

EVALUATION OF RUTTING PERFORMANCE OF NEAT AND MODIFIED BINDERS USING ZERO SHEAR VISCOSITY

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ABSTRACT

Permanent deformation, one of the most common distresses in asphalt pavements, depends substantially on viscoelastic properties of bituminous binders. Resistance to permanent deformation is defined by the elastic portion of complex shear modulus ($G^/\sin\delta$) in Superpave Binder Specification. However, recently revealed insufficiency of this criterion especially in characterization of viscoelastic behavior of polymer modified bitumens (PMBs) led to the new research on alternative testing methods such as zero shear viscosity (ZSV). In this study, ZSV (CEN/TS 15325) and $G^*/\sin\delta$ (EN 14770) using Dynamic Shear Rheometer (DSR) was determined for three different penetration grade base bitumen from two different bitumen sources and those modified with SBS and Elvaloy®RET modifiers at the temperatures of 35°C, 40°C, 45°C, 50°C, 55°C, 60°C. Rutting performance of asphalt mixes prepared with both unmodified and modified binders were evaluated using asphalt concrete specimens tested under repeated creep tests (EN 12697-25) at the temperatures of 40°C and 50°C. The results demonstrate that notwithstanding confirmed difficulty and repeatability problem, ZSV is strongly superior to $G^*/\sin\delta$ value in correlation with permanent deformations especially in PMBs.*

Keywords: Dynamic shear rheometer, zero shear viscosity, rheology, permanent deformation

1. INTRODUCTION

The resistance of hot mix asphalt (HMA) layers to permanent deformations mostly depends on the rheological characteristics of bituminous binders. In this sense, it is very important to know the rheological properties of bitumen and to select the convenient test method for this purpose.

Dynamic Shear Rheometer (DSR) test and the value of $G^*/\sin\delta$ (G^* : complex modulus, δ : phase angle), determined under a oscillated shear stress with the frequency of 10 rad/s (1.6 Hz), are used in Superpave binder specification as a permanent strain performance criteria. It has been mainly accepted in this approach that permanent deformation is controlled by the stiffness and elasticity bituminous binder. While stiffness is presented by complex modulus, phase angle is thought to represent elasticity. In order to resist rutting, a binder should not deform too much and should be able to return to its original shape after load deformation. Therefore, the complex shear modulus elastic portion, $G^*/\sin\delta$ should be large. Intuitively, the higher the G^* value, the stiffer the asphalt binder is, and the lower the δ value, the greater the elastic portion of G^* is.

Nevertheless, at phase angle values higher than 60° , the ratio of $G^*/\sin\delta$ is not very sensitive to the value of $\sin\delta$ in fact. Phase angles of modified bitumens are rarely smaller than 70° ($\sin\delta$ value 0.94), and the value of $\sin\delta$ poorly demonstrates the effect of modification on elasticity, which is the main target. The reason for this is thought as the fact that DSR oscillation test carried out under the short-term loading conditions (1.6 Hz) does not represent the 'delayed elastic strain' behavior modified bitumens generally have (1). Since it has been examined in previous research that there is unsatisfactory correlation between $G^*/\sin\delta$ value of binder and the rutting performance in both field and laboratory, new rheological test methods that could provide better correlation have been a subject of research. In this sense, zero shear viscosity of bituminous binders, which is determined by the creep test carried out by applying a constant load for a while, has been started to use as an alternative evaluation method and in some studies, it has been detected that ZSV value provides better results in determining the permanent strain of modified bitumen recently.

On the other hand, the resistance of hot mix asphalt materials against permanent deformation is conventionally determined by wheel tracking tests. However, thanks to the performance tests which have been developed recently, creep and strain behaviors can be tested by triaxial cyclic pressure test of cylindrical mixture samples which are prepared in gyratory compactor.

In this study, $G^*/\sin\delta$ and zero shear viscosity values of B50/70, B70/100, B160/220 penetration grade bitumens which belong to Kırıkkale refineries and B50/70 grade bitumens which belong to Batman refineries and their bitumens produced with two different polymer additives, SBS and Elvaloy, have been determined and on the asphalt mix samples prepared in the laboratory with the gyratory compactor, permanent deformation test has been conducted by the cyclic pressure test.

2. RHEOLOGY AND ZERO SHEAR VISCOSITY OF BITUMINOUS BINDERS

Rheology is a science concerned with the time-temperature dependent flow and strain characteristics of substances exposed to stress and it is used, in general, for determining the strain characteristics of solids and the flow characteristics of fluids. And the behavior of bituminous binders is dependent on both loading and temperature conditions. While bitumens behave like a viscous fluid under constant loading and hot climate conditions, they behave like an elastic solid under fast loading and cold climate conditions.

Rheologically, while elastic materials show a sudden strain under the effect of external load, strain remains constant as long as the load remains constant. If the load is removed, the material quickly returns to its former shape. When a load is applied to viscous materials, creep deformation occurs by time. When the load is removed, strains cannot be recovered and remain as plastic strain. Bituminous binders generally show viscoelastic behavior demonstrating these two characteristics together. Viscoelastic materials primarily show sudden elastic strain under constant loading, and then time-dependent delayed elastic strain and viscous strain. When the load is removed, in a similar fashion, primarily elastic recovery and then time-dependent delayed elastic recovery occur. Viscous strains cannot be recovered and remain as permanent strain (Figure 1).

In Superpave binder grading system, the delay between the applied stress and strain, which is the phase angle (δ), is used as a criterion of viscoelastic behavior of bitumens. It is accepted that the smaller the phase angle is, the more elastic it is; the bigger the phase angle is, the more viscous it is. On the other hand, the amount of deformation in material under load changes according to the stress intensity to which the substance is exposed, implementation speed and direction and the viscosity of the substance from which the it was produced.

While modulus of elasticity (E) is used as a criterion for the internal resistance of elastic solid substances against strains, the value of viscosity is used as the criterion for the internal resistance of viscous fluids against flow. Viscosity (η), which is defined as the resistance to flow, is considered as the ratio of shear stress to shear rate ($\eta = \tau/\dot{\gamma}$).

In some fluids, shear rate and shear stress are directly proportional; viscosity is constant and does not change with the shear rate. These kinds of fluids are known as Newtonian fluids (ideal fluids). There is a linear relation between shear rate and resistance force in Newtonian fluids. And in non-Newtonian fluids, the value of viscosity changes in different shear rates, that is different viscosity values are obtained for fast loading and slow loading conditions. The fluids, which are not Newtonian, separate into two as pseudoplastic (shear-thinning) and dilatant (shear-thickening) fluids. If the apparent viscosity of the fluid decreases with the increasing shear rate, it is called as pseudoplastic; and if the apparent viscosity (instant viscosity) of the fluid increases with the increasing shear rate, it is called as dilatant. Asphalt is a "thinning fluid" at medium temperatures because its shear rate increases while its viscosity decreases. Some modified asphalts are thickening fluids and some of them are thickening fluids. In thickening fluids, as the shear rate between the layers increases, the viscosity also increases (Figure 2).

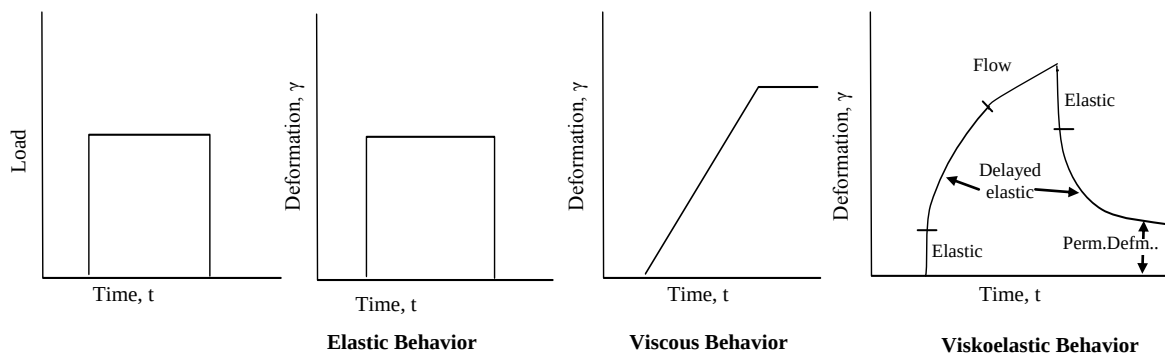


Figure 1: Elastic, viscous and viscoelastic behaviors of the materials

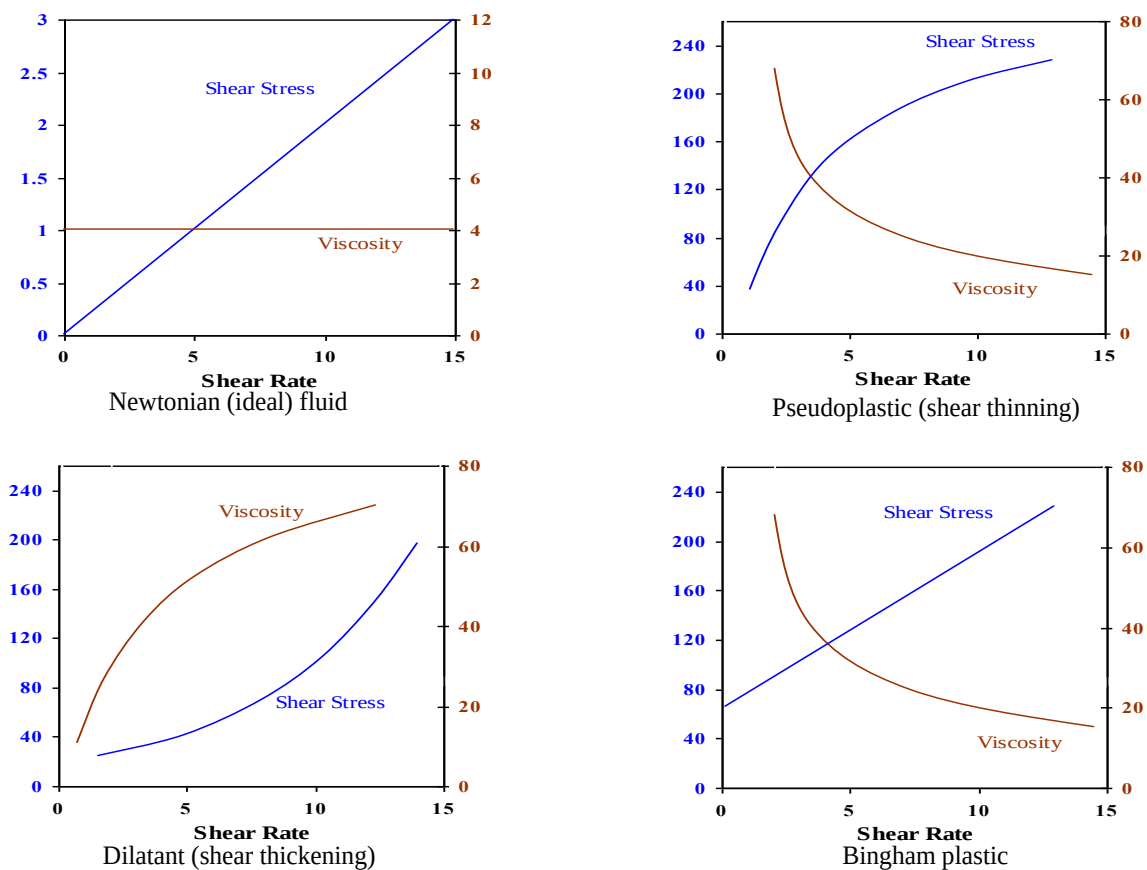


Figure 2: Rheological behaviors of materials [6].

Zero Shear Viscosity (ZSV)

Both normal and modified bitumens generally have non-Newtonian fluid characteristics. The viscosity of non-Newtonian fluids changes as the shear rate changes. On the other hand, all fluids reach to a constant viscosity value at very low and very high shear rate values. These limits are named as 1st Newtonian zone and 2nd Newtonian zone respectively. The change of the pseudo-plastic viscosity value depending on the shear rate of the materials is described in Carreau equation as given below. Figure 3 shows the physical meaning of Carreau's equation [3].

$$\eta_a = \eta_\infty + (\eta_0 - \eta_\infty) \left[1 + (\lambda \dot{\gamma})^2 \right]^{\frac{c-1}{2}} \quad (1)$$

Here; η_a = instant (apparent) viscosity; η_0 = zero shear viscosity; η_∞ = infinite shear viscosity, $\dot{\gamma}$ = shear rate; λ , c = constant. As is seen in figure 3, with a decrease in shear rate, the bitumen viscosity will finally reach a maximized constant value called ZSV, an asymptotic viscosity value when the shear rate approaches zero. when $\lambda \dot{\gamma}$ converge to 0 ($\lambda \dot{\gamma} \rightarrow 0$), η_a also converges to η_0 ($\eta_a \rightarrow \eta_0$).

Due to the viscoelastic behavior of bituminous binders, ZSV can be calculated only after the viscous flow component is separated from the delayed elastic strain component. In creep test, zero shear viscosity can be calculated from the slope of creep curve and/or creep recovery curve from sufficiently long duration creep test.

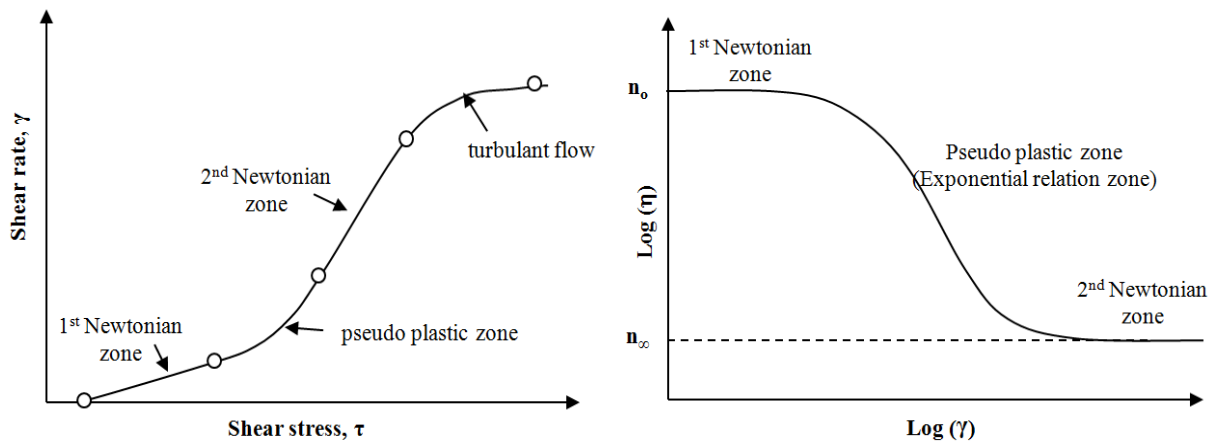


Figure 3: Curves of shear stress verses to shear rate and viscosity versus shear rate (3).

3. EXPERIMENTAL

In this study, by detecting the values of $G^*/\sin\delta$ and zero shear viscosity (ZSV) which belong to different types of bitumens and the permanent strains of the bituminous mixtures prepared by these bitumens, it has been tried to determine which one of these two parameters (DSR and ZSV) gives better results for rutting. For this purpose, 12 bitumens including B50/70, B70/100, B160/220 penetration grade bitumens which belong to Kırıkkale refinery and B50/70 grade bitumens which belong to Batman refinery and their modified bitumens prepared with SBS at the rate of 5% and Elvaloy at the rate of 2% have been used in total. The performance grades (PG) of these bitumens according to Superpave binder grading system were determined beforehand. In this study, $G^*/\sin\delta$ and ZSV values at different temperatures, and permanent strains of these bitumens have been determined by carrying out cyclic compression test on a standard asphalt surface course prepared by these bitumens. The details of the studies conducted are given below.

3.1. Rolling Thin Film Oven Test (RTFOT)

Bitumens used in this study were primarily aged in rolling thin film oven in accordance with the standard EN 12607-1. In this test, 8 bitumen samples of (35±0.5) g in glass container each was placed into a carriage rotating

at a constant rate of rate of $(15,0 \pm 0,2)$ r/min were heated in rolling thin film oven to the temperature of 163°C with a constant supply of air for 75 minutes.

3.2. Dynamic Shear Rheometer Test

$G^*/\sin\delta$ values of the bitumens were determined in accordance with the AASHTO T315 standard by applying a oscillatory shear stress with Dynamic Shear Rheometer (DSR) device in oscillation mode. In the test, 25 mm loading plate were used and spaces of 1 mm were left between DSR loading plates. The test was implemented on the RTFOT aged bitumens at 6 different temperatures of 35°C , 40°C , 45°C , 50°C , 55°C and 60°C . Before each test, the samples reaching to test temperature were kept waiting for 10 minutes until they became steady at desired temperature. Oscillatory shear stress was applied with 10 rad/s (1,6 Hz) frequency. After the test finished, complex modulus ($G^*=\tau/\gamma$), phase angle (δ) and $G^*/\sin\delta$ value were calculated.

3.3. Zero Shear Viscosity Test

Zero Shear Viscosity was carried out at the temperatures of 35°C , 40°C , 45°C , 50°C , 55°C and 60°C using Dynamic Shear Rheometer (Bohlin Gemini) in the creep test mode in accordance with CEN/TS 15325 European Technical Specification. In the test, a constant, torsional creep stress was applied to the binder specimens placed between two parallel plates with the diameter of 25 mm and with the gap of 1 mm for 30 minutes in normal bitumens and 7 hours in polymer modified bitumens which was experienced to be long enough to reach steady-state position. After removal of load, samples were kept waiting for a certain period of time for recovery to compare ZSV in creep mode with that in recovery mode. But only ZSV in creep mode has been used in this paper.

Determination of the constant stress level to be applied in ZSV test is very important. This is generally between 10 Pa and 50 Pa for polymer-modified bitumen. Stress level must be selected so as to remain within the linear viscoelastic range of the binder. As the stress level is inverse proportional to shear rate ($\dot{\gamma}=\tau/\eta$), if the strain rate is too low, the equipment limits can be reached and the viscosity can be scattered, or if the strain rate is too high, the conditions of “zero shear” are not achieved and the integrity of the sample can be threatened.

In light of stress sweep tests, the stress level used in the ZSV tests was selected as 10 Pa for unmodified bitumen and 30 Pa for modified bitumen. On the other hand, because it is required to complete the delayed elastic strain, it is compulsory to continue the test until the steady-state at which this condition is provided in order to obtain a valid result.

	Normal bitumens	Modified bitumens
DSR plate diameter/space between the plates	25 mm/1000 μm	25 mm/1000 μm
Thermal equilibrium time	10 minutes	10 minutes
Shear stress	10 Pa	30 Pa
Creep time	30 minutes	7 hours
Recovery time	30 minutes	1 hour

Table 1: Test parameters used in ZSV tests

Since the actual change of strain is dependent upon the applied stress, it is usual to talk about the compliance rather than the strain. The compliance is defined simply as the ratio of the strain to the applied stress and is denoted by the letter J ($J=\text{strain}/\text{stress}=\gamma/\tau$). By using this notation, creep curves may be directly compared even if they were not measured under the same applied stress.

Time-creep and time-creep compliance graphics can be monitored on the computer automatically during the test. The time-creep compliance curve becomes nearly linear when it reaches to steady-state position. Zero Shear Viscosity (η_0) is calculated from the average of viscosity over the last 15 minutes. This is obtained by dividing the change in time (Δt) by the change in compliance (ΔJ) over the last 15 min (900 s) of the test. This is expressed in mathematical terms as follows:

$$\eta_o = \Delta t / \Delta J = 900 / (J_{\text{end}} - J_{15 \text{ min-before-end}}) \quad (2)$$

Here; the value of $J_{15 \text{ min-before-end}}$ indicates the value of creep compliance 15 minutes before the load is removed. The value of zero shear viscosity can also be calculated similarly via the curve in steady-state obtained at the end of recovery curve. However, only the ZSV value in creep situation was calculated in this study.

3.4. Triaxial Cyclic Compression

In this test, a standard design of asphalt concrete wearing course whose mixture characteristics were determined by designing the mixtures beforehand. The materials used in the mixture are given the table below and these materials were compacted at the height of 12 cm with Superpave gyratory compactor in the molds with the diameter of 10 cm until they reach to the desired density degree. These samples were cut by 1 cm from top and bottom and cylindrical test samples at the diameter of 10 cm and at the height of 10 cm were prepared in total by smoothing the surfaces on which pressure would be applied.

AC Wearing Course Mix Design		Properties of the Aggregates	
Aggregate type	Basalt	Physical properties	Value
Bulk density, g/cm ³	2.473	Los Angles abrasion loss, %	11.7
Optimum bitumen, %	5.25	Soundness (Na ₂ SO ₄), % loss	2
Max.Teo.Density, g/cm ³	2.567	Resistance to stripping, %	30-35
Air void, %	3.66	Polishing stone value (PSV)	53
Voids in mineral aggregates, %	14.6	Flatness index, %	24.5
Voids filled with asphalt, %	75.0	Coarse aggregate specific gravity	2.799
Target gradation		Apparent specific gravity of coarse aggregate	2.894
Sieves	% Passed	Absorption of coarse aggregate, %	1.2
3/4"	100	Fine aggregate specific gravity	2.69
1/2"	90	Apparent specific gravity of fine aggregate	2.737
3/8"	80	Absorption of fine aggregate, %	0.6
No.4	47	Apparent specific gravity of filler	2.743
No.10	33		
No.40	15		
N0.80	9		
N0.200	7		

Table 2: Physical and granulometric properties of aggregates used in triaxial cyclic compression test.

Cyclic compression test was carried out by UTM-100 (Universal Testing Machine with the capacity of 100 kN) test device as per the standard EN 12697-25. The surface of cylindrical samples were coated with membrane and 100 kPa pneumatolitic confining pressure and 300 kPa axial cyclic load with the frequency of 2,5 Hz were applied on these samples. Four HMA samples were prepared for each bitumen. Tests were carried out at two different temperature values; one half of four cylindrical samples produced from each bitumens were tested at 40°C and the ather half were tested at the temperature of 50°C. Each sample were loaded for 15000 times at least. As a result of each drop, strains were recorded by taking the values through the computer from two LVDT on the triaxial cell.

EXPERIMENTS			Kırıkkale Refinery Bitumens									Batman Refinery Bitumens		
Peneration Garde			50/70	70/100	160/220	50/70	70/100	160/220	50/7	70/100	160/220	50/70	50/70	50/70
Polymer Type			-	-	-	SBS	SBS	SBS	Elvaloy	Elvaloy	Elvaloy	-	SBS	Elvaloy
Polimer contetnt			-	-	-	5%	5%	5%	2%	2%	2%	-	5%	2%
Performance Grade			64-22	58-22	52-28	76-16	64-16	64-16	70-22	70-22	64-22	70-22	88-16	88-16
DSR Test	G*/sinδ (kPa)	35 °C	130.3	95.0	69.6	419.5	240.3	174.5	299.0	139.1	121.0	93.0	682.9	208.9
		40 °C	92.8	52.8	28.3	173.3	91.8	92.2	150.9	71.2	57.6	46.3	341.6	107.7
		45 °C	45.8	38.3	12.2	87.5	79.1	47.0	77.8	86.0	36.6	24.7	197.2	62.7
		50 °C	32.0	9.3	5.2	72.6	53.8	26.3	34.9	39.4	18.6	14.2	118.6	35.0
		55 °C	14.5	4.9	2.7	41.4	26.4	14.0	20.0	16.9	10.4	8.9	56.8	20.6
		60 °C	4.9	2.6	1.4	26.9	13.8	7.5	12.2	5.2	6.1	5.7	34.2	13.1
Zero Shear Viscosity (ZSV) (Pa.S)		35 °C	175.2	63.33	19.06	1906	1435	1024	2845	515.1	328.4	1450	4739	4210
		40 °C	16.13	19.74	7.304	1137	805	393	1311	193.1	103.7	883.7	1915	2219
		45 °C	9.773	6.36	2.767	550	210.3	156.9	577.7	101.8	54.47	127.4	850	1220
		50 °C	5.388	1.828	1.007	384.8	104.3	56.08	191.8	62.32	24.74	71.49	536.6	721
		55 °C	2.041	0.7633	0.4309	131.1	79.01	35.25	76.39	23.45	10.97	16.02	352.1	285.9
		60 °C	1.038	0.5234	0.1782	74.33	37.77	12.38	27.25	12.38	4.4	7.156	171.2	66.26
Cyclic compression test	Minimum strain rate	40 °C	0.0795	0.1940	0.0750	0.0060	0.0380	0.1750	0.0030	0.2410	0.0110	0.0175	0.0065	0.0175
		50 °C	0.1130	0.2560	0.1690	0.1315	0.1065	0.3485	0.0035	0.1500	0.0470	0.0220	0.0470	0.0160
	Number of loading at min. strain rate	40 °C	24144	37312	28192	15232	16608	19728	24576	14960	30736	21856	28608	27216
		50 °C	21632	20064	28336	12160	16896	19664	28512	27552	31088	28976	28752	28480
	Strain at min. strain rate, mm	40 °C	1.247	3.636	2.862	0.279	0.480	0.804	0.207	0.814	0.310	0.399	0.335	0.276
		50 °C	2.146	2.529	3.590	0.381	0.518	1.111	0.386	1.897	0.555	0.617	0.395	0.308
	Deformation at the end of 10000 th loading, mm	40 °C	1.254	1.760	2.175	0.279	0.463	0.697	0.207	0.766	0.295	0.391	0.332	0.263
		50 °C	1.893	2.037	2.454	0.422	0.546	0.840	0.386	1.524	0.503	0.580	0.362	0.292

Table 3: Pivottable showing the test results

4. EVALUATION OF TEST RESULTS

The summary results of the test carried out are given in Table 3. As is seen on the G*/sinδ-strain and ZSV-strain graphics drawn according to the test results, the value of ZSV provides a higher degree correlation with the occurred permanent strains (Figure 4). The best correlation is obtained exponentially and it complies with the pseudoplastic behavior model given in Figure 3. In this sense, it can be said that the value of zero shear viscosity represents the viscoelastic behavior of the bitumen better.

At the same time, as is seen on Figure 5, permanent strains occurring on modified mixtures doped with SBS are generally higher than the ones occurring on modified mixtures doped with Elvaloy. Although this

situation shows compliance on the values of ZSV, it has been seen that modified bitumens doped with SBS have provided better results on the contrary, when compared in terms of $G^*/\sin\delta$ test (Figures 6 and 7).

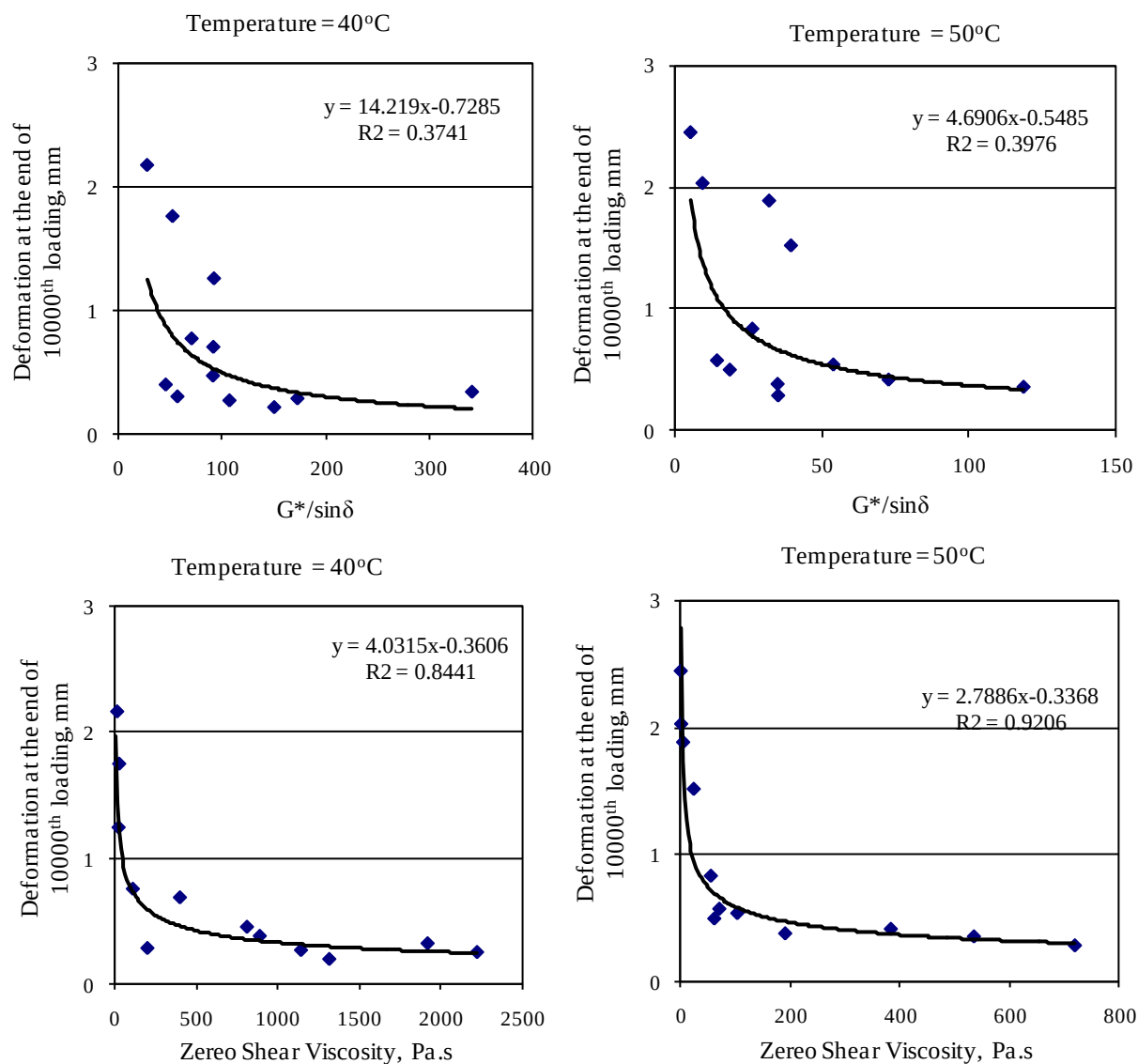


Figure 4: The graphics of ZSV versus deformation and $G^*/\sin\delta$ versus deformation at 40°C and 50°C

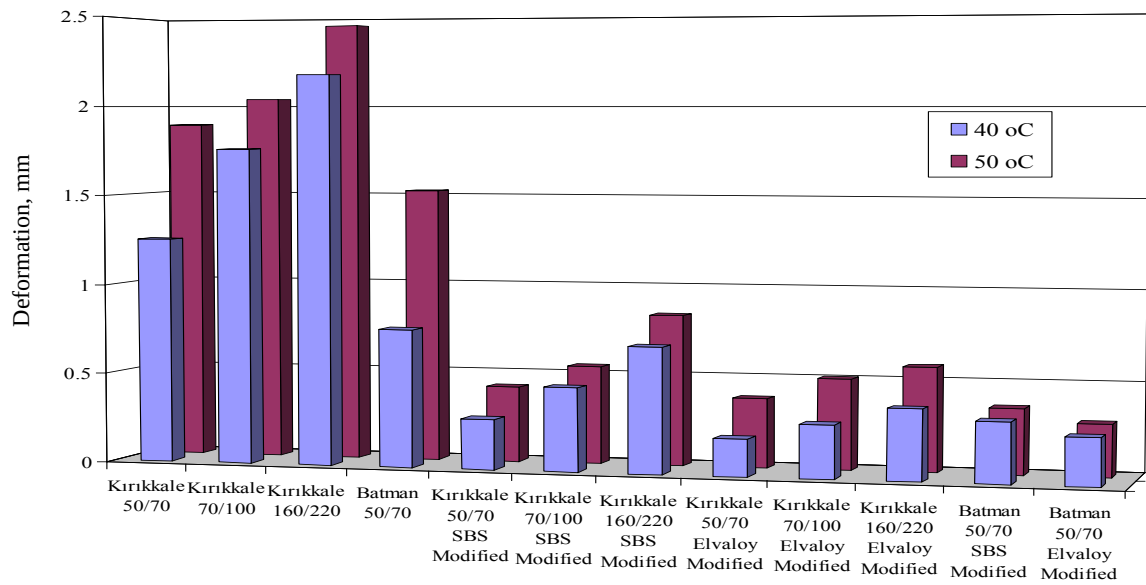


Figure 5: Graph showing the permanent deformation test results of HMA samples.

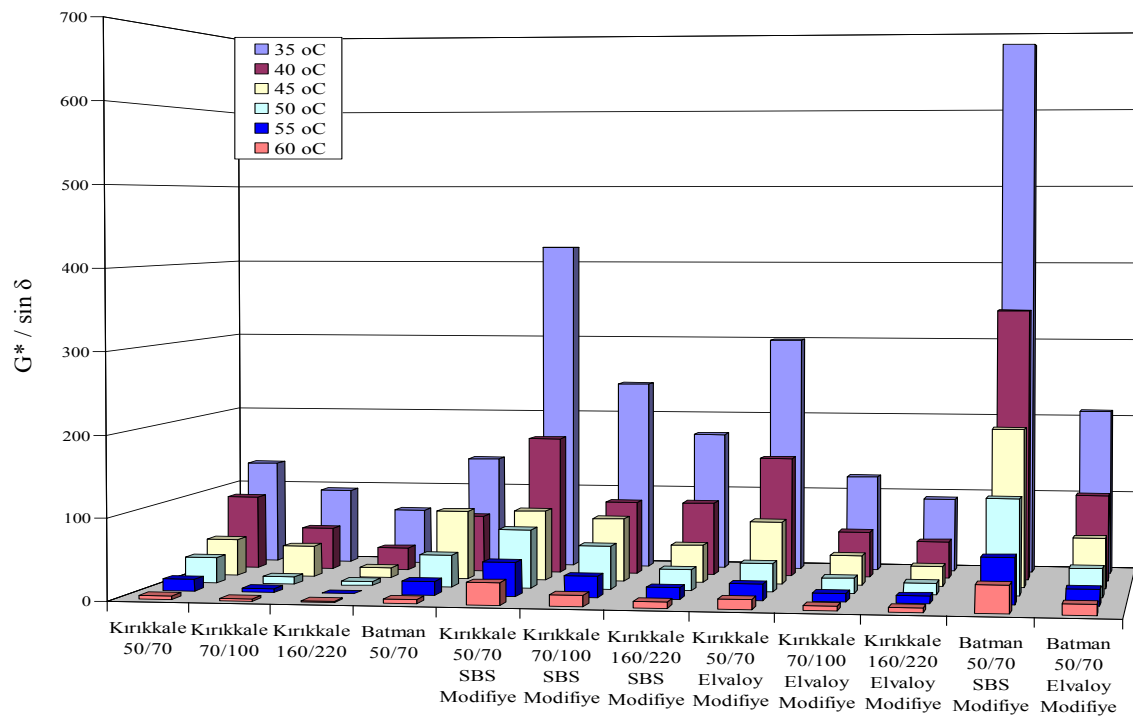


Figure 6: Graph showing the $G^*/\sin\delta$ values of binders

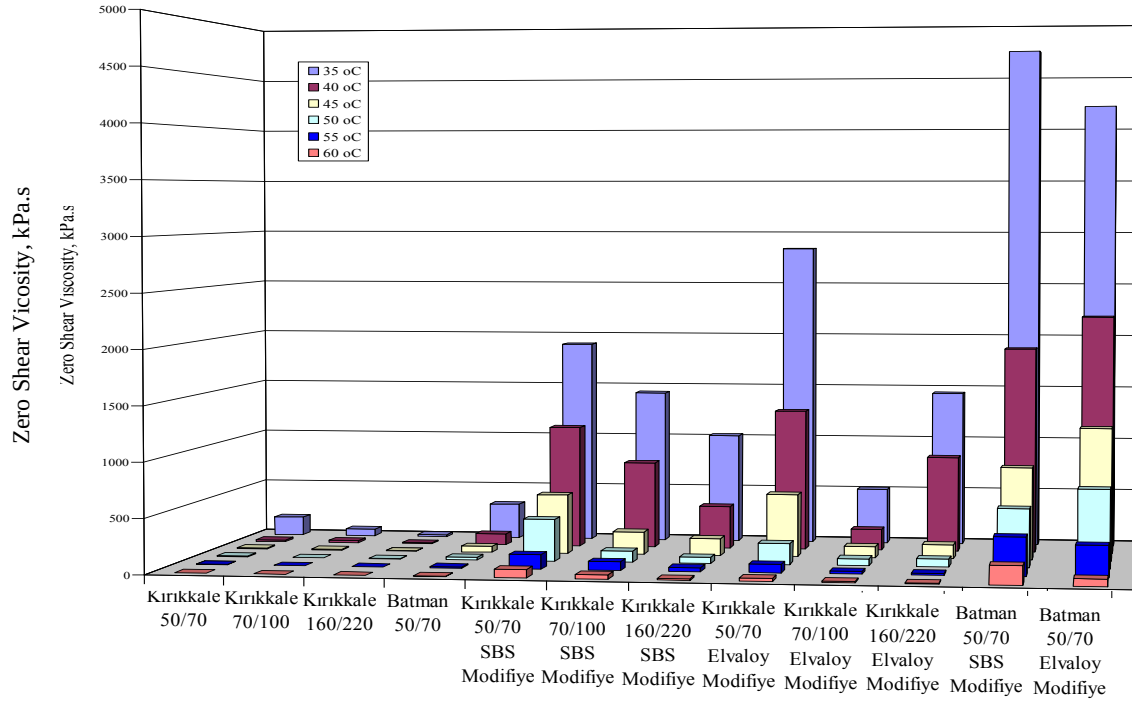


Figure 7: Graph showing the zero shear viscosity values of binders

5. CONCLUSIONS

In this study, the values of $G^*/\sin\delta$ and zero shear viscosity (ZSV) of B50/70, B70/100, B160/220 penetration grade bitumens of Kırıkkale refinery and B50/70 grade bitumens of Batman refinery and their modified bitumens produced with SBS and Elvaloy type polymers have been determined at different temperatures; asphalt concrete wearing course samples mixture samples have been prepared for each bitumen and their permanent strains have been found by cyclic pressure test.

According to the test results; it was ascertained that the correlation between ZSV and permanent strains ($R^2=84\%$ at 40°C ; $R^2=92\%$ at 50°C) is much higher than the correlation between $G^*/\sin\delta$ and permanent strains. It was explicitly seen that ZSV better represents the viscoelastic behavior of PMBs than DSR test. On the other hand, the ZSV tests especially on modified bitumens takes too much time, sometimes 8-10 hours and even more, to reach steady-state where the creep curve becomes linear, and even if the samples are kept waiting for this period, steady state cannot be guaranteed at all times and the material may fail before it reaches to steady-state. In this sense, although the value of ZSV provides better correlation with permanent deformations, a great number of diverse results may be obtained.

Even though there are many research in literature about zero shear viscosity, it has been scarcely compared with $G^*/\sin\delta$ using cyclic compression asphalt mixture tests and scarcely documented its superiority over $G^*/\sin\delta$ in a clarity given in this paper. Thus, this paper is thought to made a significant contribution to research in this field.

However, although the value of ZSV can be used for determining the resistance of bituminous binders to permanent deformation, due to its difficulty in performing, there needs to be deeply studied alternative testing methods like frequency sweep test. Furthermore, the Multiple Stress Creep Recovery (MSCR) test which is the actual research topic and has been incorporated into AASHTO Performance-Graded Binder specification (AASHTO MP 19) needs to be compared with ZSV in terms of rutting performance.

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