



Research Article

# Investigation of physical properties of base and SBS modified bitumens by rheological test methods

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**Abstract:** Bitumen is modified with various modifiers to diminish the deformation occurred in flexible pavements due to traffic loads and the effects of climate. Polymer modification and more specifically Styrene-Butadiene-Styrene (SBS) copolymer modification is one of the most common methods to enhance the physical properties of bitumen. However, the polymer modified bitumens could exhibit different rheological properties compared to original bitumen. In this work, it is aimed to investigate the effects of SBS copolymer on thermorheological properties of bitumen by means of state of art test methods. To this end, a rheological program including small amplitude oscillation shear test (SAOS), construction of master curves by using time-temperature superposition (TTS) principle, determination of zero shear viscosity (ZSV) and multiple shear creep recovery tests (MSCR) were employed along with other fundamental tests. SAOS test result signifies a positive effect of SBS on the viscoelastic deformation nature of bitumen. The master curves of the complex viscosity of binders reveal that SBS modifier reduced the Newtonian flow properties of bitumen. The decrements in non-recoverable creep compliance and the increment in percent recovery signify that SBS modifier has dramatically enhanced the applicability of bitumen as a binder in flexible pavement at mid to high temperature ranges.

**Keywords:** Rheology, bitumen, polymer modification, viscoelasticity, master curves.

## 1. Introduction

Bitumen, employed as a binder in flexible pavements, is a thermoplastic and viscoelastic material. The stiffness of a thermoplastic material is quite susceptible to the changes in temperature. (Dobson, Monismith, Puzinauskas, & Busching, 1969; McNally, 2011). Thus, bitumen is in glassy elastic form at low temperatures or in other words in cold regions, whereas at elevated temperatures, due to loss of stiffness, it behaves as a highly viscous fluid (Habib, Kamaruddin, Napiyah, & Isa, 2011). However, at mid-range temperatures, bitumen exhibits the combination of both two deformation behaviors which is called viscoelasticity. Viscoelasticity represents a type of deformation model that includes dependency of loading time and elastic recovery of material when applied load removes (Roman & Garcíea-Morales, 2018; Y. Zhang & Gao, 2021). Hence, it is quite safe to say that the thermoplastic and viscoelastic nature of bituminous binders determine the deformation properties of the material, and eventually these binder properties are responsible for some certain types of failure occurred in highway pavements. Recently, the traffic level and numbers of heavy vehicle in a traffic composition have gradually been increasing all over the world which means additional stress to the structure of flexible pavements (Kaya, Topal, Kacmaz & Sengoz, 2020). Sustainability of highways remains a concern as it has a huge impact on economy (Vega-Zamanillo, Calzada-Pérez, Lastra-González, Indacochea-Vega, & Fernández-Ortega, 2017). For this reason, most of bituminous binders are modified with various modifiers to limit the permanent deformation. Polymers based materials are generally satisfactorily used as

modifiers in bitumen, and Styrene-butadiene-styrene (SBS) copolymer is one of the most employed polymer modifiers in the world (Airey, 2003; Sengoz & Isikyakar, 2008). The accumulation of permanent deformation over the service life of a flexible pavement can be limited by employing SBS in bitumen which enhances the physical properties of the material. SBS modification has been studied by researchers over the years as the modifier is frequently used in highway constructions (Airey, 2003; Sengoz & Isikyakar, 2008; Wu, Pang, Mo, Chen, & Zhu, 2009).

However, polymer modification can greatly affect some fundamental rheological properties of bitumen, and there have been limited research conducted on thermorheology of SBS modified bitumens by means of employing state of art test methods and analysis. To this end, laboratory research was conducted on SBS modified binders to determine the effects of SBS copolymer on bitumen's thermoplasticity, viscoelasticity, Newtonian flow behavior, zero shear viscosity, activation energy and so on. By examination of these rheological properties of SBS modifier binder, it is possible to better understand the mechanism of deformation behavior of SBS modified bitumen which also help the optimum and proper usage of SBS modifier in bitumen to diminish pavement distresses.

Within this context, first, modified bituminous binders having different amounts of SBS copolymer (3.5%, 4.0%, 4.5%, 5.0% by total weight of the mixture) were prepared by means of high shear mixer. Various conventional test methods such as penetration, softening point, ductility was applied to base (non-modified) and SBS modified binders to examine the physical changes after the modification. The viscosity of the binders was measured with rotational viscometer at 135 °C and 165 °C. The ideal mixing and compacting temperatures of binders were calculated by the mathematical equations of log-viscosity curves. The test program was continued with the various rheological approaches. Amplitude sweep test was conducted to determine viscoelastic limits (LVE) of base and SBS modified binder to understand the elastic region where the deformation is totally recovered. LVE value needs to be known for small amplitude oscillatory shear (SAOS) test which is a non-destructive method. SAOS tests were conducted for the evaluation of complex modulus ( $*G$ ), phase angle ( $\delta$ ) parameters. By applying time-temperature superposition (TTS) principle the master curves of complex viscosity ( $\eta^*$ ), storage moduli ( $G'$ ) and loss moduli ( $G''$ ) that belong to the binders were constructed.

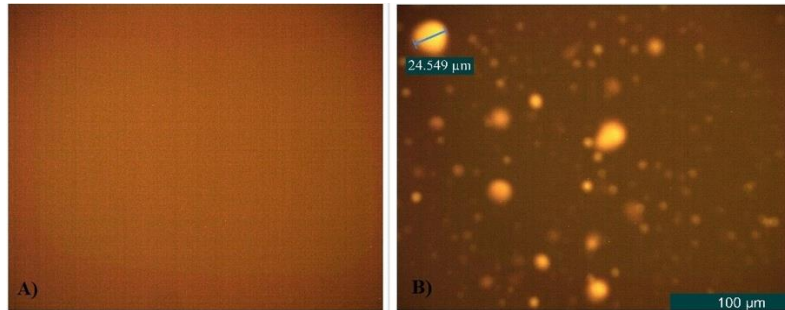
The shift factors, employed in master curves and calculated by means of experimental data were compared with the fitting curves of Arrhenius and WLF equations. Zero shear viscosity (ZSV) parameter of base and SBS modified binders were calculated by means of four-parameter cross model, and the findings were compared with master curves outcomes. Finally, multiple shear creep-recovery test which is an up-to-date method as an alternative to conventional Superpave method, was applied to the binders to examine elastic recovery nature of SBS modified bitumens.

## 2. Materials and methods

Bitumen, obtained from the Tüpraş Aliğa refinery, having a penetration of B160/220 was employed as a binder. The bitumens classified as B160/220 are relatively soft binders compared to usually employed bitumens, however this enables applying of heavier modification levels without experiencing any excessive hardening of binder. Kraton D1101 block SBS copolymer was used as a co-polymer additive in this research. The modification process was done by means of Silverson L5M high shear mixer (Dong et al., 2014). At the beginning of the modification process, the neat bitumen was first heated up to 170 °C, then poured down to a temperature-controlled flask. The total amount of SBS copolymer was added to bitumen at intervals where the total process took 15 minutes.

During the addition of the SBS modifier, the mixing rate was fixed at 800 rpm. Once all the modifier was poured into bitumen, the high shear mixer was adjusted to 3000 rpm in order to let SBS particles be milled over, melt and blend into bitumen. This process took another 60 minutes. End of the modification, the samples having 3.5%, 4.0%, 4.5%, 5.0% of SBS were prepared for the testing program. The samples were kept in aluminum containers (resistant to high temperature) and stored. Short-term aged binders were also prepared to employ in some rheological tests which was done accordance with ASTM D2872 (ASTM D2872, 2012).

Fluorescent microscope, Leica DM750, was employed to visualize the dispersion of SBS polymer particles into the bitumen (Kaya et al., 2019). The images, that belong to base Fig. 1.a and heavy SBS modified bitumen (5.0%) Fig. 1.b, were acquired with an objective having a magnification of 40x. The fluorescent microscope image given in Fig. 1.b shows dispersion of the polymer particles into bitumen which are generally less than 25  $\mu\text{m}$  indicating that pallet-shaped SBS particles were milled into smaller pieces by means of high shear mixer.



**Figure 1.** Fluorescent microscope images of base and SBS modified binders.

### 2.1. Conventional test methods

Conventional test methods namely penetration tests and softening point tests were conducted accordance with their standards (ASTM D5, 2013; ASTM D36, 2006). Penetration index, a parameter indicates the temperature susceptibility of the materials were calculated by means of penetration and softening point values that belongs to base and SBS modified binders using Eq.1.

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

where; PI: penetration index, SP: softening point,  $Pen_{25}$ : the penetration at 25 °C (Read & Whiteoak, 2003).

### 2.2 Rotational viscosity test

The viscosity of base and SBS modified binders were determined by Brookfield viscometer detailed in ASTM D4402 (ASTM D4402, 2015). The ideal compacting and mixing temperatures of binders (the processes of mixing with aggregates and compacting the hot mix) can also be calculated by the exponential equation of viscosity vs temperature curves. To this end, two different RV tests were conducted on the base and modified binders at 135 and 165 °C, and the viscosity values obtained from these tests were marked in logarithmic viscosity temperature curves.

### 2.3 Small amplitude oscillation shear test

Small amplitude oscillation sweep (SAOS) test is a non-destructive test method that carried out within viscoelastic limit (LVE) of bitumen. 1.2% of deformation rate was selected for the tests. SAOS tests are done by means of dynamic shear rheometer under deformation-controlled mode. The SAOS tests in this study were conducted at a range of 0.01-100 1/s. The tests temperatures were selected as 30-70 °C at 10 °C increments where bitumen exhibit mostly viscoelastic behavior. 25 mm parallel plate and 1 mm gap between the plates were used as the geometry of the test (Laukkanen & Winter, 2018).

### 2.4 Construction of master curve by TTS

Isotherm curves obtained from SAOS test can be shifted to a reference temperature by applying time-temperature superposition (TTS) principle. Master curves are theoretical plots where the temperature dependence of mechanical properties of a viscoelastic material can be examined (Chan, Shyu, & Isayev, 1995). The master curves of binders were plotted at 30 °C by using TTS. Complex viscosity ( $\eta^*$ , ( $\omega$ )), storage moduli ( $G'$ ) and loss moduli ( $G''$ ) were the rheological parameters that were included in the master curves of the binders. The test was done between 10-50 °C by an increment of 10 °C, the strain was

fixed at 0.8 %, the frequency range was 0.01-100 1/s, the geometry of the spindle was 8 mm parallel plate and the gap between plates was 1 mm. The shift factors are used for shifting isotherm plots from a measured test temperature to reference temperature. In this study, the calculated shift factors were also compared with the shift factors obtained by means of employing Arrhenius (Eq.2) and Williams–Landel–Ferry (Eq.3) equations. By using Arrhenius equation model, the activation energy of Base and SBS modified bitumens were calculated (Lytton et al., 1993; Williams, Landel, & Ferry, 1955).

$$\log a_t = C \left( \frac{1}{T} - \frac{1}{T_{ref}} \right) = \log e \frac{\Delta H}{R} = \left( \frac{1}{T} - \frac{1}{T_{ref}} \right) \quad (2)$$

$\Delta H$ : activation energy (J/mol), R: Universal gas constant which is 8.314 J/(mol.k). T: test temperature,  $T_{ref}$ : reference temperature for master curve,  $a_t$ : shift factor.

$$\log \frac{\eta_{0,T}}{\eta_{0,T_{ref}}} = \log a_t = \frac{c_1(T-T_{ref})}{c_2+(T-T_{ref})} \quad (3)$$

$\eta_{0,T_{ref}}$ , the viscosity at the reference temperature,  $\eta_{0,T}$  the viscosity at the test temperature,  $C_1$ ,  $C_2$  constants for bituminous binders.

### 2.5 Determination of zero shear viscosity (ZSV)

ZSV has been seen as an alternative to rutting parameter for prediction of permanent deformation occurred due to traffic loads under high temperature conditions. Although ZSV could be a good predictor for rutting, it is also reported that it is not easy to achieve to reach the conditions that needs to determinate ZSV of polymer modified bitumens (Zhang, Zou, & Xu, 2009). Four-parameter cross model was used for determination of ZSV in this study, Eq.4 belong to this model was given below (D. A. Anderson, Le Hir, Planche, Martin, & Shenoy, 2002):

$$\eta^*(\omega) = \frac{\eta_0^* - \eta_\infty^*}{1 + (K\omega)^m} + \eta_\infty^* \quad (4)$$

where,  $\eta^*(\omega)$ ; viscosity at angular frequency,  $\eta_0^*$ ; zero shear viscosity,  $\eta_\infty^*$ ; infinite viscosity, K and m are material constants.

### 2.6 Multiple shear creep recovery test

Multiple shear creep recovery (MSCR) test has been developed to elaborately investigate the deformation behavior of a binder including elastic recovery ability when applied load removes (M. Anderson, D'Angelo, & Walker, 2010). The procedure of the test was detailed in AASHTO TP70. According to the standard, two different rates of shear stress are used during the test. 10 cycles of 0.1 kPa of shear stress is first applied to the RTFO aged binder, and then another 10 cycles applied at 3.2 kPa. Every cycle consists of 10 seconds of loading-resting phase where corresponding load applied for 1 second and 9 seconds are for the rest (the recovery period) (Yang & You, 2015). In this study, 58 and 64 °C are selected as test temperatures. AASHTO M332 prescribes use an equation (Eq. 5) whether the polymer modification is adequality done at corresponding test temperature by employing non-recoverable creep compliance ( $J_{nr}$ ) and percent recovery (%R) values (AASHTO, 2010);

$$\%R = 29.37 * J_{nr}^{-0.26} \quad (5)$$

### 3. Results and discussions

#### 3.1 Conventional test findings

The results obtained from conventional tests namely penetration, softening point and ductility were given in Table 2. The amount of penetration is in decreasing trend that is accompanied by an increment in softening point as SBS copolymer content in bitumen increases. This result is a clear indication of increased stiffness of bitumen after the modification. In addition, the stiffening effect of SBS on bitumen becomes more distinct at heavy modification levels. According to this, base bitumen has a penetration of 185 dmm, whereas at low modification level (B-SBS3.5) penetration decreases to 133 dmm, and finally, it is measured as low as 69 dmm for B-SBS5.0 (at heaviest modification level).

Gain in stiffness of bitumen after SBS modification is an expected result as reported from previous works. (Galooyak, Dabir, Nazarbeygi, & Moeini, 2010). Ductility, elongation ability of binder without breaking, increases with increasing polymer content that emphasize an enhancement in elastic response of a viscoelastic material. Penetration indexes (PI) of binders increases with increasing modifier content. PI is an indicator on temperature susceptibility of bitumen. Lower value of PI indicates bitumen is more vulnerable against the temperature changes. This means that the temperature susceptibility of bitumen is clearly reduced after the modification, as SBS modified bitumens have relatively higher PI compared to that of base binder.

**Table 1.** Conventional test results.

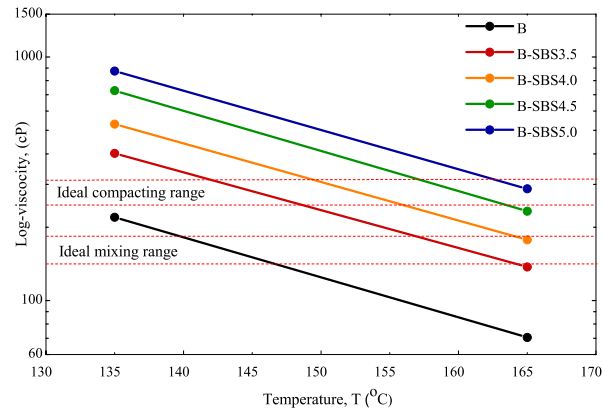
Properties	Binder type				
	B	B-SBS3.5	B-SBS4.0	B-SBS4.5	B-SBS5.0
Penetration (dmm)	185	133	118	91	69
Softening point (°C)	37.5	42.5	47.0	50.5	53.5
Ductility (cm)	102	107	113	118	119
Penetration index (PI)	-1.55	-0.35	0.35	0.52	0.46

#### 3.2 Rotational viscosity of base and SBS modified bitumens

The results of rotational viscosity tests conducted on Base and SBS modified bitumens were given in Table 2 and Fig 2. The viscosity has dramatically increased after SBS modification, as base bitumen has a viscosity of 465.5 cP at 135 °C, whilst viscosity of B-SBS3.5 was determined as 827.5 cP. Even though this result suggests that polymer modified bitumen has become more resistant against shear stress compared to base at the same temperature, bitumen should also be in fluid form during mixing and compacting processes. The calculated ideal compacting and mixture temperatures are listed in Table 2 (Zaniewski & Pumphrey, 2004). The modification indexes, calculated by the viscosity of modified binder divided by the viscosity of original binder ( $\eta_{\text{modified}} / \eta_{\text{base}}$ ), are used for evaluating the changes in viscosity after polymer modification. The modification indexes of B-SBS5.0 are 3.98 at 135 °C and 4.11 at 165 °C that means the viscosity has risen roughly four times after SBS modification. Hence, at heavy SBS modification level, bitumen can become excessively hard which might end up with some compacting and mixing problems where binder needs to be adequately fluid.

**Table 2.** Rotational viscometer test results.

Binder type	RV (cP)		$\eta_{\text{modified}} / \eta_{\text{base}}$		Compaction temp. (°C)	Mixing temp. (°C)
	135°C	165°C	135°C	165°C		
B	220.0	70.0	1.00	1.00	126-132	139-145
B-SBS3.5	402.0	137.5	1.83	1.96	141-147	155-161
B-SBS4.0	530.0	177.5	2.41	2.54	152-158	165-172
B-SBS4.5	722.5	235.5	3.28	3.36	157-163	171-178
B-SBS5.0	875.0	287.5	3.98	4.11	163-169	177-183



**Figure 2.** Rotational viscosity curves of base and SBS modified binders.

### 3.3 Small amplitude oscillation shear test results

The findings of small amplitude oscillation sweep (SAOS) test are presented in Fig 3. The complex modulus ( $G^*$ ) parameter of modified bitumens have distinctly higher values compared to base bitumen. This result signifies that the bitumen gains resistance against shear stress at mid to high temperature range, which represents traffic loads applied to flexible pavement. In addition, the enhancement in  $G^*$  values after the modification become more evident at lower frequencies that corresponds to slow traffic speeds.

The isotherm curves of  $G^*$  values are quite proportional for base bitumen at overall range of frequencies (Fig.3.a), whereas the change in temperature significantly affects the trend of  $G^*$  vs frequency curves of SBS modified bitumens. (Fig.3.b-c-d). The difference between the trends can be explained by the fact that in mid to high temperature range, base bitumen has lost most of the storage modulus component, and thus viscous flow behavior is clearly dominant.

The phase angle ( $\delta$ ) indicates the elastic/viscous deformation components of a viscoelastic material.  $\delta$  of base bitumen almost reaches to  $90^\circ$  over  $60^\circ\text{C}$  and at a frequency range of 0.1 to 0.01 1/s, represents the complete viscous behavior Fig.4.a, which also confirms the  $G^*$  findings. SBS copolymer modified bitumens have considerably lower  $\delta$  value than that of base bitumen that emphasizes the viscoelastic response of the material (Wu, Pang, Mo, Chen, & Zhu, 2009). The positive effect of SBS polymer on temperature susceptibility of bitumen can be confirmed from  $\delta$  values, as the modified binders (especially B-SBS4.5 and B-SBS5.0 Fig.4.c-d) are in semi-solid form at the temperatures below  $50^\circ\text{C}$  where  $\delta$  values range between  $50$  to  $60^\circ\text{C}$ . SAOS tests showed that SBS copolymer aside from providing increased stiffness, it also enhances the elastic response of the material especially under longer loading times.



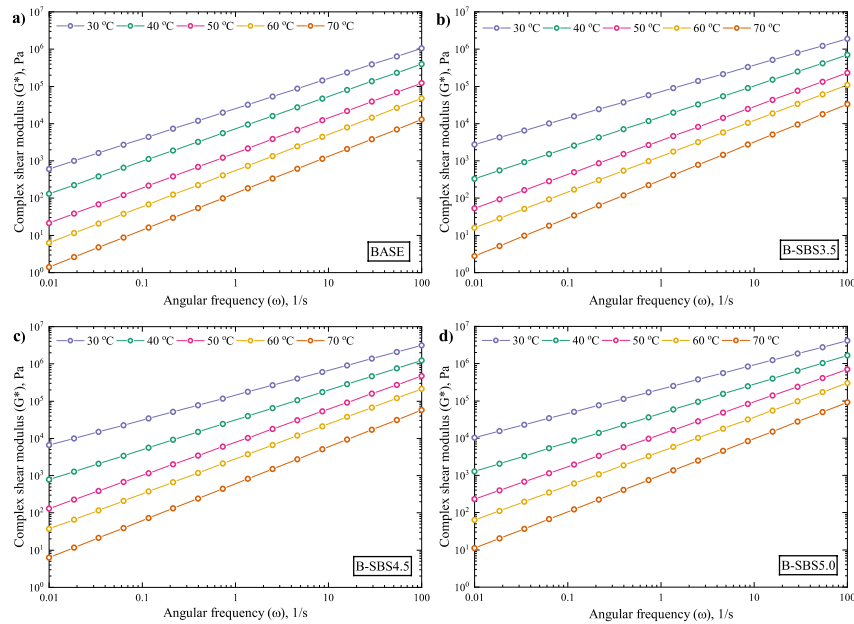


Figure 3.  $G^*$  vs. angular frequency curves of (a) base; (b) B-SBS3.5; (c) B-SBS4.5; (d) B-SBS5.0.

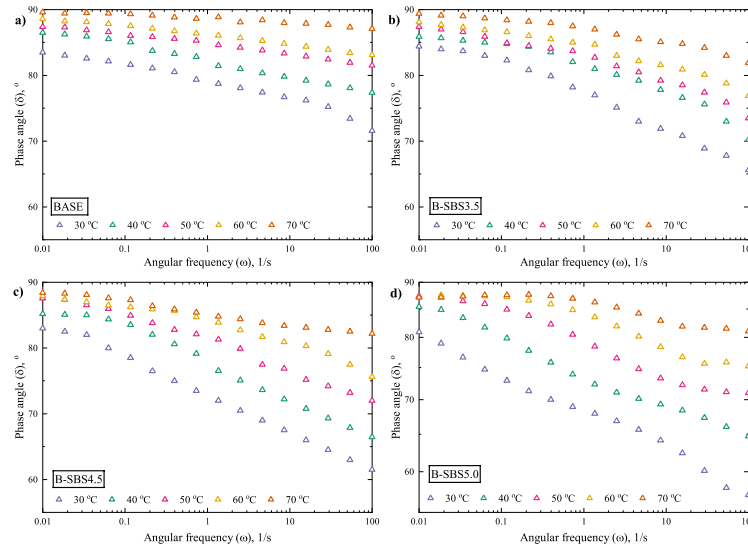
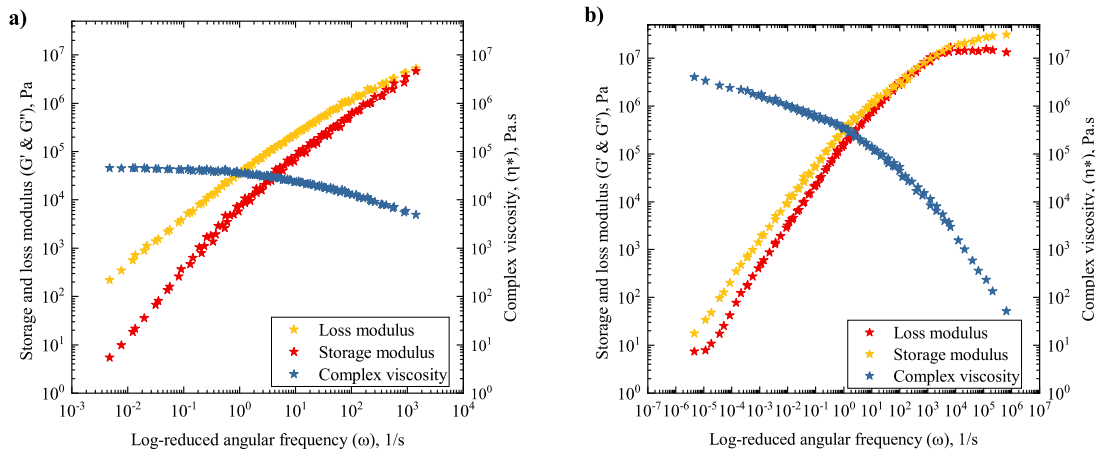


Figure 4.  $\delta$  vs. angular frequency curves of (a) base; (b) B-SBS3.5; (c) B-SBS4.5; (d) B-SBS5.0.

### 3.4 Master curve findings

The master curves of binders were constructed by employing the loss and storage moduli of binders ranging at 0.01-100 1/s and 10-50 °C. The complex viscosity, storage and loss modulus parameters of Base and B-SBS5.0 at 30 °C are included in the master curves presented in Fig. 5. The complex viscosity ( $\eta^*$ ) curve of base bitumen, shown in Fig. 5.a, has a plateau-like trend between low to mid frequency range which is accompanied by a slight decrease at higher frequencies. Previous works suggest that Cox-Merz transformation, an empirical rule, can be applied to bituminous binders which determines the interchangeability between steady flow viscosity-shear rate curve and complex viscosity-angular frequency curve (Gunay, Tomkovic, & Hatzikiriakos, 2020; Saboo & Kumar, 2016). Hence, a plateau along with extended range of frequency implicate Newtonian behavior of a viscoelastic material. In this frequency range, the applied shear stress and resultant deformation are

quite proportional. On the other hand, a plateau region was not observed in the  $\eta^*$  curve that belongs to SBS modified bitumen, although the slope of the curve slightly decreases at lower frequencies, especially below  $10^1$  1/s. This finding shows that SBS copolymer has an impact on Newtonian behavior of bitumen, since non-Newtonian behavior is clearly dominant after the modification (Saboo & Kumar, 2016; Wang, Liu, Apostolidis, & Scarpas, 2018).



**Figure 5.** The master curves of  $G'$ ,  $G''$  and  $\eta^*$  vs angular frequency ( $\omega$ ) for (a) base; (b) SBS modified bitumen.

SBS modified bitumen have higher  $\eta^*$  values throughout frequencies compared to that of base bitumen which confirms rotational viscosity findings. At very low frequencies,  $\eta^*$  curve belonging to SBS modified bitumen (Fig.5.b) is in a continuously increment trend which possibly signifies difficulty of the determination of zero shear viscosity for heavily SBS modified bitumens, as steady complex viscosity phase was not attainable in the master curve, even at very low frequencies (Behzadfar & Hatzikiriakos, 2013; Gunay et al., 2020). The crossover frequency of SBS modified bitumen, where storage and loss moduli curves intersect, was observed in a frequency range of  $10^1$ - $10^3$  1/s for SBS modified bitumen (Fig. 5.b), whereas crossover frequency of base bitumen at 30 °C should theoretically be at higher frequencies (over  $10^3$  1/s). The material is in gel-like structure at the frequencies above the crossover, whilst the frequencies under the crossover, where  $G'' > G'$ , the binder shows fluid structure or viscoelastic fluid properties (Loeber, Muller, Morel, & Sutton, 1998). As known, storage moduli represent the elastic response of a viscoelastic material, whereas loss moduli correspond to dissipated energy of resultant deformation that arises from applied load. At very low frequencies, the decreasing trend of storage moduli of SBS modified bitumen become flattened (Fig. 5.b), as the storage moduli maintains a minimum constant value throughout the lower frequencies which signifies the presence of solid particles in bitumen (Behzadfar & Hatzikiriakos, 2013).

The shift factors obtained from experimental test as well as calculated by means of WLF and Arrhenius equations were presented in Fig. 6. The shift factors, belong to Base and SBS modified bitumens, obtained from experimental test are quite compatible with the calculated shift factors by means of WLF and Arrhenius equations. The activation energy of Base and SBS modified bitumens was calculated using Arrhenius equation and given in Fig. 6.a. A higher activation energy means a higher minimum energy that need to initialize a chemical reaction (Mezger, 2011).

The activation energy of SBS modified bitumens are higher compared to that of the base bitumen (Fig. 6.a). Lower activation energy of a viscoelastic material signifies reduced sensibility to temperature (Garcia-Morales et al., 2004). This result can be explained with the crosslinking properties of SBS modification. An increment in activation energy for stiffer bitumens is also reported some other works (Cao et al., 2019).

$C_1$  and  $C_2$  material constants calculated by WLF equations are compatible with that of the previous works (Chailleux et al., 2006; Y. Yang et al., 2018).



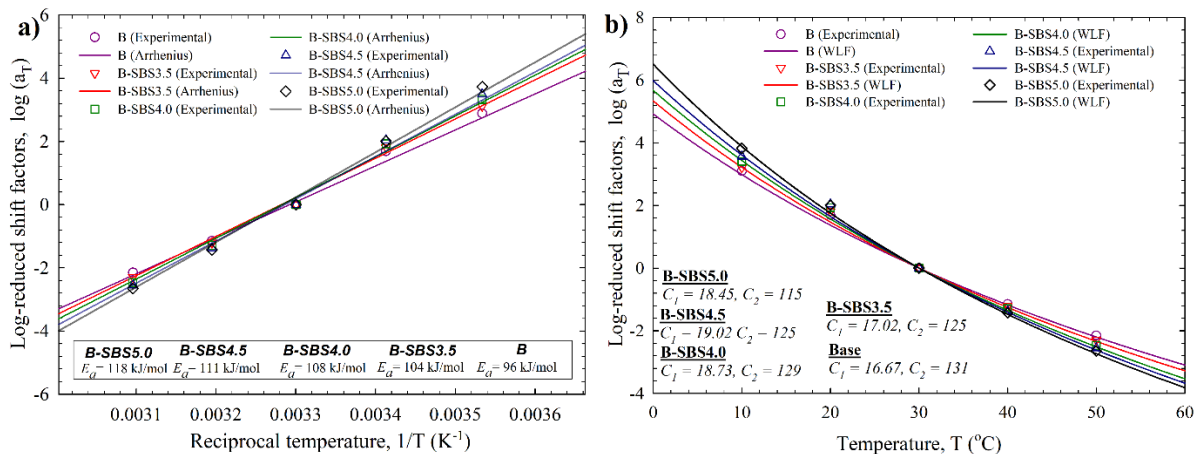


Figure 6. Comparison of log-reduced shift factors with Arrhenius (a) and WLF equation, and (b) experimental data.

### 3.5 Zero shear viscosity of binders

Zero shear viscosity (ZSV) of base and SBS modified bitumens were given in Table 3. ZSV values have dramatically increased after SBS modification, as B-SBS3.5 has almost 10 times higher ZSV than that of base bitumen at 10 °C.

Table 3. Zero shear viscosity values of base and SBS modified bitumens.

Binder type	ZSV (Pa.s) at 10°C	ZSV (Pa.s) at 30°C	ZSV (Pa.s) at 50°C
B	6.67x10 <sup>6</sup>	6.30x10 <sup>4</sup>	9.76x10 <sup>2</sup>
B-SBS3.5	6.28x10 <sup>7</sup>	1.08x10 <sup>6</sup>	9.72x10 <sup>4</sup>
B-SBS4.0	9.21x10 <sup>7</sup>	1.68x10 <sup>6</sup>	1.88x10 <sup>5</sup>
B-SBS4.5	1.48x10 <sup>8</sup>	2.77x10 <sup>6</sup>	3.25x10 <sup>5</sup>
B-SBS5.0	2.41x10 <sup>8</sup>	4.38x10 <sup>6</sup>	6.45x10 <sup>5</sup>

At elevated temperatures, the differences between ZSV of Base or B-SBS3.5 is even more dramatic, as base bitumen loses its storage modulus indicating viscous flow exhibits. This excessive increment in ZSV after the modification could arise from presence of solid particles floating in polymer-bitumen matrix. Besides, at elevated temperatures (mid- to high temperature region) SBS modified bitumens might still be in semi-solid form where binder shows combination of elastic solid and viscous fluid deformation behaviors. Higher ZSV signifies less permanent deformation, as it is an indication of the rutting resistance of a flexible pavement (Jahanbakhsh, Karimi, & Nejad, 2020).

It is also reported that ZSV could be a convenient parameter only for original bitumens for the prediction of permanent deformation, as there have been some obstacles to reach an extremely low rate of frequencies where ZSV polymer modified can properly be calculated (Zhang et al., 2009). Heavily polymer modified bitumens hardly (or never) reaches to a steady viscosity values at a mid-range temperature where rutting occurred at most (Fig. 7). According to this, base bitumen has a flat-like viscosity curve (almost steady) throughout all-frequency range, whereas the complex viscosity curve of B-SBS5.0 has no plateau region, as it maintains a gradual increment in viscosity with decreasing angular frequencies. This dramatic change in ZSV due to an unattainable steady state after the modification might emphasize the unclarity of ZSV as a rutting predictor for heavily polymer modified bitumens. The difficulties in determining ZSV were also discussed in previous works (D. A. Anderson et al., 2002; Morea et al., 2010; Zhang et al., 2009).

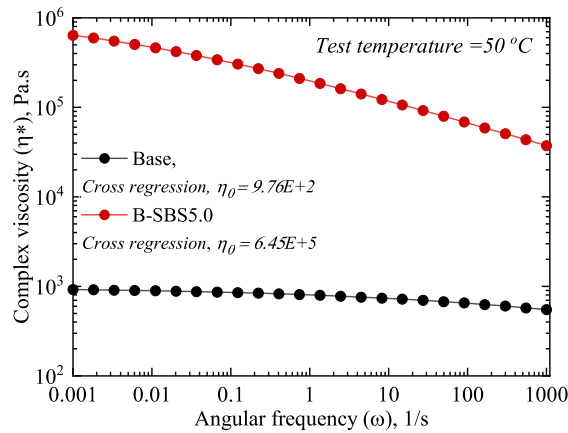


Figure 7. ZSV of base and SBS modified binder.

### 3.6 Multiple shear creep recovery test results

Multiple shear creep recovery (MSCR) tests were done on short-aged base and SBS modified binders at 58 °C and 64 °C, and the results were given in Fig. 8 and Fig. 9.  $j_{nr}$  parameter, represents non-recoverable part of the viscoelastic deformation, has dramatically been decreased after SBS modification (from Fig. 8). This result is a clear indication of enhanced elastic response of bitumen with SBS modification (Król, Radziszewski, & Kowalski, 2015; Saboo, Kumar, Kumar, & Gupta, 2018). As known, MSCR tests are conducted at two different load rates of 0.1 kPa and 3.2 kPa. Altering loading rates help to simulate in real life conditions (the applied load from the existing traffic), and thus the amount of the permanent deformation of a flexible pavement can be predicted with higher accuracy. The positive effect of SBS can also be observed within this sense, as there is a significant increment in  $j_{nr}$  values of base bitumen at 3.2 kPa compared to that of 0.1 kPa, whereas the change in  $j_{nr}$  values at different stress rates are quite proportional for SBS modified bitumens.

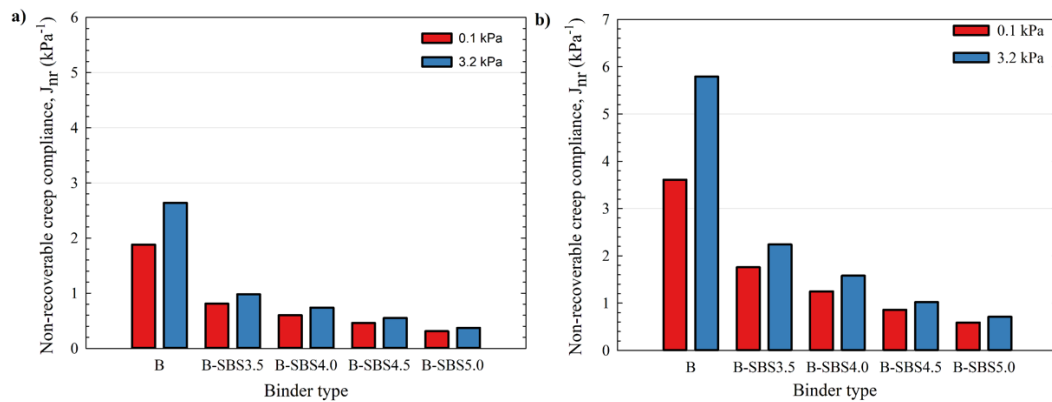


Figure 8.  $j_{nr}$  value of base and SBS modified bitumens at (a) 58 °C, and (b) 64 °C.

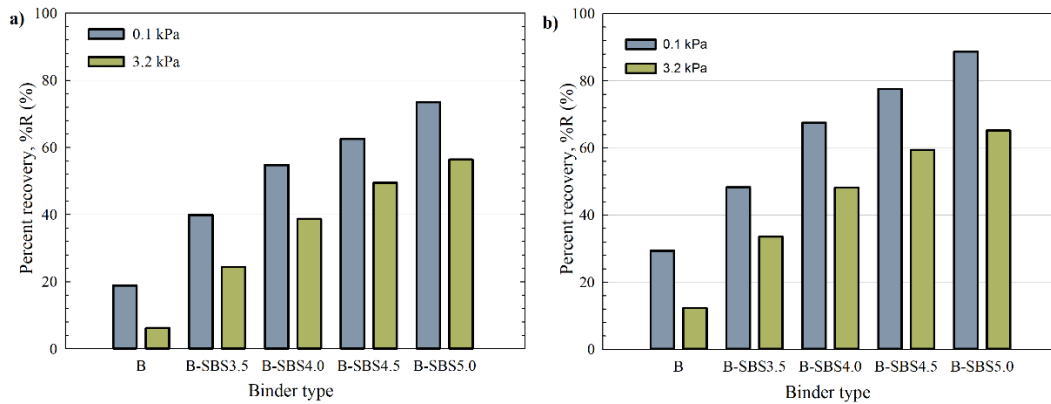


Figure 9. %R value of base and SBS modified bitumens at (a) 58 °C, and (b) 64 °C.

Percent recovery parameters (%R) of binders have higher values as the amount of the modifier increase (Fig. 9). According to this, at 58 °C and 3.2 kPa load rate SBS modified binder has almost 2.5 times higher %R compared to base bitumen (Fig. 9.a). AASHTO (AASHTO M332) proposed an equation which is  $\%R = 29.37 * J_{nr}^{-0.26}$  in order to evaluate whether the binder is sufficiently polymer modified. The marker that belongs to B-SBS3.5 is above the line along with the other markers of the binders containing higher amount of SBS. Therefore, 3.5% of SBS provides sufficient elasticity to the binder at the temperature below 58 °C. However, as the value of  $j_{nr}$  (for B-SBS3.5) exceeds to maximum level of 2.0 kPa<sup>-1</sup> at 64 °C, the modification level of SBS should be at least 4.0% (Fig. 10). Nevertheless, the stiffness or penetration of original binder also effects the optimum usage of polymer.

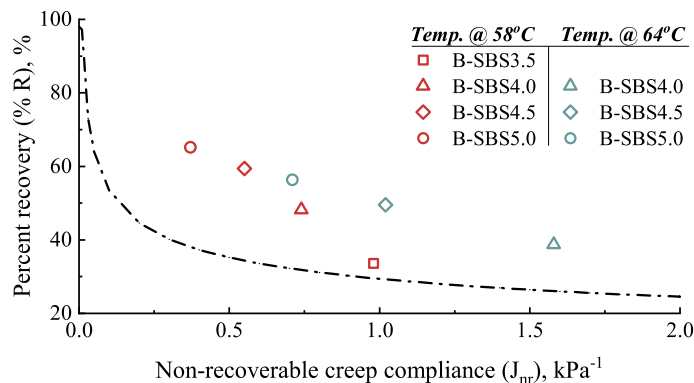


Figure 10. %R vs.  $j_{nr}$  curves of base and SBS modified bitumens at 58 °C and 64 °C.

#### 4. Conclusions

The findings of physical and thermorheological tests conducted on the Base and SBS copolymer modified binders can be summarized as follows: The conventional tests confirms that SBS copolymer modification results in an enhancement in the stiffness of binder which is also observed in rotational viscosity test. However, at a high modification level (5% of SBS), the ideal mixing and compacting temperatures excessively increases, which might affect some other circumstances such as the cost effectiveness, energy consumption and environmental impact of the material. SAOS test result signifies the positive effect of SBS on permanent deformation of binder. According to this, apart from physical hardening, SBS modifier also enhanced the elastic response of bitumen as phase angle values remarkably decreased after the modification. Moreover, this enhancement is more evident at relatively lower frequencies that equivalent to slow traffic speed where permanent deformation occurs at most.

The complex viscosity ( $\eta^*$ ), complex shear modulus values divided by frequency, is distinctly higher for SBS modified bitumen that compared to that of base bitumen, similar to the viscosity values of binder determined by RV. At very low frequencies,  $\eta^*$  is slightly in an increasing trend which emphasize that the zero shear viscosity of the polymer modified bitumen might not correctly be estimated.  $\eta^*$  of SBS modified bitumen has a gradual increment trend with decreasing frequency, and a clear plateau region was not observed in the master curve indicating non-Newtonian behavior of the material. On the other hand, storage moduli of SBS modified bitumen is almost flattened at very low level of frequency which signifies the presence of solid particles in the binder at 30 °C.

The excessive increment at ZSV after the SBS copolymer modification at mid temperature ranges might suggest the inconveniency of ZSV as predictor for rutting, as constant viscosity values could be attainable at very high levels of temperatures. This finding also confirms with the master curve of  $\eta^*$  that belongs to SBS modified bitumen. MSCR test results signify the importance of the elastic recovery ability of a binder to predict deformations in flexible pavement, as both  $J_{nr}$  and %R parameters are associated with elastic response of the material. The up-to-date methods and regulations such as MSCR test might also limit the usage of modifiers that only enhances the stiffness of bitumen, as contemporary methods are mainly associated with elastic nature of bitumen.

**Conflicts of interest:** The author would like to declare that the author do not have any potential conflict of interest regarding with this research.

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