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Kısa Dönem Yaşlandırılmış Saf ve SBS Modifiyeli Asfalt Bağlayıcıların Yaşlanma Davranışlarının Reolojik Araştırması

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Öz

Esnek üstyapılar bitümlü bağlayıcı ve uygun gradasyonda agreganın karışımından elde edilmekte ve bitümün performansı kaplamanın nihai performansı üzerinde belirleyici faktör olmaktadır. Bitüm, üretilmesinden uygulanmasına kadar geçen sürede kısa dönemli yaşlanmaya maruz kalır ve bu yaşlanma servis ömrü boyunca meydana gelecek yaşlanmanın büyük kısmını oluşturur. Bu sebeple kısa dönemli yaşlanma davranışı oldukça önemlidir. Bu çalışmada çeşitli analitik yaklaşımlar ile 70/100 saf bitüm ve Stiren-Butadien-Stiren (SBS) modifiyeli bitümün yaşlanma dayanımları reolojik olarak incelenmiştir. Bitüm numuneleri üzerinde Dinamik Kayma Reometresi (DSR) cihazı ile farklı frekans (0.01-10 Hz) ve sıcaklık (40, 50, 60, 70°C) aralıklarında Frekans Tarama testi uygulanmıştır. Deneysel veriler işlenerek bağlayıcıların kompleks modül ve kompleks viskozite ana eğrileri elde edilmiş, eğriler reolojik analizlere tabi tutulmuştur. Sonuçlar, SBS modifikasyonu ile yaşlanma esnasında elastik özelliklerin korunma kabiliyetinin artırılarak yaşlanma direncinin iyileştirildiğini göstermiştir. Ayrıca, ana eğriler reolojik modellere yüksek doğrulukta uygulanmıştır.

Anahtar kelimeler: Bitüm, SBS, Reolojik model, DSR, Frekans taraması

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Rheological Analysis of The Aging Behavior of Short-Term Aged Pure And SBS Modified Asphalt Binders

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Abstract
Flexible pavements consist of a bituminous binder and certain graded aggregate. The performance of the bitumen determines the overall pavement performance. Bitumen undergoes short-term aging from production to application, accounting for most of its total aging during service life. This study rheologically investigates the short-term aging behavior of 70/100 pure bitumen and SBS-modified bitumen. Frequency Sweep test was performed on bitumen samples with Dynamic Shear Rheometer (DSR) device at different frequency (0.01-10 Hz) and temperature (40, 50, 60, 70°C) ranges. The complex modulus and complex viscosity master curves of the binders were obtained by processing the experimental data and the curves were subjected to rheological model analysis. The study showed that SBS modification enhanced aging resistance by preserving elastic properties. Moreover, the master curves were applied to rheological models with high accuracy.

Keywords: Bitumen, SBS, Rheological model, DSR, Frequency sweep

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1. Introduction

Bitumen is a thermoplastic material obtained by refining petroleum [1]. After complex processes such as volatilization, oxidation and condensation, bitumen becomes harder and more brittle, which leads to various structural problems [2-3]. This whole process is called aging of bitumen. The aging process is analyzed in two parts as short-term and long-term aging [4-5]. Both types of aging significantly change the physical and chemical properties of bitumen, but in different time periods and conditions [6]. Considering the entire aging process of bitumen, the greatest aging occurs in the short term. For this reason, the measures to be taken in the short-term aging process are critical.

Short-term aging occurs during the production and application of bitumen [7]. During hot mix asphalt production, binder is mixed with hot aggregate and exposed to high temperatures. This process usually takes place between 140°C and 160°C. The high temperature starts to change the chemical structure of the bitumen [8]. Under the influence of heat, the more volatile components of bitumen evaporate, oxidation of larger molecules becomes easier and hardening begins. Oxidation is one of the main causes of short-term aging [9]. During heating, the surface of the bitumen comes into contact with oxygen in the air and oxidative reactions begin. These reactions result in the oxidation of aromatic and unsaturated components, especially in bitumen. During transportation and paving of the bitumen mixture from the production plant to the construction site, the bitumen still remains at high temperatures. At this stage, oxidation and loss of volatile components continues [10]. Since the surface layer of the bitumen is in contact with more oxygen, short-term aging may be more pronounced on this surface. After the mixture is laid, compaction is performed. During this process, the temperature of the asphalt mixture is still high. During compaction, both oxidation continues and some components in the mix condense and cause hardening [7-12]. Short-term aging is simulated in the laboratory using the Rolling Film Thin Oven Test (RTFOT) and the aging resistance of different bitumens can be determined after this test method [13-14].

Various additives are used to improve the aging resistance of bitumen, and the effects of polymer additives are generally investigated. Zhang et al. emphasized that aging of modified bitumen has significant effects on pavement properties and investigated the effect of polymer modification on aging by a series of experiments in the laboratory. The results showed that the stress relaxation property of bitumen decreases with aging and tends to behave with higher viscosity due to the decrease in phase angle values. Polymer additive was found to help bitumen to show good elastic properties under short-term aging conditions [15]. Liu et al. stated in their study that SBS, which is the most common polymer additive, may cause various disadvantages due to its high viscosity. Within the scope of the study, the effect of SBS in combination with polyphosphoric acid (PPA) on the aging performance of bitumen was investigated. The results showed that some physical properties such as complex modulus of SBS changed when PPA was added and some properties such as zero shear viscosity, relaxation stress were not affected independently of RTFO short-term aging. It was concluded that PPA would improve the short-term aging resistance [16]. Another common additive is crumb rubber (CR) derived from waste vehicle tires. CR provides significant performance gains to bitumen. While obtaining a CR modified asphalt pavement, various negative effects can occur due to the increase in construction temperatures. Jin et al. subjected CR modified bitumen to RTFOT at different aging temperatures. The experimental results show that the appropriate RTFOT temperature is related not only to the viscosity of the CR modified binders but also to the mix gradation. Binders with higher viscosity require elevated RTFOT temperatures between 173 °C and 193 °C to simulate short-term aging, especially for mixtures with higher air voids. Furthermore, swelling of CR in bitumen has been associated with improved aging performance [17]. In another study, the aging behavior of bitumen with SBS and CR was investigated. As a result of the study, it was determined that CR would increase the aging resistance by preventing polymer degradation in SBS modified bitumen [18].

In this study, 70/100 pure bitumen and bitumen containing 2% SBS were subjected to short-term aging (RTFOT) and their aging performances were investigated. In addition to experimental data, various rheological models and mathematical analyses were used to deepen the evaluation of aging effects. Within the scope of the study, bitumen samples were subjected to frequency sweep testing with a dynamic shear rheometer (DSR) device. The effects of different temperatures and loading rates on unaged and aged binders were investigated and the modification effect was determined. The frequency sweep test was performed at

10 different frequencies (0.01-10 Hz) and 4 different temperatures (40, 50, 60, 70°C) and master curves were obtained using the complex modulus values obtained from the experiment.

2. Materials and Method

In this section, the bituminous binder, additives and analysis methods used in the study are discussed. In addition, the experimental preparation process and modified bitumen preparation process are given. The experimental and theoretical approaches preferred in the study are explained in accordance with the purpose of the study.

2.1. Materials

In this study, 70/100 penetration grade pure bitumen obtained from TÜPRAŞ Batman refinery was used. Styrene-butadiene-styrene (SBS) was used as additive. The properties of pure bitumen and SBS are given in Table 1.

Table 1. Properties of 70/100 bitumen and SBS

Bitumen			SBS	
Property	Unit	Value	Property	Value
Penetration	dmm	86.5	Molecular structure	Linear
Softening Point	°C	51.4	Styrene/butadiene ratio	31/69
Flash Point	°C	230	Density (gr/cm^3)	0.94
Density	gr/cm^3	1.034	Oil content	N/A
			Melting index (kgw/cm^2)	<1
			Stiffness	70

2.2. Method

Within the scope of the study, the differences in the mechanistic and rheological behavior of pure and SBS modified bitumen after short-term aging were analyzed. The experimental flowchart is given in Figure 1.

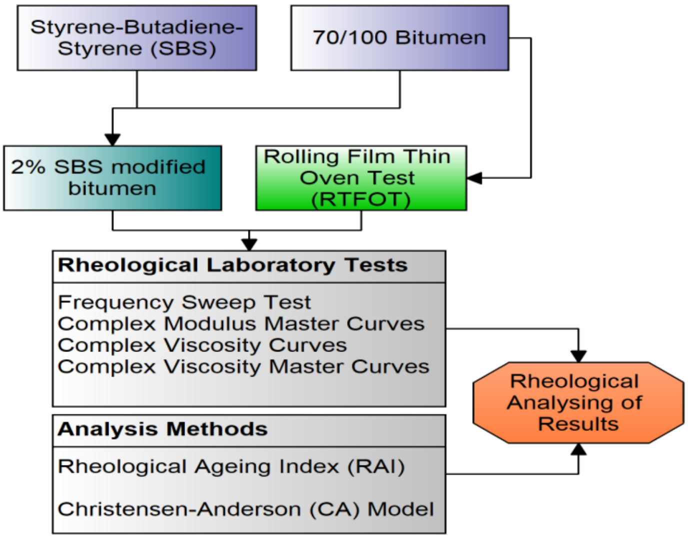

Figure 1. Flowchart of the experimental procedure

2.2.1. Preparation of SBS modified bitumens

Modified bitumen was obtained by adding 2 wt% SBS additive to bituminous binder with 70/100 penetration degree liquefied at appropriate temperature. The modification process was carried out at 180°C, 1000 rpm speed for 1 hour.

2.2.2. Rolling film thin oven test (RTFOT)

The RTFOT test is conducted in accordance with TS EN 12607-1 [19] and is designed to replicate the aging process that occurs from the time of production to the stages of paving and compaction. This testing methodology is applicable to both unmodified and modified binders, focusing on the thermal and oxidative aging associated with short-term aging. In the course of the test, an bitumen film is created and subjected to airflow at a temperature of 163°C for a duration of 85 minutes. This process accelerates the oxidation of the bitumen, resulting in an increase in the binder's viscosity and a notable alteration in its hardness. The implications of short-term aging are critical, as they influence the initial performance characteristics of the bitumen, particularly regarding workability, adhesion, and resistance to cracking within the mixture. The RTFOT test for modified bitumens is an essential instrument for assessing the impact of additives, thereby facilitating the evaluation of the binders' resistance to aging. RTFOT test device was given in Figure 2.

Figure 2. RTFOT device and test samples

2.2.3. Frequency sweep test and master curve construction

The frequency sweep test is performed with the Dynamic Shear Rheometer (DSR) device and the behavior of bitumen under oscillation loading at different frequencies is examined. This loading at different frequencies characterizes the viscoelastic properties of bitumen. Different frequencies within the scope of the experiment represent different loading speeds and it has been determined that a loading frequency of 10 Hz corresponds to a speed of 60-65 km/h [20]. Oscilatory tests were performed on pure and SBS modified bitumen at four different temperatures (40°C, 50°C, 60°C and 70°C) and ten different frequencies (0.01-10 Hz). The DSR device and test specimens are shown in Figure 3.

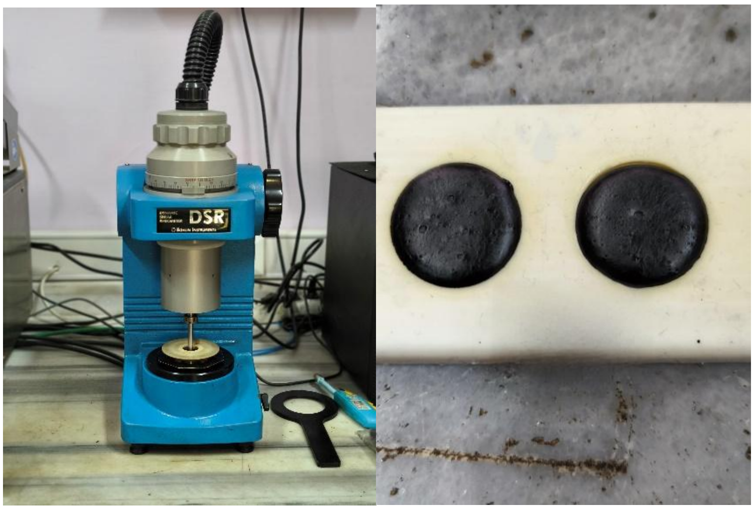

Figure 3. DSR device and test samples

As a result of the experiment, important rheological parameters such as elastic modulus (G'), viscous modulus (G''), complex modulus (G*), phase angle (δ), complex viscosity are obtained for different frequency values of binders at each temperature. The G* values obtained after the experiment are converted into a "master curve" at a certain reference temperature value in order to examine the rheological behavior of bitumen in a wide frequency range by adhering to the Time-Temperature Superposition Principle (TTSP). The obtained master curves were subjected to rheological analysis according to the Christensen-Anderson (CA) Model. In this model, presented in Equation 1, the rheological behavior is described in terms of G* values as a function of the frequency applied to the bituminous binder. Although the model was originally intended to characterize pure bitumen, it has recently been used to describe the behavior of modified bitumen. Numerous studies have been conducted with the CA model [21-23]. After CA Model analysis, glass modulus (Gg), crossover frequency (ωc) and rheological index (R) values are obtained for pure and modified binders. As a result of the studies, it was determined that the Gg value can be accepted as $1x10^9$ for all bituminous binders.

ωc represents the frequency at which the viscous and elastic modulus values are the same. It is also the point where the viscous asymptote and the glassy asymptote overlap. ωc characterizes the overall hardness of the bitumen. R is defined as the difference between the complex modulus at ωc and the intercept asymptotes. It is also called shape factor. An increase in R indicates a decrease in the viscous properties and an improvement in the elastic properties of the binder at intermediate loading times and temperatures and gives the idea that it will show wider relaxation spectra.

$$|G^*| = G_g \left[1 + \left(\frac{\omega_c}{\omega}\right)^{\frac{log2}{R}}\right]^{\frac{-R}{log2}} \quad (1)$$

2.2.4. Rheological ageing index (rai)

Complex modulus values obtained at different temperatures and loading rates (frequency) show that the mechanistic behavior of bitumen changes significantly with temperature and frequency. In previous studies, the area between the master curves of bitumen before and after aging was found to be an important indicator of aging, and the rheological aging index (RAI) was introduced to evaluate aging over a wide frequency range [24-25].

3. Results and Discussions

The TTSP master curves of the G* values of pure and 2% SBS modified bitumen before and after short-term aging process (RTFOT) are given in Figure 4.

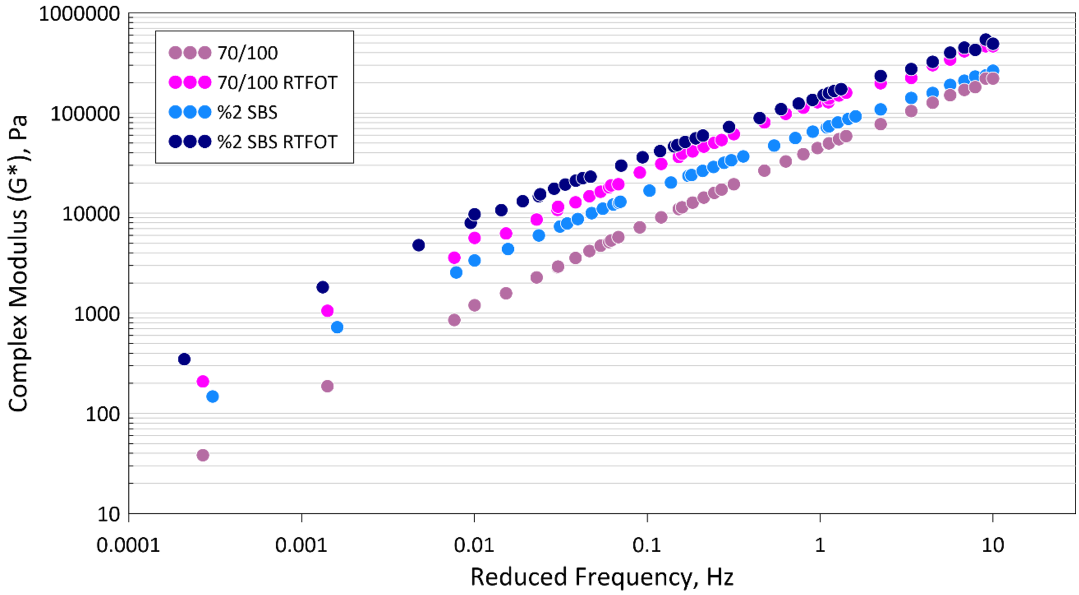

Figure 4. Master curves of pure and 2% SBS modified bitumens

When creating complex shear modulus (G*) master curves, a reference temperature is selected and other isothermal curves are shifted to this temperature value. Asphalt pavements are usually subjected to loading at moderate temperatures (20°C–60°C). A reference temperature is usually chosen within this range because the rheological properties of the bitumen are particularly important. For the master curve given in Figure 4, the reference temperature is 40°C [26]. The complex modulus values increased with increasing frequency, i.e. loading rate. When low frequency (long-term loading rate) is applied, the shear strength of the binders decreases. This is associated with the viscoelastic behavior of the material. Among the unaged bitumen, the lowest G* values were observed in 70/100 bitumen, while G* values increased with the addition of 2% SBS. After RTFOT, G* values of all binders were higher than their pure state. The short-term aging process constitutes the major part of the total aging process and the bituminous binder is subjected to intense oxidation during this process. The material hardens due to the removal of volatile components from the bitumen, the functional groups formed in the bitumen due to oxidation and the increase in the proportion of components with high molecular weights. The desired phenomenon when obtaining the master curve is that the curve is smooth and continuous. When Figure 4 is examined, it is seen that the curve is smooth and continuous for each binder type, indicating the applicability of TTSP and that the binders are “thermo-rheologically simple”.

The master curves were analyzed using the Christensen-Anderson (CA) Model to conduct detailed rheological evaluations. The curves were fit according to Equation 1 and the model parameters were obtained and given in Table 2.

Table 2. CA Model paramaters

Sample Name	Parameter	Value	R^2
70/100	Gg	1.00E+09	0.99
	ωc	1974.085	
	R	2.30379	
70/100 RTFOT	Gg	1.00E+09	0.99
	ωc	183.9779	
	R	2.62321	
2% SBS	Gg	1.00E+09	0.99
	ωc	72.98642	
	R	3.0771	
2% SBS RTFOT	Gg	1.00E+09	0.99
	ωc	24.1748	
	R	3.12519	

Table 2 confirmed that the CA Model successfully fits both aged and unaged bitumen master curves (R^2>0.99). As mentioned in the methodology section, the Gg value was fixed at 10^9 and R and ωc values were left free. It was observed that both pure and 2% SBS binder presented higher R values with the aging effect after RTFOT. The R-value of pure bitumen increased by about 14% after RTFOT, while that of modified bitumen was 1.63%. The R-value is related to the total amount of colloidal matter in the bitumen and is therefore reported to be proportional to the asphaltene content [27-28]. With aging, maltenes with lower molecular weights degrade and some of their components, especially aromatic components, are converted into asphaltenes. As the maltene ratio decreases and the asphaltene ratio increases, R values also increase. The ωc value refers to the frequency value at which the storage and viscous modulus are equal and is interpreted as the point at which the material switches from elastic to viscous behavior. Since bitumen hardens, it starts to exhibit viscous behavior at low frequencies and therefore ωc decreases. When Table 2 is analyzed, the lowest ωc value is observed in 2% SBS RTFOT binder, while the highest ωc value is observed in 70/100 pure bitumen. The ωc values of both binders decreased with the effect of aging.

Figures 5 and 6 show the calculation of the RAI value representing the aging of 70/100 and 2% SBS modified bitumen.

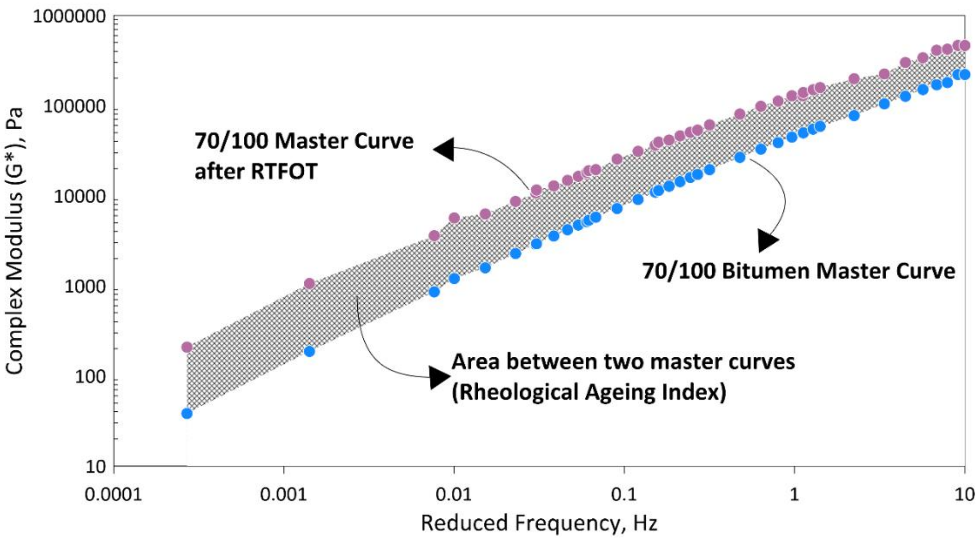

Figure 5. RAI calculation of 70/100 bitumen

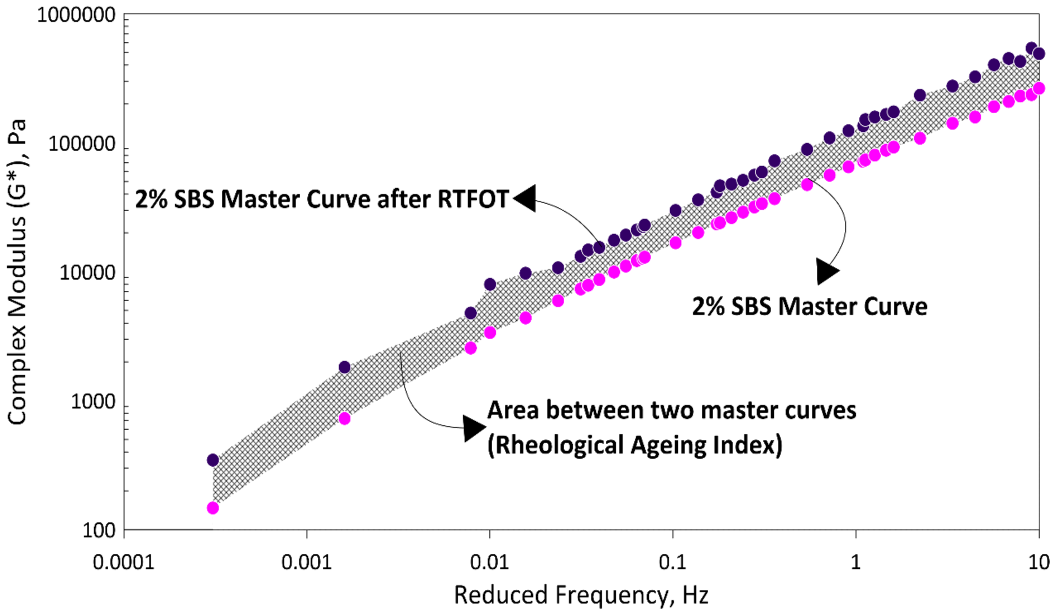

Figure 6. RAI calculation of 2% SBS modified bitumen

The area between the master curve obtained from the frequency sweep test performed on the bitumen sample after aging with RTFOT and the master curve of the unaged sample is associated with aging. It is practically the difference of the areas under both curves. The RAI values of 70/100 and 2% SBS modified bitumen are given in Table 3.

Table 3. RAI values of binder samples

Sample Name	Unaged	RTFOT-aged	RAI
70/100	$1.21x10^6$	$3.03x10^6$	$1.82x10^6$
2% SBS	$1.64x10^6$	$3.37x10^6$	$1.72x10^6$

The RAI value is effective for determining the rheological behavior characteristics of pure and modified bitumen over a wide frequency range. When Table 3 is analyzed, the area under the curve values increased with the increase in G* values with aging. As a result of SBS modification, RAI values were lower than those of the pure binder. The decrease in the area under the master curve indicates a bitumen that is more resistant to aging and reveals the modification effect. Higher RAI values indicate lower aging resistance [25-29]. The results in Table 3 show that SBS additive increases the aging resistance of pure bitumen over a wide frequency range. The complex viscosity values obtained from the frequency scan test of 70/100 % pure and 2% SBS modified bitumen are given in Figure 7.

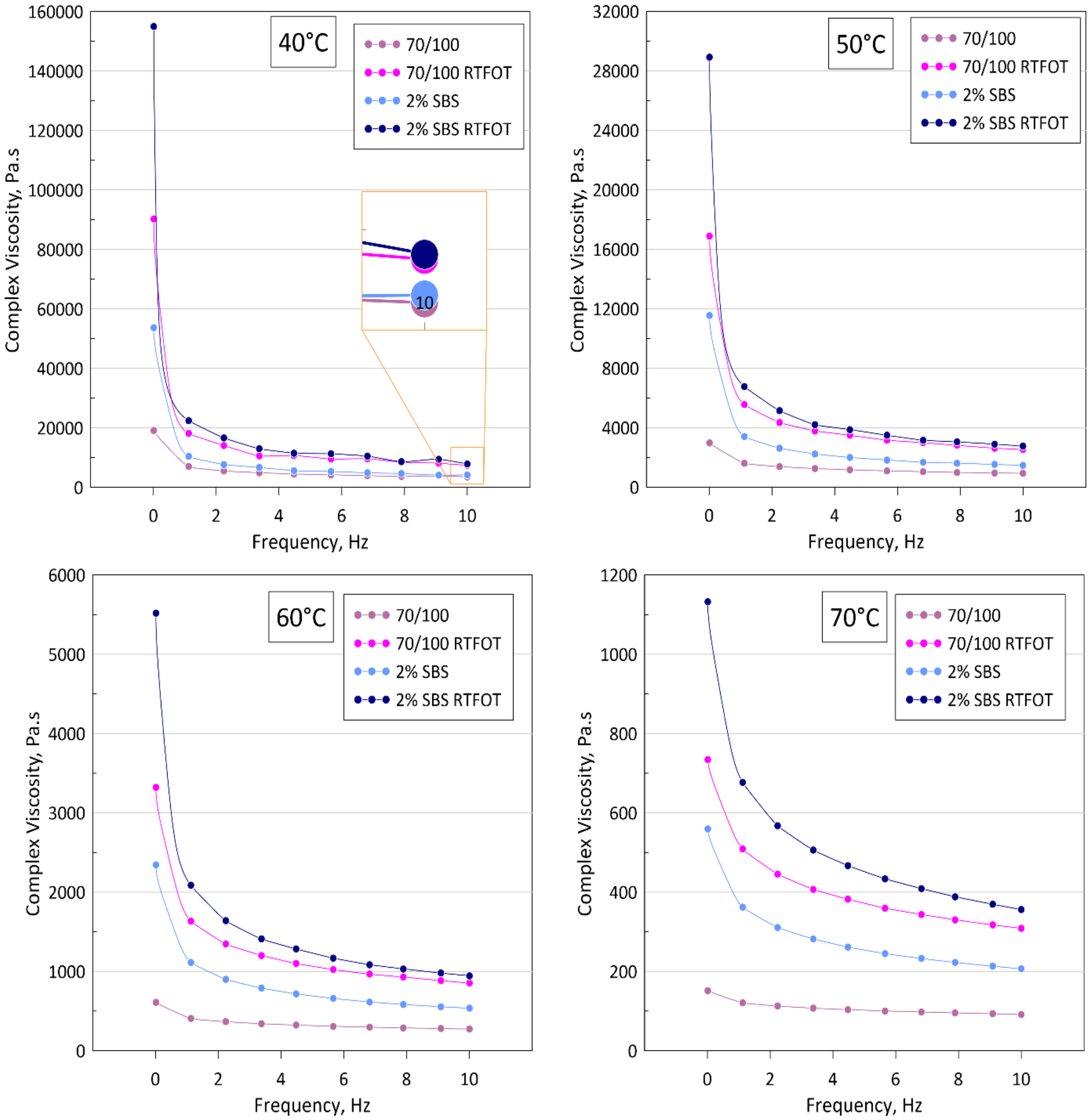

Figure 7. Complex viscosity values of pure and modified bitumen before and after RTFOT

Complex viscosity tends to decrease with increasing temperature. This is associated with bitumen becoming more fluid at higher temperatures. Tests conducted between 40°C and 70°C show that the viscosity decreases as the temperature increases and the bitumen deforms more easily. However, thanks to the SBS modification, this reduction is more limited compared to pure bitumen, because the elastic nature of SBS allows the material to better maintain its shape in the face of temperature. At low frequencies, bitumen exhibits a more viscous and fluid behavior, while at high frequencies elastic properties become dominant. At low frequencies, such as 0.01 Hz, the complex viscosity is higher, while at 10 Hz these values decrease. SBS-modified bitumen better compensates for these frequency differences and increases the elastic behavior, showing better resistance especially at low frequencies. It was observed that the viscosity values of the bitumen increased significantly after RTFOT.

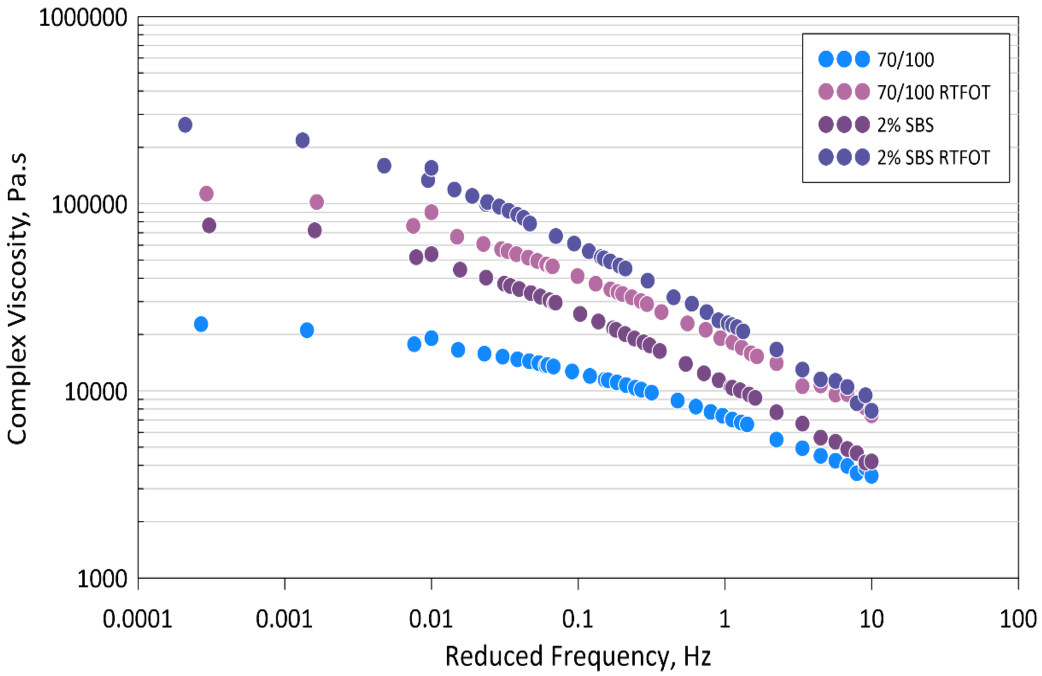

Figure 8. Complex viscosity master curves

In order to observe the rheological behavior at different frequency and temperature values in a single curve, complex viscosity master curves were obtained just like G* values and presented in Figure 8. When Figure 8 is examined, the differences between the bitumens are more pronounced at low frequencies, while the curves get closer to each other as the frequency increases. The highest complex viscosity value was observed in the 2% SBS-RTFOT binder, while the lowest viscosity values were determined in the 70/100 binder. Figure 8 shows that TTSP can be successfully applied to complex viscosity curves.

4. Conclusions

This study examined the short-term aging effects on 70/100 pure and 2% SBS-modified bitumen using RTFOT and frequency sweep tests with a DSR device. The frequency scan test was performed at ten different frequencies (0.01-10 Hz) and 4 different temperatures (40, 50, 60, 70°C). The results obtained from the study are compiled below:

- When the TTSP G* master curves obtained from the test data were analyzed, the results showed that G values significantly increased with aging and SBS modified bitumen presented higher G* values compared to pure bitumen. The master curves are very smooth and continuous.
- The master curves were analyzed using the Christensen-Anderson (CA) Model to conduct detailed rheological evaluations over a wider frequency range. CA Model results showed that rheological index (R) values increased with aging, while ωc values decreased. These effects deepened with the additive and showed that the additive increased the ability of the binder to maintain its elastic properties after aging.
- The Rheological Aging Index (RAI) results showed that the SBS modification increased the aging resistance of 70/100 bitumen. The area under the main curve was found to increase significantly after short-term aging for both binders.
- According to the complex viscosity values, the viscosity values decreased with increasing temperature and were limited by the use of additives. The modification effect, which was more pronounced at low frequencies, lost its effect slightly as the frequency increased. Viscosity values increased significantly with aging.

- Complex viscosity master curves provided the opportunity to analyze the viscosity behavior in a wide frequency range and showed that complex viscosity master curves can be successfully obtained with TTSP.

In this study, 2% SBS additive was selected because it is a widely used concentration in practice and is considered the threshold at which modification effects begin to manifest. This concentration provides an effective balance between performance enhancement and economic feasibility. However, it is acknowledged that the performance of SBS-modified binders can vary with different molecular structures or concentrations, and future research should explore these aspects to broaden the scope of the findings.

5. Author Contribution Statement

In the realized study, Author 1 undertook the idea, design, literature review, experimental study and writing of the article.

6. Ethics Committee Approval and Conflict of Interest

"Ethics committee permission is not required for the prepared article"
"There is no conflict of interest with any person/institution in the prepared article."

7. Ethical Statement Regarding the Use of Artificial Intelligence

No artificial intelligence-based tools or applications were used in the preparation of this study. The entire content of the study was produced by the authors in accordance with scientific research methods and academic ethical principles.

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