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Research Article

Investigation of rheological properties of aged polymer modified binders

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Abstract

Bituminous pavement layers used in road flexible superstructure are the top layer that is directly exposed to the effects of traffic and the environment and therefore must be very robust in terms of mechanical properties. To enhance the resistance of pavements against traffic loads and permanent deformations, Styrene Butadiene-Styrene (SBS) polymer additive is generally preferred. The high demand for SBS often presents challenges in obtaining it when needed, leading to a consideration of alternative additives. In this study, three different polymers, namely 701, 611, and SBS, were compared to assess their effectiveness as modifiers for bituminous materials. Modified bitumen was prepared using SBS, 701 and 611 polymers prepared under specified conditions. The modified binders were subjected to long and short term ageing. For both pure and modified binders, the Superpave binder (dynamic shear rheometer) test was conducted. Consequently, these binders improved the performance of the pure binder when the Superpave binder test findings of the four distinct binders employed in the study were analyzed.

Keywords: SBS, Modified asphalt, Rheology, Aging, Elastomer

1. Introduction

PMA, or polymer-modified asphalt, is widely used in the building of pavements [1–4]. The polymers absorb the lighter components in the binder and swell after integrating with the basal asphalt binder. When compared to the pure binder, the modified binder's improved engineering qualities are mostly due to appropriately swollen polymers. These enhancements include increased fatigue life at transient temperatures, increased stiffness and elasticity at higher usage temperatures, and improved crack resistance at lower temperatures [3,5,6]. Styrene-butadiene-styrene (SBS) polymer is a preferred polymer for asphalt modification [7,8].

On the other hand, a variety of SBS polymers with different topologies are recognized. Monomers are joined to form polymers through chain or network architectures [9]. In SBS polymers, flexible polybutadiene (PB) blocks interact with stiff polystyrene (PS) blocks to form a two-phase system [10–12]. At lower temperatures, the rubbery PB blocks offer flexibility and resilience, while the PS blocks increase stiffness and tensile strength at higher temperatures. The method that PS and PB blocks are compounded is critical in deciding how SBS polymers behave because of the major variances in their properties. Three different SBS polymer structural configurations are shown in Figure 1, including two-block, linear three-block, and radial SBS with PB and PS blocks [13,14].

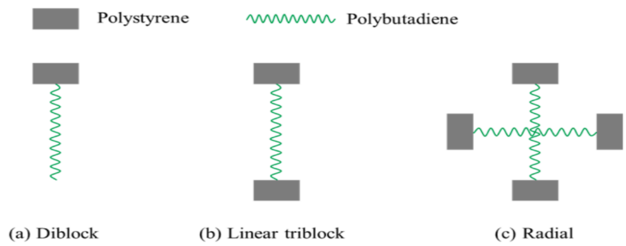

Figure 1: Examples of SBS polymers in various structural configurations [15]

Variations in the structures of polymers lead to differences in the attributes of SBS polymers, encompassing features like rigidity, flow resistance, and tensile resilience. These distinctions also extend to the properties of altered asphalt binders [11,16]. Studies indicate that radial SBS brings about notable enhancements in high-temperature functionality. In contrast, linear three-block SBS provides a well-rounded performance concerning PMA viscosity, elasticity, and compatibility. Meanwhile, two-block SBS leads to relatively reduced viscosity in polymer-modified asphalt (PMA) [17,18]. Analyses of the morphological properties in modified binders indicate that, in comparison to linear triblock and diblock SBS, radial SBS is generally associated with a less uniform dispersion within the asphalt phase [19,20]. Furthermore, an earlier study assessing the thermal degradation of SBS polymer in asphalt binder under high-temperature storage settings revealed that diblock SBS outperformed the other two kinds of SBS polymers in terms of thermal durability [21]. In spite of these discrete investigations, a comprehensive assessment of the impact of polymer structure on the properties of polymer-modified asphalt (PMA) is still lacking.

Because polymer content, material manufacture, and building techniques are involved, the effect of SBS polymers on the performance of polymer-modified asphalt (PMA) is further complex. While highly modified asphalt (HiMA) uses about 6-7.5% SBS polymer, regular PMA treatment typically uses about 3–4.5% SBS polymer [22,23]. The exact mechanism by which polymer interactions, content, and structure interact to influence PMA or HiMA performance is currently unknown. When creating and producing asphalt mixtures, SBS polymer might deteriorate due to heat and oxidation [24–26]. The unsaturated C-C bonds in polybutadiene (PB) blocks, in particular, are vulnerable to molecular-level breaking under thermal oxidative aging conditions. This can lead to the degradation or loss of modifying effects [21,27]. Research shows that when the polymer content of SBS increases, the particle size of SBS also increases dramatically. This results in the formation of network structures at 5-6% SBS, leading to improved elastic recovery. However, with aging, SBS polymer undergoes significant degradation, causing a reduction in size and diminished elastic rebound [15].

The predominant polymer additive in bitumen modification is the widely used SBS additive. However, the high demand for SBS additive often poses challenges in ensuring a timely and consistent supply. To address these issues, alternative polymer additives have been developed with the aim of reducing the reliance on SBS. This study focuses on the utilization of two distinct polymers, namely 611 and 701 polymers, in conjunction with SBS. The rheological properties of the modified bitumen, prepared by using these additives, are examined and compared both in short and long-term aging scenarios. All experiments were performed on aged binders. The aim of this study is to determine the effect of polymers on aging. The study aims to assess the rheological performances of the newly acquired elastomers in comparison to SBS after undergoing the aging process. This comprehensive analysis seeks to shed light on the impact of aging on the effectiveness of these polymer additives in bitumen modification.

2. Material and Methods

2.1. Raw materials

The unchanged binder employed in this study was B 50/70 grade bitumen, characterized by a density of 1.044 g/cm^3, and supplied by the TÜPRAŞ Batman refinery. In order to scrutinize the physical attributes of the pure binder, essential parameters such as penetration at 25°C, softening point, and viscosity at 135°C were meticulously examined. The results of these analyses are presented in Table 1.

Table 1: Bituminous binder properties

Property	Unit	Standard	Results
Penetration	mm^{-1}	EN 1426	62
Softening point	˚C	EN 1427	53.3
Penetration Index (PI)	-	-	0.119
Flash Point	˚C	EN ISO 2719	245
Density	g/cm^3	ASTM D70 - 18a	1.044
Solubility	%	EN 12592	100
Mixing temperature range	˚C	-	167.5–173.3
Compaction temperature range	˚C	-	155.2–160.6

Three different polymers were used in this study. Figure 1 illustrates the external appearance of the modifications. The 701 and 611 polymers were procured from Mpolimer Company, whereas the additive SBS (Kraton D 1101) was obtained from Shell Company. It is noteworthy that the unique products 701 and 611 polymers from Mpolimer Company have not been previously investigated. Table 2 presents an extensive inventory of these additives' attributes.

Figure 2: Additives employed in the study included: (a) SBS polymer (b) 701 epolymer (c) 611 polymer

Table 2: Characteristics of the additives used in the study [28]

Features	SBS	701	611
Molecule Structure	Liner	Liner	Radial
Styrene/Butadiene Ratio	31/69	33/67	31/69
Density (cm/m^3)	0,94	0,93	0,94
Fat Content	Yok	Yok	Yok
Viscosity (5%, cps)	-	14	24
Melting index (190°C/kgw)	<1	<1	<1
Tensile strength (kgw/cm^2)	324	220	200
Stiffness	70	80	83
Ash content	-	0,2	0,2
Elongation at break (%)	880	600	700

2.2. Preparation of Modified Asphalt

In order to create target binders, three different polymers were added to pure bitumen at different ratios (2%, 3%, and 4%). Mechanical mixer equipment was used in the preparation of the modified binder. First, the pure bitumen was heated for 30 minutes at 170±5°C in an oven to fluidize it. After that, 500 grams of the fluidized bitumen were added to the mixer's metal hopper. The bitumen in the metal hopper was left in a thermal shirt on the heater source conditioned at 170±5°C, keeping the heater running until it reached a thermal equilibrium of 170±5°C. This ensured a homogeneous thermal source. In predetermined weight percentages of the designated bitumen, the polymers were added to the hot bitumen. In order to prepare the bitumen containing polymers, a mechanical mixer running at 1000 rpm for four hours was used [29]. To counteract the oxidation impact, the pure binder was also mixed by using the same technique. By putting the pure binder through the modified bitumen mixing process, the S1 binder was produced. Pure bitumen with a penetration grade of 50/70 is indicated by the S2 binder. Table 3 provides a summary of the acronyms used in the study for binders that contain three different polymers as well as pure binders.

Table 3: Abbreviations and definitions for diverse asphalt binders are outlined in Table 3.

Asphalt type abbreviation	Polymer Content (%)			
	0	2	3	4
SBS	-	M1	M3	M5
701	-	M6	M8	M10
611	-	M11	M13	M15
Pure bitumen	S1-S2	-	-	-

2.3. Aging

The aging (short-term aging) of the binder during mixing under the influence of temperature and air is simulated in the laboratory with the Rotational Thin Film Oven Test (RTFOT). The bitumen to be used in the experiment is heated until it becomes fluid. 8 RTFOT test bottles are filled with 35 ± 0.5 g of bituminous binder and placed in the rotating system at 15 rpm around the horizontal axis. In the experiment carried out for 85 minutes at a temperature of 163 ± 0.5°C and an air flow of 4000 ± 200 ml/min, the bituminous binder completely covers the inside of the bottle and forms a thin film layer due to the rotational movement in the system, temperature and the effect of blowing air into the bottles at each rotation.

Pressure Aging Vessel (PAV) is used for long term aging of bituminous binders, i.e. to determine the long term hardening properties of bituminous binders during the service life of the pavement. After the aged bitumen samples obtained from the RTFOT test are heated and fluidized, they are poured into each of the steel test containers weighing 50 grams in accordance with the AASHTO PP1 standard and placed on the shelves. They are allowed to age under high pressure of 2.070 kPa for 20 hours in a closed container at a temperature of 100°C (temperature level varies according to the type of bituminous binder used).

2.4. Dynamic shear rheometer test

The phase angle represents the temporal gap between applied shear stress and the resulting shear deformation. In contrast, the complex shear modulus reflects the entire resistance to deformation during binder torsion within a certain timescale. A heightened phase angle implies a more viscous nature of the bituminous binder. According to [30], the rutting parameter ($G^*/\sin\delta$) for aged binders is kept at values higher than 2.2 kPa in order to prevent permanent deformation. Untreated and Rolling Thin Film Oven Test (RTFOT) aged bituminous binders are used to evaluate bituminous binder resistance to rutting, whereas Pressure Aging Vessel (PAV) aged binders are used to evaluate bituminous binder fatigue behavior. Samples are 25 mm in diameter and 1000 microns in height for rutting resistance testing and 8 mm in diameter and 2000 microns in height for fatigue resistance testing. The Bohlin DSRII rheometer is used to evaluate both pure and modified bitumen in accordance with ASTM D7175. The experiments are carried out using a frequency of 1.59 Hz, a plate with a 25 mm diameter and a 1 mm plate aperture, and temperatures ranging from 58°C to 88°C.

3. Rheological Properties of Aged Binders

Short-term aged binders were obtained by subjecting 611 (M11, M13 and M15), 701 (M6, M8 and M10), and SBS (M1, M3 and M5) modified binders to a rotational thin film heating test (RTFOT) and obtaining pure binder (S2) and pure binder (S1) prepared under the preparation conditions of the modified binder. The DSR test was performed on short-term aged binders, and G*/sinδ values were obtained. These values were then compared with the specification requirement (2200 Pa) after RTFOT. Figures 3-5 show the variations in G*/Sinδ values of aged binders with temperature. Figure 3 shows the results of the binders obtained with 611 elastomers. Analyzing the rutting strength characteristics (G*/sinδ) shown in Figure 3, it can be shown that these values steadily rise when additives are used and steadily fall when temperature rises. The G*/sinδ values obtained from the three additive ratios utilized are observed to differ. The G*/sinδ values are observed to approach each other after 82 °C. Upon evaluating the rutting strength limit value of 2200 Pa for aged binders using the Superpave method, it was found that all binders (all modified bitumen) met the specification criteria at 52 °C, 58 °C, 64 °C, and 70 °C, with the exception of S2 binder at 76 °C. The specification requirements were met by the S1 binder at 82 °C and by all modified binders at 88 °C. For S1 binder, the G*/sinδ values increased by 2.7, 3.9, and 4.6 times at 88 °C, while for S2 binder, the increases were 3.0, 4.4, and 5.1 times.

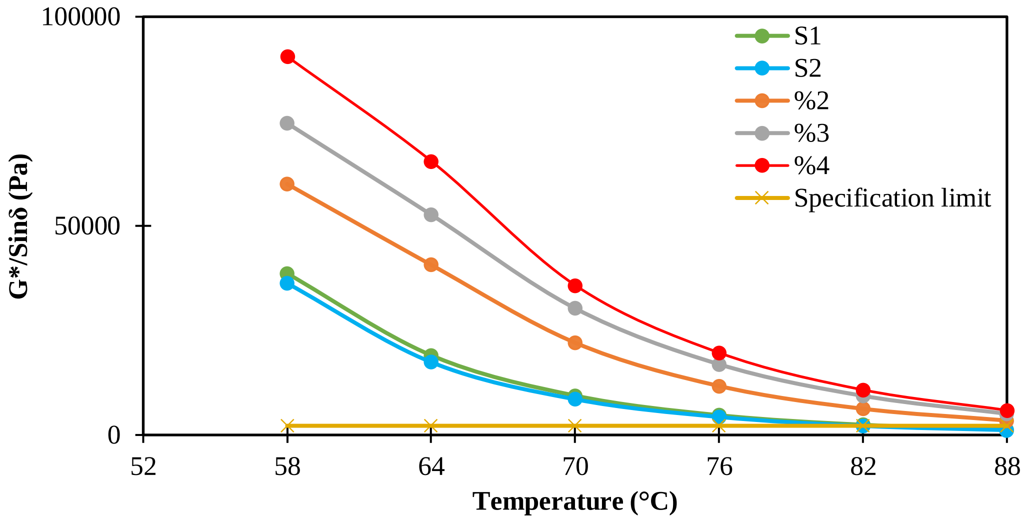

Figure 3: G*/Sinδ-temperature relationship of aged binders obtained from 611 modification

Figure 3 shows the results of the binders obtained with 701 elastomers. When the rutting strength parameters (G*/sinδ) given in Figure 4 are analysed, it is seen that these values increase steadily with the use of additives in aged binders and decrease steadily with the increase in temperature. When Figure 4 is analysed, G*/sinδ values of 2% and 3% additive contents are very close to each other. The closeness in G*/sinδ values is not observed in the unaged binder. The assessment revealed that the S2 binder at 76 °C, S1 binder at 82 °C, and modified bitumen binder at 88 °C all met the specified criteria. In comparison to the S1 binder, G*/sinδ values at 58 °C increased by 1.7, 1.8, and 2.5 times, respectively, whereas compared to the S2 binder, the increases were 1.8, 1.9, and 2.7 times, respectively. At 88 °C, the G*/sinδ values in relation to the S1 binder escalated by 2.2, 2.3, and 4.3 times, respectively; compared to the S2 binder, the increases were 2.4, 2.5, and 4.7 times, respectively. Notably, the binder containing 4% of 701 (M10) exhibited the highest G*/sinδ value.

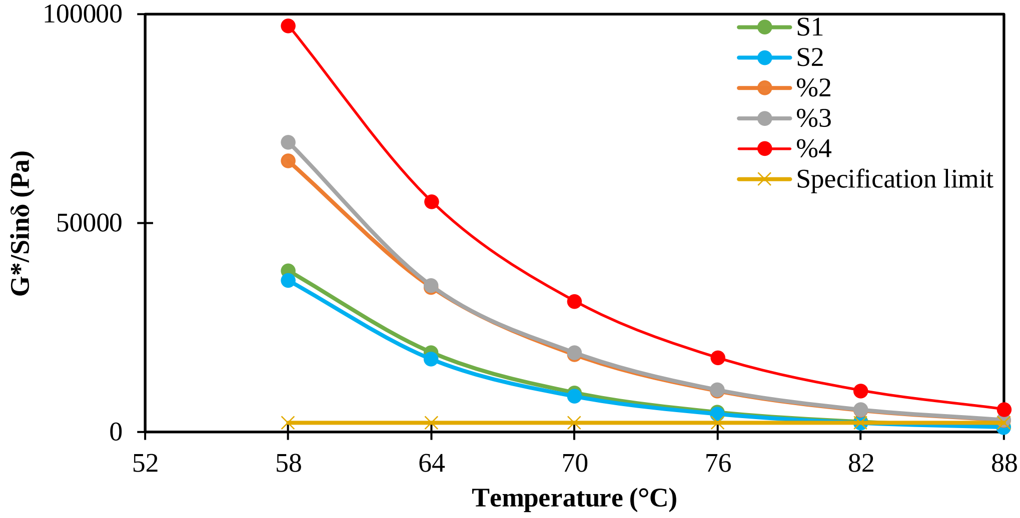

Figure 4: G*/Sinδ-temperature relationship of aged binders obtained from 701 modification

Figure 4 shows the results of the binders obtained with SBS elastomers. When the rutting strength parameters (G*/sinδ) given in Figure 5 are analysed, it is seen that these values increase regularly with the use of additives in aged binders. There is a regular decrease in G*/sinδ values with increasing temperature. After 70 °C, G*/sinδ values became close to each other. In SBS elastomer, G*/sinδ values are close to each other in all three additives after ageing. There is not much difference between them. At 88 °C, it was determined that the binders met the specification criteria in all three additive contents. For S1 binder, G*/sinδ values increased by 1.97, 2.17, and 2.32 times at 58 °C, and for S2 binder, by 2.10, 2.31, and 2.47 times. At 88 °C, G*/sinδ values increased by 2.89, 3.14, and 3.62 times, respectively, in comparison with S1 binder; in comparison with S2 binder, they grew by 3.22, 3.50, and 4.02 times, respectively. The binder containing 4% SBS (M5) yielded the highest G*/sinδ value, whereas the S2 binder yielded the lowest value.

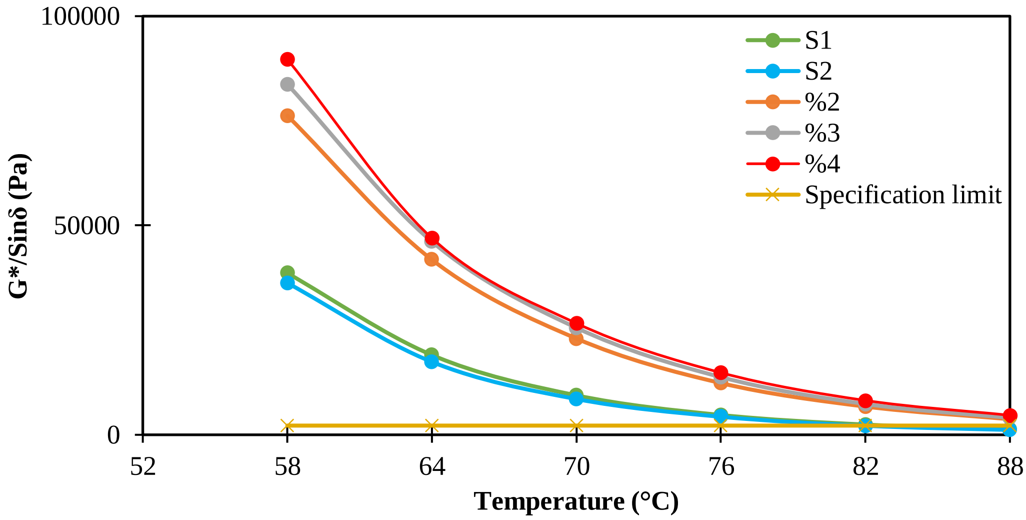

Figure 5: G*/Sinδ-temperature relationship of aged binders obtained from SBS modification

Figure 6 illustrates that the M15 binder consistently exhibits the highest G*/sinδ value across all temperatures. Notably, G*/sinδ values at 58 °C vary significantly. The G*/sinδ values of Properties 701 and SBS binder markedly differ from the G*/sinδ values of unaged binders. Following the M15 binder, both the M5 and M10 binders demonstrate the highest and relatively similar performance. Moreover, at 2% and 3% additive content, the G*/sinδ values of SBS-modified binders surpass those of 701-modified binders. It was determined that these results were due to aging. At 4% additive content, the opposite result is observed. When the G*/sinδ values at 70 °C are analysed, the change compared to S1 and S2 binder is very high compared to the other two temperatures. As the additive content increases, the change in G*/sinδ values is observed. At 88 °C, the difference between G*/sinδ values increases as the additive content increases. In addition, the difference between the elastomers used increased. At 88°C, the highest G*/sinδ value occurs in M15 binder as in other temperatures. Regardless of other properties, it can be said that M15 binder offers the best performance among the aged binders only in terms of rutting resistance. After M15 binder, it can be said that M5 and M10 binders have the best rutting resistance. When Figures 5.38-5.40 are analysed, the best rutting resistance among the aged binders is given by binders modified with 611 elastomer. Compared to the unaged binders, the binders treated with SBS and 701 elastomers have a different rutting resistance. Furthermore, an analysis of the data shows that elastomers raise the G*/sinδ values four to five times higher than pure binders. According to these findings, using elastomers will help the coatings endure longer. Modified bitumen preparation conditions were used to prepare S1 binder. At this point, it was shown that the aging of pure bitumen was responsible for the rise in G*/sinδ values when compared to pure binder (S2).

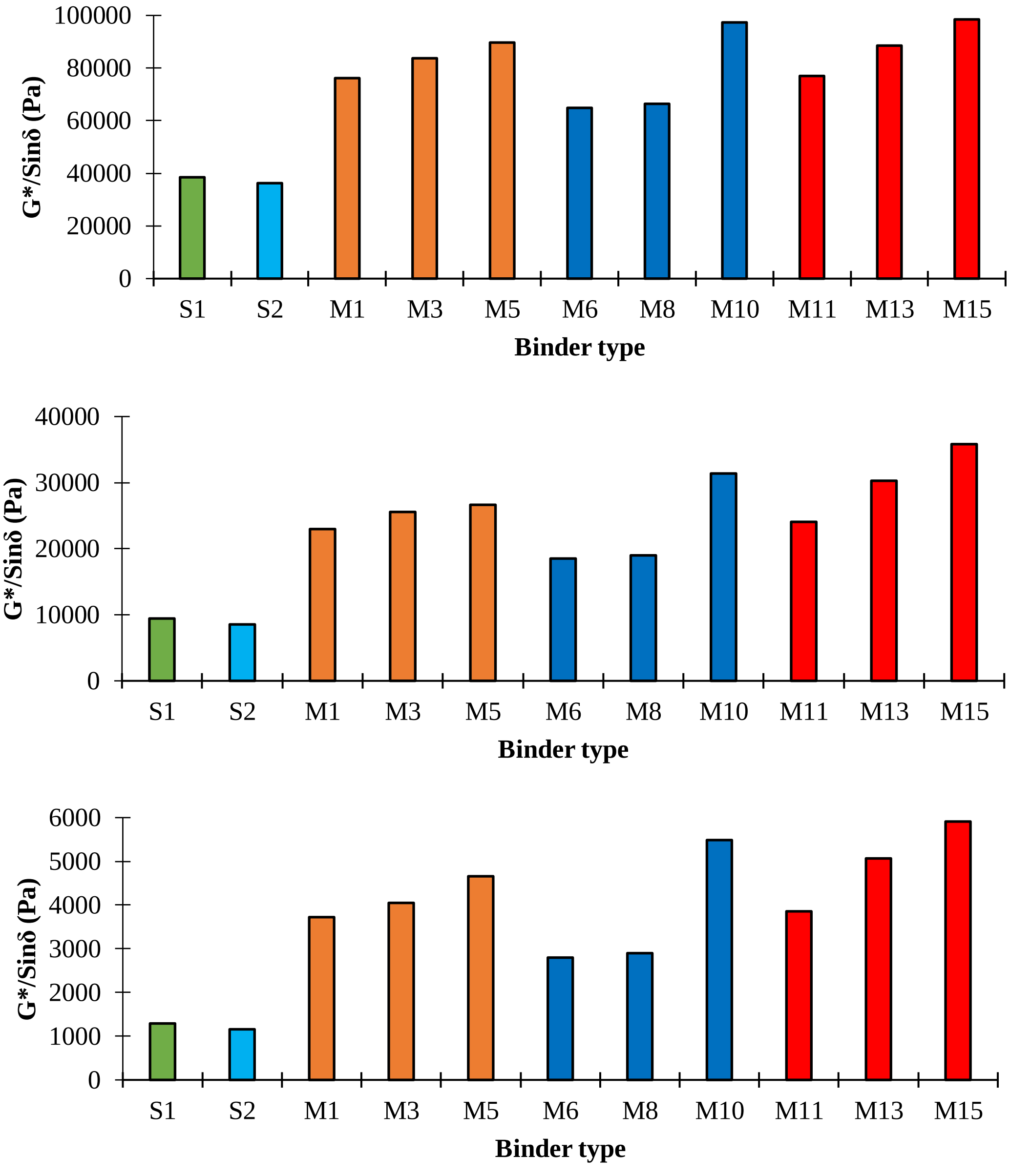

Figure 6: G*/sinδ values of aged binders.

The changes in the phase angles of the aged binders with temperature are given in Figures 7-9. As seen in Figure 7, the lowest phase angle was obtained at 4% additive content in aged binders as in unaged binders. After 76 °C, the phase angle values of 3% additive content and 4% additive content are close to each other. In addition, an increase in phase angle values is observed as the temperature increases. According to these results, the elasticity of the binders increases as the additive content increases. The binder with 4% additive content is the most elastic. The binder with the highest value is the S2 binder.

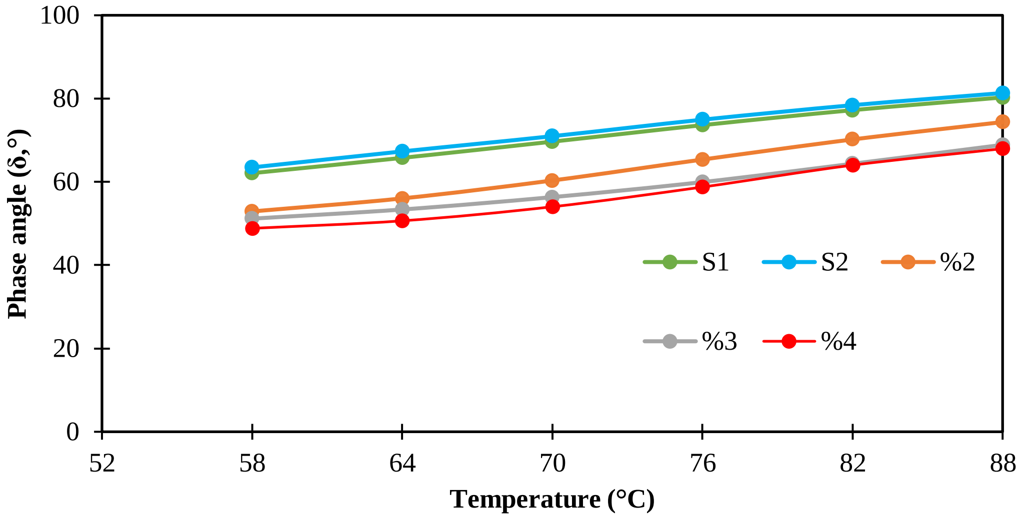

Figure 7: Relationship between phase angle and temperature of old binders derived from 611 modification

As seen in Figure 8, as the additive content increases, the phase angle values of the aged binders decrease. This decrease is maximum at 4% additive content. Phase angle values of 3% and 4% additive contents of 611 additive differ compared to 701 additive. Unlike 611 additive, the phase angle values of 3% and 4% additive contents in 701 additive are similar to each other. As the temperature value increases, phase angle values increase. The phase angle values of pure binders are close to each other. As seen in Figure 8, the phase angle values at 58°C decreased by 10.86%, 14.06% and 18.88% for S1 binder and 12.79%, 15.93% and 20.64% for S2 binder, respectively. The phase angle values at 88°C decreased by 6.23%, 7.20% and 13.52% for S1 binder and 7.44%, 8.40% and 14.63% for S2 binder, respectively.

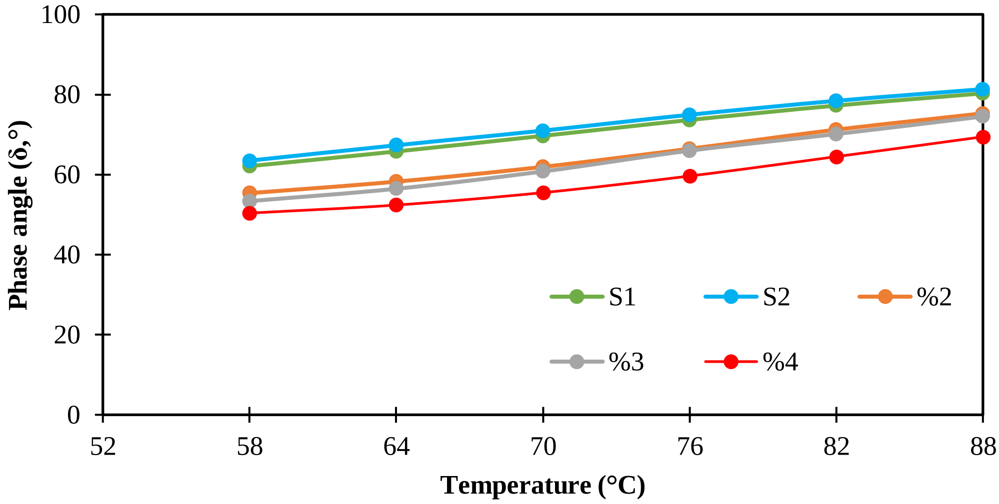

Figure 8: Relationship between phase angle and temperature of aged binders derived from 701 modification

In Figure 9, phase angle values decrease as the additive content increases. This decrease is maximum at 4% additive content. Phase angle values are similar at 2% and 3% additive contents. As the temperature value increases, phase angle values increase. Phase angle values are very close at high temperatures. As seen in Figure 9, the phase angle values at 58°C decreased by 12.95%, 13.92% and 18.26% for S1 binder and 14.84%, 15.79% and 20.04% for S2 binder, respectively. The phase angle values at 88°C decreased by 10.38%, 11.81% and 13.86% for S1 binder and 11.53%, 12.95% and 14.98% for S2 binder, respectively. The lowest phase angle value of 4% additive content is the most flexible behaviour for SBS binder.

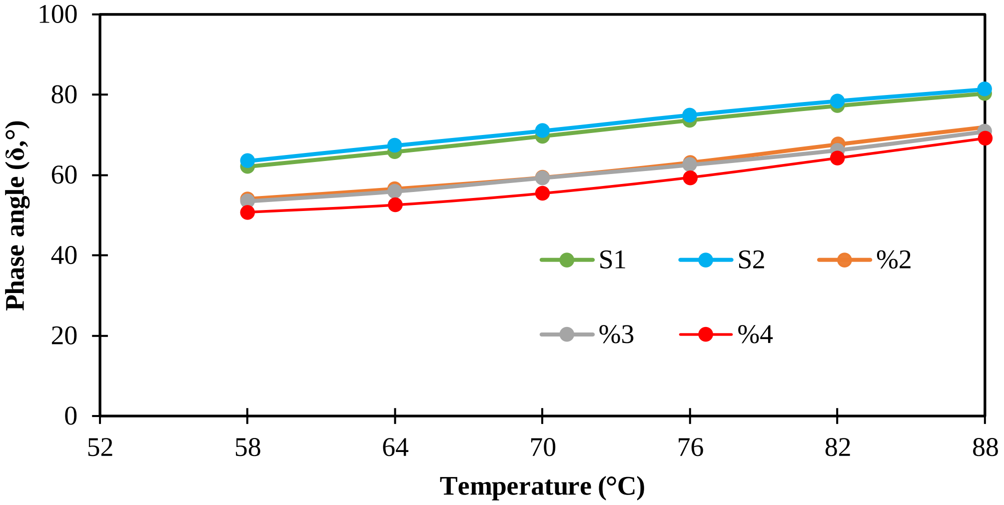

Figure 9: Aged binders' phase angle-temperature relationship as determined by SBS modification

The results obtained from the DSR test applied to binders aged for a long period by PAV method are given in Table 4. The DSR test was performed in 8 mm diameter and 2 mm specimen height geometry. As a result of the tests, the fatigue parameters (G*/sinδ) values of the binders were determined. According to the Superpave specification, the G*/sin δ parameter should be maximum 5.00E+06 (5,000,000) Pa at the test temperature.

Table 4: DSR test results applied to long-term aged binders using the PAV method

Binder type	Temperature (°C)	G* (Pa)	δ (°)	G*.sinδ (Pa)
S1	22	6.37E+06	35.73	3.72E+06
	25	4.47E+06	37.40	2.71E+06
	28	3.10E+06	39.02	1.95E+06
	31	2.17E+06	40.49	1.41E+06
	34	1.49E+06	42.07	9.99E+05
S2	22	6.69E+06	37.52	3.91E+06
	25	4.69E+06	39.27	2.85E+06
	28	3.26E+06	40.97	2.05E+06
	31	2.28E+06	42.5	1.48E+06
	34	1.56E+06	44.17	1.05E+06
M1	22	7.71E+06	34.28	4.34E+06
	25	5.57E+06	35.73	3.25E+06

	28	3.99E+06	37.07	2.40E+06
	31	2.85E+06	38.39	1.77E+06
	34	2.05E+06	39.64	1.31E+06
M3	19	8.19E+06	32.88	4.44E+06
	22	6.07E+06	34.11	3.41E+06
	25	4.56E+06	35.12	2.62E+06
	28	3.42E+06	36.10	2.01E+06
	31	2.52E+06	37.21	1.53E+06
	34	1.87E+06	38.27	1.16E+06
M5	19	9.08E+06	32.47	4.88E+06
	22	6.69E+06	33.87	3.73E+06
	25	4.94E+06	34.97	2.83E+06
	28	3.61E+06	36.24	2.13E+06
	31	2.64E+06	37.34	1.60E+06
	34	1.94E+06	38.48	1.20E+06
M6	22	6.80E+06	34.67	3.87E+06
	25	4.91E+06	36.07	2.89E+06
	28	3.52E+06	37.33	2.13E+06
	31	2.51E+06	38.63	1.57E+06
	34	1.51E+06	39.12	0.89E+06
M8	22	7.83E+06	34.09	4.39E+06
	25	7.83E+06	34.09	4.39E+06
	28	5.69E+06	35.63	3.31E+06
	31	4.11E+06	37.01	2.48E+06
	34	2.96E+06	38.39	1.84E+06
M10	22	7.32E+06	33.19	4.01E+06
	25	5.49E+06	34.42	3.10E+06
	28	4.00E+06	35.69	2.33E+06
	31	2.93E+06	36.91	1.76E+06
	34	7.32E+06	33.19	4.01E+06
M11	22	7.36E+06	33.68	4.08E+06
	25	5.37E+06	35.08	3.09E+06
	28	3.90E+06	36.40	2.32E+06
	31	2.82E+06	37.78	1.73E+06
	34	7.36E+06	33.68	4.08E+06
M13	22	7.21E+06	34.85	4.12E+06
	25	5.51E+06	36.00	3.24E+06
	28	4.13E+06	37.39	2.51E+06
	31	3.10E+06	38.72	1.94E+06
	34	7.21E+06	34.85	4.12E+06

M15	19	8.82E+06	31.83	4.65E+06
	22	6.82E+06	33.18	3.73E+06
	25	5.24E+06	34.47	2.97E+06
	28	4.05E+06	35.98	2.38E+06
	31	3.08E+06	37.23	1.86E+06
	34	2.34E+06	38.66	1.46E+06

4. Conclusion

The impact of employing three different elastomers (SBS, 611, and 701) on bitumen characteristics was examined in this work. Both short- and long-term aging was applied to pure and modified binders. For every aged binder, the Dynamic Shear Rheometer (DSR) test was conducted. The test outcomes were contrasted. The aging resistance of the binder is much higher with the elastomer modification than it is with the pure binder. An analysis of the rutting resistance metrics (G*/sinδ) reveals that these values continuously rise when additives are added to aged binders and steadily fall when temperature rises.

As the amount of elastomer addition increased, so did the binders' complex modulus values. The influence of elastomer content decreased at high frequencies, while the effect of additives was more noticeable at low frequencies. The binders containing 4% additive content exhibited the greatest values of complex modulus. Out of the three-elastomer additives, the binder with the highest complex modulus was found to have a 4 701% additive content. It was found that the lowest phase angle values of three elastomer types lowest phase angle values, which ranged from 55 to 60°C, were produced in 4% modified bitumen. The modified bitumen's phase angle values rose after reaching the G* value of 1.0E+5 Pa, suggesting that the modified bitumen made with all three elastomers had comparable boundary shear stress strengths. The elasticity behavior of the binders rises as the phase angle value lowers. In aged binders, 611 elastomer gives higher rutting performance than SBS elastomer. In addition, the binder containing 611 elastomer was determined to be more elastic than the binder containing SBS.

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