosity whereas softening point decreases. The addition of amine to the bitumen decreases the penetration and increase the softening point. Relatively phenol modified bitumen gives higher viscosity than the amine modified bitumen. Phase angle at 90 ^{0}C for the amine modified bitumen is more than the phenol modified bitumen. This shows the more viscous material. For 5% of phenol modified bitumen at 45^{0}C the complex modulus is high with respect to the amine modified bitumen. The phenol modified bitumen has higher complex modulus than plain bitumen which means that it is more stable. This means that using phenol with plain bitumen increases the binder elasticity at high temperature and improves the flexibility at low temperatures thereby lessening both rutting and fatigue cracking. Therefore, it is recommended to use the 5% phenol modified bitumen.

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