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Optimum Blending Requirements for EVA Modified Binder

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Abstract: Increase in traffic, new axle configurations and high temperature demands the use of modified binders for resisting the stresses induced in pavement. Ethylene Vinyl Acetate (EVA) is a plastomer, which is a copolymer obtained by copolymerization of ethylene and vinyl acetate. Though a potential modifier, problems of phase separation have been encountered attributable to the presence two separate phases of bitumen and polymer which are incompatible with each other.

This paper focuses on obtaining the optimum blending requirement for EVA copolymer. A VG 10 binder was modified at varying percentages of EVA from 1 to 7%. Modification was carried out at different combination of mixing temperature, blending time and shear rate and a total of 80 combinations were obtained. Statistical package for social sciences (SPSS) and SOLVER function was used to assess the optimum blending requirement. Further the paper evaluated the optimum modifier content for obtaining a homogenous blend which could be stable at high temperature. Physical and rheological properties of the modified binder was also evaluated and compared with the base binder.

It was found that temperature is the most critical parameter for EVA modification. Shear rate had the minimum influence over obtaining a storage stable blend. Fluorescence microscopy showed change in morphology as the modifier content increased, which could be used to assess the optimum modifier content for modification. Rheological response of the modified binder significantly improved. EVA modification was found to be best suited at high temperatures and could be very effective to resist rutting of mixtures.

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***Key words**: Bitumen; Modifier; Pavement; Rheology.*

Introduction

Bitumen is a viscoelastic material and the only component in the pavement structure showing thermo-rheological behavior [1]. Increase in temperature, loading and introduction of new axle configuration in highways demands better materials and design methods. Conventional binder are no longer able to satisfy these increasing demands and use of modified binders has become one of the best ways to offset these issues [2-7]. Modification of bitumen brings changes in its mechanical properties by increasing the viscoelastic response of the binder [3, 4, 8, and 9].

Polymers, both elastomers and plastomers are one of the potential modifiers which improve both conventional and rheological properties of binder [5-7, and 10]. This paper is divided into two parts. The first part describes the procedure for development of modified binder by finding the optimum blending requirements, followed by assessing the optimum modifier content for obtaining a homogenous blend. The second part presents the rheological properties of the Polymer Modified Binder (PMB), prepared with optimum modifier content, as obtained in the first part of the study.

Experimental Investigation

Materials

Modified binder was produced using the following materials:

1. VG 10, viscosity graded bitumen, collected from Mathura refinery.
2. EVA copolymer supplied in pellet form of 2-3 mm in size. Modification was done using different percentage of EVA varying from 1-7%.

EVA is a thermoplastic copolymer obtained by copolymerization of ethylene and vinyl acetate. Depending on the percentage of vinyl acetate, the properties of the copolymer changes, low vinyl acetate showing similar behavior to that of low density polyethylene (LDPE). EVA is characterized by its melt flow index (MFI) and vinyl acetate content. MFI measured in g/10 minutes is a viscosity test which is inversely related to molecular weight. Higher the MFI lower is the molecular weight and viscosity. Vinyl acetate on the other hand provides amorphous and rubbery properties to the bitumen. The polyethylene segments which are crystalline in nature are bonded together with the vinyl acetate group, the later disrupting the crystalline nature of the system. Hence higher the vinyl acetate lower is the crystalline nature of the bitumen.

Methods

Optimum Blending Requirements

When a polymer is mixed in bitumen, compatibility due to difference in polarity, molecular weight and typical structure of polymer and base bitumen plays a critical role [1, 11, and 12]. Also the competency of the polymer and asphaltene for the solvency of maltene fraction in the bitumen may disturb the polymer-bitumen system leading to phase separation. Various researchers [11-14] have used different methods for modifying bitumen using EVA. The main difference is found in use of different mixing temperature, blending time and shear rate for producing the PMB.

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So the first part of the study focusses on obtaining optimum blending requirements for EVA modified binder by varying the above mentioned parameters. Modification was done at four different mixing temperatures (160˚C to 190˚C with increment of 10˚C), four different mixing time (20, 30. 40 and 60 minutes) and five altered shear rate (300, 600, 900, 1200 and 1500 rpm). A total of 80 combinations were obtained. The range of values for different parameters was selected after reviewing various literatures.

Storage stability (SS) value (Separation test) as mentioned in IRC SP 53-2010 [15] was used as the variable for achieving the goal. Storage stability is one of the most important tests which needs to be conducted when dealing with polymer modified bitumen. It explains the tendency of the base bitumen to sustain the polymer as a homogenous phase within the bitumen-polymer system. It may so happen that, two binders of similar grade, but of different chemical composition will have different forms of compatibility with the polymer. Storage Stability test will hence help to judge this discrepancy through physical testing. It is believed that at higher temperatures the modifier tends to separate from the base bitumen which could be influenced by inappropriate blending requirements used for modification. An aluminum tube, 25.4 mm diameter and 136.7 mm height, is filled with hot modified bitumen and is kept vertically at 163˚C for 48 hours. It is then immediately transferred to a freezer having temperature of 6.7 ± 5˚C and left for 4 hours to solidify. The tube is cut into three equal parts and ring and ball softening point is conducted on the bitumen sample obtained from the top and bottom parts. The difference in softening point temperature should not be more than 3˚C for the required storage stability.

Optimum Modifier Content

Once the blending requirement was set, PMB’s at different percent of modifier were produced maintaining the obtained values of mixing parameters. Next, the objective was to obtain the optimum modifier content for producing a homogenous mix which would be stable at high temperatures. Storage stability test and Fluorescence microscopy was used to achieve the second objective. Conventional test like penetration and softening point were also carried out to see the effect of modification on the consistency of bitumen.

The results obtained from penetration and softening point test were used to determine the temperature susceptibility of the base and modified binders. It provides a mean to assess the change in behavior of material response at varying temperature conditions. It is defined as the change in consistency parameter as a function of temperature. A classical approach as given in Shell Bitumen Handbook [16] was used to calculate the value of Penetration index (PI).

$$PI = \frac{1952 - 500\log(Pen_{25}) - 20 \times SP}{50\log(Pen_{25}) - SP - 120} \tag{1}$$

where, Pen_{25} is the penetration at 25˚C and SP is the softening point temperature of the PMB. The value of PI ranges from -3 for highly temperature susceptible bitumen’s to +7 for low temperature susceptible and highly blown bitumen [16].

Morphology of the EVA PMB’s was studied using Fluorescence Microscopy (FM). The nature and quality of dispersion of the modifier in the bitumen was assessed using this technique. It is based on the principle that polymers swell due to absorption of some of the light fractions of bitumen (mainly maltene fraction) and hence fluoresce in ultraviolet (UV) light. This fluorescence is due to the aromatic oils absorbed by the polymer. It is by far one of the most valuable methods to study the phase morphology of modified bitumen and assessing the homogeneity and the structure in raw state. The sample preparation method involves diluting the bitumen and preparing it over glass slide so that the beam could pass through the sample. The samples were examined using a Nikon Eclipse LV 100 microscope using appropriate magnification. High pressure Xenon lamp was used for excitation and the wavelength was maintained between 510-560 nm.

Rheological Viscoelastic Properties

The rheological behavior of PMB modified at optimum modifier content i.e. 5% was compared with that of the base binder i.e. VG 10 used in the study. The viscoelastic behavior of bitumen (especially polymers) 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 [17]. 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.

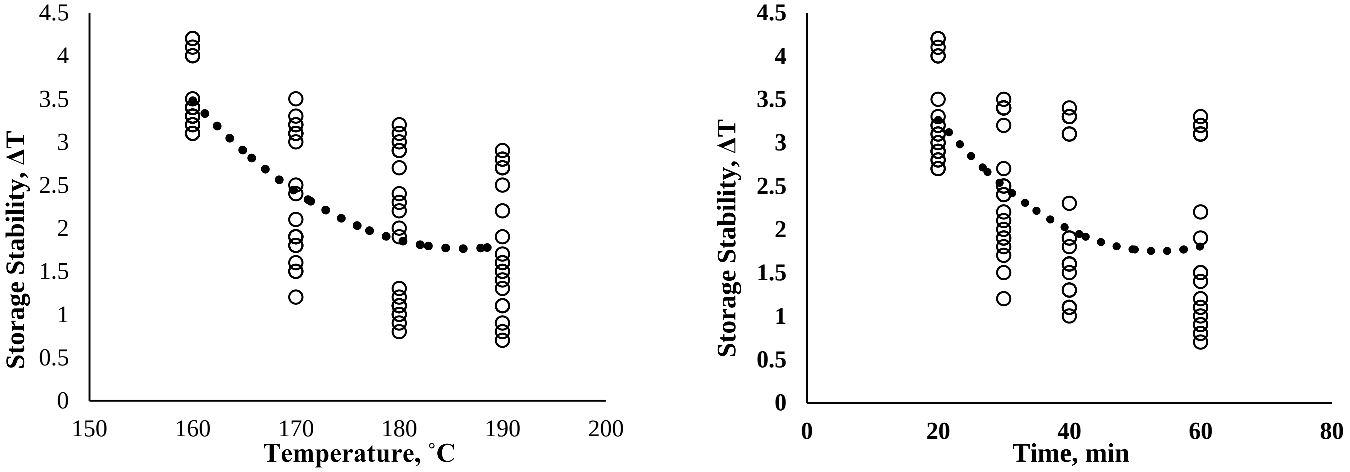
DSR testing can be conducted for a wide range of temperature and frequency. Different plate diameters are used for different testing conditions depending on the stiffness of the binder. The DSR test was done under controlled strain condition using temperature sweep test applied in the range of 10 to 70˚C at two different frequencies (0.2 and 1.97 Hz) corresponding to two different speed conditions (10 and 80 km/hr).

All the tests were done in linear viscoelastic (LVE) range depending on the stiffness of different blends [18, 19]. LVE was defined as the strain at which the complex modulus does not differ by more than 5% from its initial value. The sample was prepared using silicon mould method. The sample was sandwiched between the spindle and the base of the rheometer. The gap was adjusted according to the plate geometry adopted and the excessive sample was trimmed. The sample was allowed to equilibrate with the temperature for 10 minutes before staring the test.

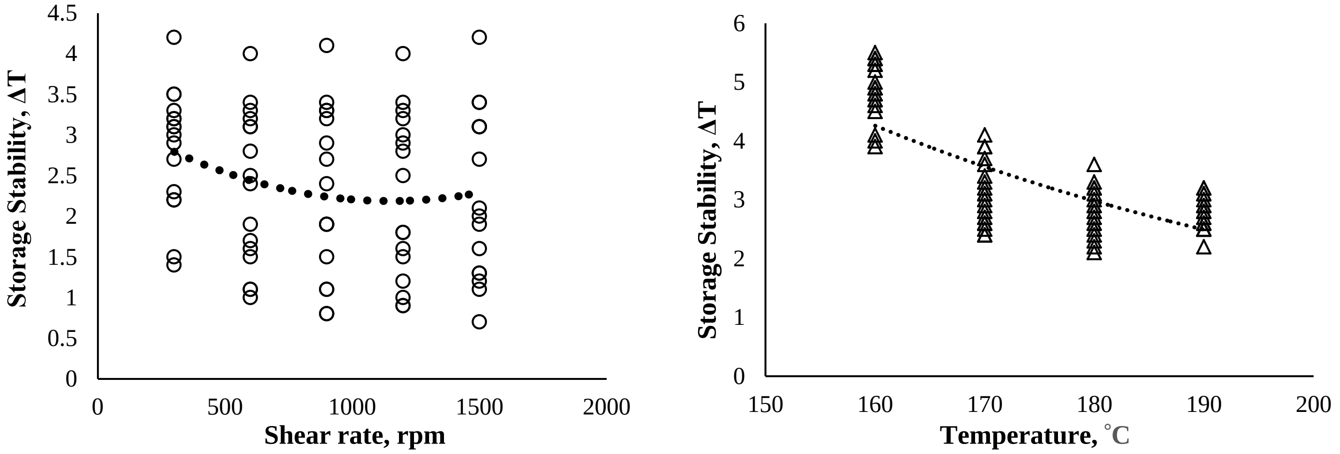
Results and Discussion

Optimum Modification Technique

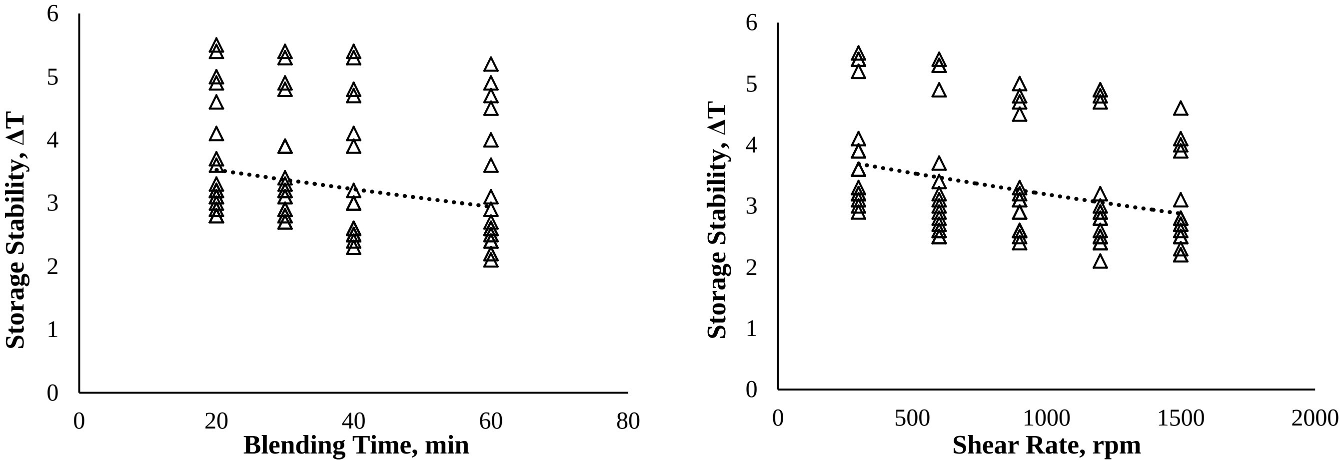
Separation test was done for all the 80 combinations obtained and for different percentage of modifiers. Fig. 1(a)-1(f) shows the variation for 2 and 5% modification. Result for all the percentages are not presented due to space restrictions. It can be seen that shear rate has the minimum effect on the variation of SS values while temperature had the maximum influence. It was found that the effect of shear rate became significant as the percent of modifier increased, showing maximum influence for 7% modification.



a) Variation of SS with Mixing Temperature, 2% EVA b) Variation of SS with blending time, 2% EVA



c) Variation of SS with shear rate, 2% EVA d) Variation of SS with Mixing Temperature, 5% EVA



e) Variation of SS with Blending Time, 5% EVA f) Variation of SS with Shear Rate, 5% EVA

Fig. 1. Variation of Storage Stability (SS) with Temperature, Blending Time and Shear rate for 2% and 5% EVA PMB.

Statistical package for social sciences (SPSS) was used to establish correlation between SS and all three parameters used. First it was checked if multi-collinearity existed between any of the independent variable used in the study i.e. temperature, time of blending and shear rate. As an example, result obtained for 3% EVA modification is presented in Tables 1 and 2. It was found that no collinearity existed between any of the independent variables,

Table 1. Correlation Values Obtained Using Linear Regression in SPSS for 3% EVA Modification.

Pearson Correlation	3% EVA	Temperature	Time	Shear
3% EVA	1	-0.728	-0.27	-0.36
Temperature	-0.728	1	0	0
Time	-0.272	0	1	0
Shear	-0.355	0	0	1

Table 2. Analysis of Variance (ANNOVA) Result for 3% EVA Modification.

Model	Unstandardized Coefficients		Standardized Coefficients	t	Correlations			Collinearity Statistics	
	B	Std. Error	Beta		Zero-order	Partial	Part	Tolerance	VIF
Constant	15.972	0.963		16.587					
Temperature	-0.066	0.005	-0.728	-12.199	-0.73	-0.81	-0.728	1	1
Time	-0.019	0.004	-0.272	-4.562	-0.27	-0.46	-0.272	1	1
Shear	0	0	-0.355	-5.944	-0.36	-0.56	-0.355	1	1

Table 3. Coefficients as Obtained Using Solver Function in MS Excel.

Percent Modifier	k	a_1	b_1	c_1
2	13.872	0.056	0.0345	0.0004
3	15.972	0.0655	0.0185	0.0008
5	16.044	0.0658	0.0137	0.0007

Table 4. Values of Optimum Blending Parameters as Obtained by the Regression Equation.

Percent Modifier (%)	Temperature (°C)	Blending Time (Minutes)	Shear Rate (rpm)
2	175	30	600
3	180	40	600
5	180	60	600

confirmed by the VIF and tolerance value in the coefficients table (VIF < 10, Tolerance > 0.1). Also inter correlation between the variables is less than 0.7. The equation generated by SPSS produced shear rate coefficient to be zero, as its effect was found to be minimum. To include the effect of shear rate, solver function in MS Excel was used to minimize the sum of least squares, assuming the relation between the dependent and independent variable to be linear of the form

$$SS = k + a_1 Temperature + b_1 BlendingTime + c_1 ShearRate \qquad (2)$$

where, k = Constant;

a_1, b_1, and c_1 = coefficients of the equation

Table 3 presents the value of the constant and coefficients for 2, 3 and 5% EVA PMB. It was seen that SPSS and solver produced almost the same equation coefficients. SPSS gives the result up to 3 decimal places and hence the coefficient for shear rate was found to be zero.

Eq. (2) was used to find the optimum blending requirement for each percent of modifier used and the constraint was set so that the SS value should be less than 3. The respective values for 2, 3 and 5% can be seen in Table 4. Examining the values for each percent modification a common blending requirement was set, irrespective of the percent modifier used. The adopted values of temperature, blending time and shear rate were set to 180˚C, 60 minutes and 600 rpm.

Optimum Modifier Content

Change in Conventional Properties

The effect of EVA modification on VG 10 can be seen from Table 5 as a decrease in penetration and increase in softening point. Fig. 2(a)-2(b) shows the variation of penetration and softening point as a function of EVA percentage. From Fig. 2(a) it can be seen that there is a sudden decrease in penetration after 2 % modification. This may be considered as the threshold percentage after which polymer network start dominating the bitumen-polymer system. Decrease in penetration and increase in softening point indicates increased stiffness and hardness of the binder after modification. In addition to this there is an increase in PI values as the percentage of EVA increases, indicating reduction in temperature susceptibility due to polymer modification.

Storage Stability Values

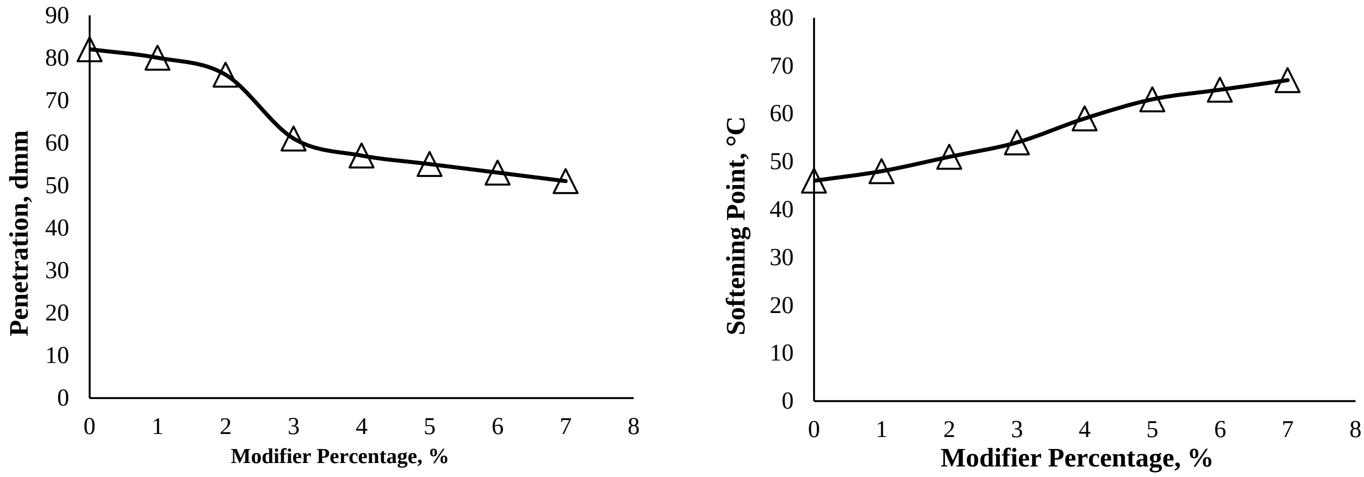
Storage stability is a measure of homogeneity at high temperatures. From Table 5 it can be seen that after 5% modification the difference in ring and ball softening point for the top and bottom samples in separation test exceeds 3˚C indicating phase separation. This may be due to the absence of adequate amount of maltene fraction to satisfy the demand of asphaltene and polymer solubility after EVA percentage exceeds this value. At low polymer content of about 2 to 3% there is practically no phase separation, hence it can be viewed as a bitumen (asphaltene) rich phase with polymer being dispersed thoroughly. So for EVA modified binder 5% can be considered as the optimum percentage for modification of VG 10 considered in the study.

Fluorescence Microscopy

The fluorescent images of PMB 0%, PMB 3%, PMB 5% and PMB

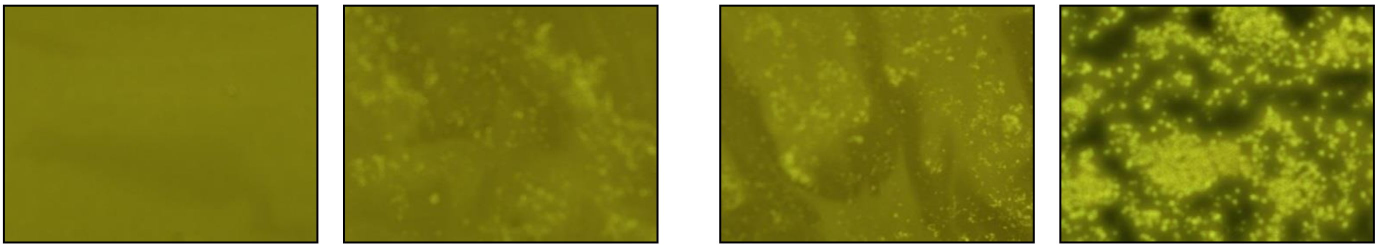
Table 5. Variation of Physical Properties Due to Modification.

Properties	Standard	Percentage of EVA (%)							
		0	1	2	3	4	5	6	7
Penetration (dmm)	ASTM D5	82	80	76	61	57	55	53	51
Softening Point (°C)	ASTM D36	46	48	51	54	59	63	65	67
Specific Gravity	ASTM D70	1.01	1.01	1.01	1	1	1	0.99	0.99
Penetration Index		-1.07	-0.56	0.11	0.24	1.16	1.86	2.13	2.38
Storage Stability (ΔT), °C	IRC SP53 2010	0	0.2	0.7	0.9	1.7	2.2	3.3	5.2



a) b)

Fig. 2. Variation of (a) Penetration and (b) Softening Point as a Function of EVA Percentage.



a) b) c) d)

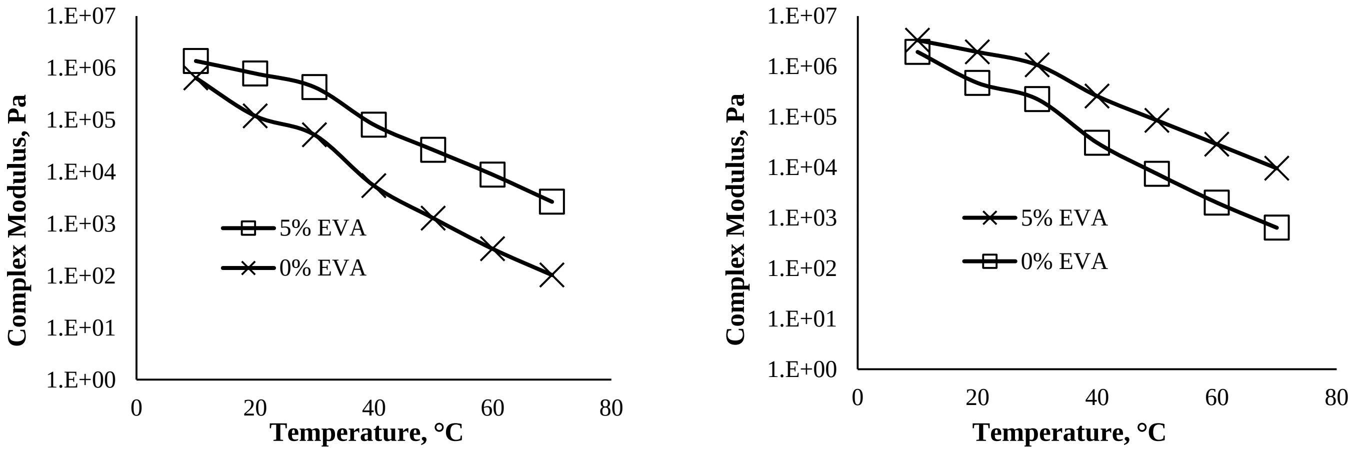
Fig. 3. Morphology of EVA PMB Obtained from Fluorescence Microscopy for a) 0% EVA, b) 3% EVA, c) 5% EVA and d) 7% EVA.

7% are shown in Fig. 3(a)-3(d). The morphology of the modified bitumen changes as the polymer content increases. At 0% EVA there is no fluorescence effect and the image appears to be single dark color. 3% modification with EVA has a bitumen rich phase with polymer being dispersed in it. The image for PMB 5% shows an interlocked phase and the starting point from where polymer phase starts dominating. EVA 7% clearly shows a polymer rich phase with large amount of fluorescence effect caused by the swelled polymer. This confirms the result of separation test in which 5% EVA was found to be optimum for modifying the base bitumen used in the study.

Rheological Evaluation

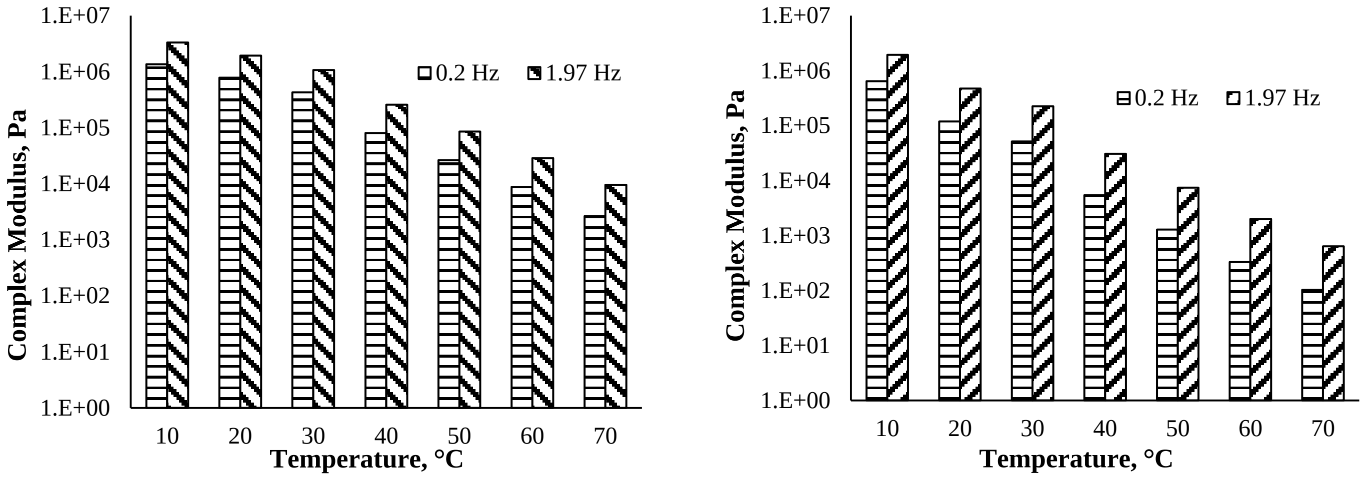
Rheological Viscoelastic Properties

Fig. 4(a)-4(b) shows the variation of complex modulus G* with temperature for the two frequencies adopted in the study. G* increases as a result of EVA modification indicating stiffening effect produced polymer network. The G* decreases for both PMB and VG 10 as the temperature increases, but the decrease for PMB is less than that in the base bitumen. This is attributable to the improvement in temperature susceptibility after modification. In



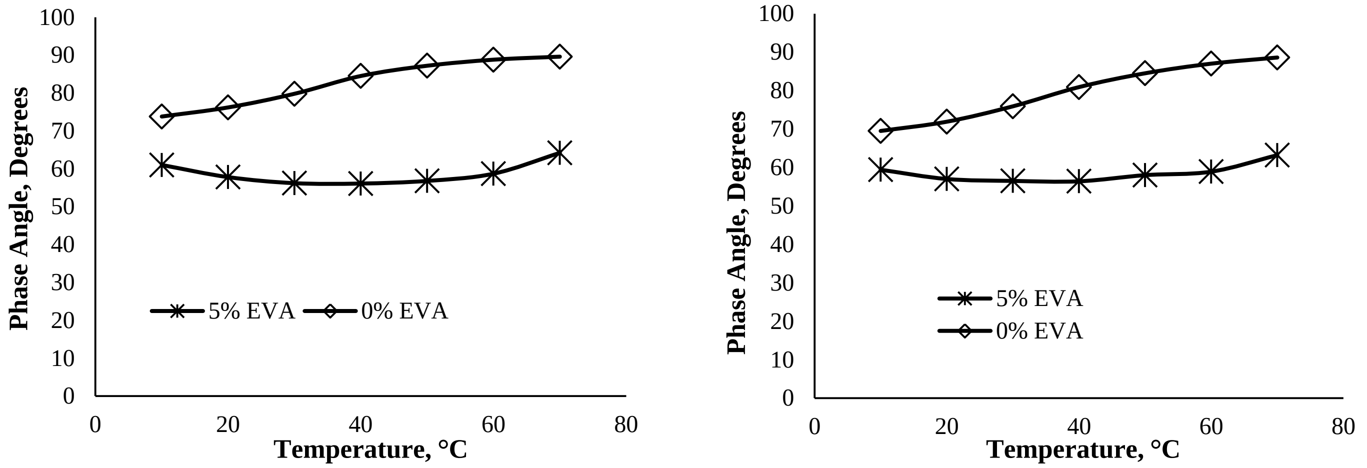
a) b)

Fig. 4. Variation of complex modulus G* with temperature for a) 0.2 Hz, b) 1.97 Hz



a) b)

Fig. 5. Variation of Complex Modulus G* with Temperature for a) 5% EVA, b) 0% EVA.



a) b)

Fig. 6. Variation of Phase Angle δ with Temperature for a) 0.2 Hz and b) 1.97 Hz.

EVA copolymers the polyethylene rich segments are arranged in crystalline form, which swells the EVA in the aromatic phase of the base bitumen leading to the formation of a rigid physical network. Fig. 5(a)-5(b) shows the change in G* for EVA and base bitumen with change in frequency. The modulus reduces as the speed (frequency) decreases. At places where lower speed (intersection) of vehicle is expected, the modulus of the pavement will be lower than in freeways (where the speed is high). It can be seen that change due to frequency is less significant in EVA PMB than in base bitumen.

Measurements of phase angle δ are generally considered to be more sensitive to the modification and chemical structure of bitumen than the other parameters like G*. Fig. 6(a)-6(b) shows the variation of phase angle, δ, with temperature for different frequencies. EVA PMB has lower phase angle for all temperatures than VG-10. Hence modification leads to improvement in elastic behavior of the base bitumen and will resist permanent deformation which mainly occurs due to viscous flow. Phase angle for higher frequency is lower than that seen for lower frequency condition. But at higher temperatures the phase angle of base bitumen approaches almost 90˚, indicating a shift towards pure viscous behavior. At these temperatures the value of δ after modification remains well below 70˚. So at regions with higher temperature and places where low traffic speed is expected, EVA modification might produce excellent results as far as permanent deformation is concerned. At regions with lower temperature and high speed facilities base bitumen might give sufficient performance. It can also be seen that after 60˚C there is a sudden increase in δ for EVA PMB. This may be attributable to the melt of EVA copolymer as the temperature reaches 60˚C, shifting the behavior of the modified binder towards the base binder [2].

Conclusions

The following conclusions were drawn from the study conducted

1. It is important to obtain the proper blending requirement for modifying bitumen with any additive. Temperature and blending time are the most crucial parameters for obtaining a homogenous blend. The influence of shear rate increases as the percent modifier increases.
2. A temperature of 180˚C, blending time of 60 minutes and a minimum shear rate of 600 rpm is required for EVA modification.

3. 5% EVA provided an interlocked phase morphology which led to significant improvement in the properties of bitumen. Increasing the modifier content will make the binder a polymer rich phase which might lead to phase separation at higher temperature.
4. EVA modification lead to an increase in viscoelastic response of the binder by increasing the G* of the base bitumen. Also the change in the modulus with change in temperature was more stable for EVA PMB than for base bitumen. So it can be concluded that the temperature susceptibility of the base bitumen is significantly improved due to polymer modification.
5. EVA modification significantly improves the elastic response of the base bitumen making it more rut resistant.

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