Research Article GU J Sci, Part A, 12(3): 691-705 (2025) 10.54287/gujsa.1633786

Gazi University
Journal of Science
PART A: ENGINEERING AND INNOVATION
http://dergipark.org.tr/gujsa

Performance Evaluation of Bitumen Modified with Thermo-Elastic Polymer

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Keywords
Modification
Polymer
Elvaloy®
Physical Properties
Rheology

Abstract
The increased traffic flow, vehicle loads, and the impacts of harsh climatic conditions necessitate more enduring and stable asphalt pavements. This requires the use of good-quality aggregates and the modification of bitumen with polymers to make pavement more resistant against heavy traffic and environmental conditions. Researchers have focused on using one of common thermo-elastic polymer called Elvaloy Reactive Elastomeric Terpolymer (Elvaloy®) to modify different Pen-Grade bitumen by using up to 2% by weight of bitumen in 0.5% increments. For performance measurement at high and intermediate temperatures, physical tests and rheological tests were used. In terms of physical and test results, significant decrease and increase due to test methods. Rutting and fatigue resistance performance under high temperatures differs as the rate of included the polymer increase. In general, the results show that as the Elvaloy® additive content increases, each sample improves at different rates and hence bitumen with different pen-grades are compatible with the additive.

Cite
Gökalp, İ. (2025). Performance Evaluation of Bitumen Modified with Thermo-Elastic Polymer. *GU J Sci, Part A*, *12*(3), 691-705. doi:10.54287/gujsa.1633786

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Article Process

Submission Date	05.02.2025
Revision Date	25.03.2025
Accepted Date	28.07.2025
Published Date	30.09.2025

1. INTRODUCTION

The materials constituting flexible pavements consist of 93-95% aggregates and 5-7% bituminous binders and additives by mass. The function of a binder is to connect the aggregate particles together by ensuring adhesion and to eliminate aggregate dispersion/segregation under repeated and varied traffic loads during construction of flexible pavement (Al-Taher, et al., 2024). Aggregates, on the other hand, provide intrinsic friction and volumetric stability within the mixture. Although the weight proportion of bituminous binders in the mix is lower, their effect on pavement performance is significant Sengoz et al. (2014). Bitumen is a valuable construction material left behind at the end of crude oil refining processes. Therefore, a series of changes are made in the process of oil refinery production parameters and techniques. The technology applied and the new production parameters used cause a significant change (either positively or negatively) in the engineering properties of the bitumen (Pipintakos et al., 2024).

Conventional bitumen is modified when it has insufficient properties for one or more purposes. The general purposes of modifying bitumen can be listed as follows; (1) To reduce viscosity, thus improving the

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workability and compaction of mixtures during construction, maintenance and repair, (2) To improve rheological properties of the mixture under different temperature from high to low; (3) To enhance adhesion between bitumen and aggregate in pavements; (4) To increase the resistance to aging and oxidation, and to provide regeneration/rejuvenation of aged bituminous binders; and finally (5) To strengthen the stability of mixtures, etc. (Rossi et al., 2015; Oner & Sengoz, 2016; Wu et al., 2024). If bitumen modification is not carried out properly, binders with the desired properties may not be obtained, hot mixes produced with modified bituminous binders may be significantly inappropriate, and the use of such hot mixes may lead to the formation of a road pavement network that does not have a long service life and requires frequent maintenance and repair, which does not provide cost effectiveness (Wilde et al., 2014; Babashamsi et al., 2016; Frangopol & Liu, 2019; Llopis-Castelló et al., 2020). Moreover, defects in road pavement increase road safety risks reduce driving comfort and cause death and/or injury in the event of traffic accidents. In this sense, significant financial and moral losses are inevitable (Kuliczkowska, 2016; Chand et al., 2021). Therefore, it has become a must for asphalt producers to supply a structurally acceptable quality/durable bituminous binder for flexible pavements.

Due to the natural structure of crude oil, the properties of the virgin bitumen often do not provide the required performance under increasing traffic loads and adverse climatic conditions. Not desired properties of bitumen may necessitate improving and this brings the modification of bitumen. Regulations are being made within the national and international standards in order to increase the quality (Zhu et al., 2014; Weigel & Stephan, 2017; Porto et al., 2019). Modifying additives may be classified into four groups according to their functions: (a) Adhesion enhancer, (b) Plasticizer, (c) Structurizer and (d) Other complex additives. Under the framework of these four groups, studies have been carried out to modify bituminous binders using various additives, polymers or valuable wastes (Belyaev et al., 2021; Wong et al., 2022; Jwaida et al., 2023; Rashid et al., 2024). The scope of the study is limited to a thermo-elastic product that is widely used and commercially produced from polymers.

Information on polymers used in the modification of bitumen binders is given briefly within the scope of this study. A polymer modified bitumen (PMB) is produced with mechanical mixing of base bitumen and polymer and/or a chemical reaction (Giavarini, 1994; Geçkil, 2019). Successful modification depends on many parameters. As a result of extensive research the parameters to be considered for a successful and purposeful PMB can be listed as follows. Chemical structure of bitumen, polymer and different type of them (Yousefi, 2003; Navarro et al., 2009), polymer/bitumen ratio (García-Morales et al., 2006) polymer particle size (Maharaj et al., 2015), and PMB production/processing conditions (Pérez-Lepe et al., 2003). Overall, modifying bitumen with the correct polymer at the appropriate temperature, mixing speed and time is of great importance for the pavement. Without taking these parameters into consideration, polymer modified bitumen and the pavement on which it is used will not be long-lasting and will return to the relevant institutions as a significant economic loss (Plati, 2019).

In this study, base bitumen with different penetrations were modified up to 2 % of Elvaloy® polymer, a thermo-elastic product by bitumen weight with 0.5% increments and their performances were evaluated by various physical and rheological tests for enhancing resistance performance of bitumen samples used in the construction of flexible pavements under different temperatures from high to low. The test including penetration, softening, and viscosity tests as physical ones, and dynamic shear resistance test as rheological ones were applied and the results were reported.

2. MATERIAL AND METHOD

2.1. Materials

Three different types of base bitumen in different Pen-Grades (PG), which are 50-70; 70-100; 100-150 were utilized for modification with Elvaloy Reactive Ethylene Terpolymer (Elvaloy®) in different rates such as 0.5, 1, 1.5, and 2 % by the weight of bitumen. The polymer was obtained from the company called KOMSA Engineering Construction Industry Trade Inc. and the bitumen samples were supplied from Batman refineries in the country with the cooperation of Highways 9th Regional Directorate 97th Branch (Batman) Chiefdom, Türkiye. To distinguish the result of samples with each other the following sample codes were labeled in Table 1.

Table 1. *Sample codes*

Samples	Codes
Base Bitumen with PG 50/70	BB1
Base Bitumen with PG 70/100	BB2
Base Bitumen with PG 100/150	BB3
Short-Term Aging for BBX	S-BBX
Long-Term Aging for BBX	L-BBX
Elvaloy Reactive Ethylene Terpolymer in different rate	EY
Bitumen with PG 50/70 - 70/100 - 100/150 modified with E® in different rate	MBX-EY
Short-Term Aging for bitumen with modified with E in different rate	S-MBX-EY
Long-Term Aging for bitumen with modified with E in different rate	L-MBX-EY
Where, BB refers to base bitumen, MB refers to modified bitumen S refers to short, L refers to long X refers to 1, 2, and 3 Y refers to 0.5, 1, 1.5, and 2 %	

The polymer has been donated by KOMSA Engineering Construction Industry Trade Inc. to be used in the present and future studies. The properties of the polymer sample that was presented by Sengoz et al., 2017 are given in Table 2 and the properties of base bitumen which were are provided by the Highways 9th Regional Directorate (Diyarbakir) and 97th Branch Chiefdom (Batman), Türkiye is given in Table 3.

Table 2. *Properties of Elvaloy®*

Properties	Values	Standards
Density	0.94 g/cm^3	ASTM D792
Bulk Density	0.58 g/cm^3	ASTM D2726
Melt Flow Rate (190 °C/2.16 kg)	8 g/10m	ASTM D1238
Melting Point (DSC)	72 °C	ASTM D3417
Maximum Processing Temperature	280 °C	-

Table 3. *Properties bitumen samples*

Properties		BB1	BB2	BB3	Standard
Penetration (dmm)		56.4	73.8	129.0	ASTM D5
Softening Point (°C)		46.8	44.3	39.0	ASTM D36
Penetration Index		-1.79	-1.88	-2.18	-
Viscosity (cP)	@ 135 (°C)	698.3	563.3	307.5	ASTM D-4402
	@ 165 (°C)	180.0	160.0	95.8	
Mixing Temperature @ 170±20 cp (°C)		166.7-164.4	165.7-162.8	157.3-151.7	
Compaction Temperature @ 280±30 cp (°C)		160.9-157.5	158.3-153.8	143.1-134.6	
Flashing Point (°C)		315	305	285	EN ISO 2592
Ductility @ 10 °C (cm)		55	76	84	EN 13398
**American Society for Testing and Materials (ASTM), International Organization for Standardization (ISO), European (EN)*					

2.2. Methods

The test including penetration, softening, and viscosity tests an penetration index as physical ones under physical test methods and dynamic shear resistance test and aging of samples under rheological ones and lastly bitumen modification procedure were identified in brief.

2.2.1. Physical Test Methods

Three physical in other words conventional test methods were applied on the samples which are penetration softening point and viscosity. At the following the test methods were briefly indicated.

Penetration test method: To determine the bitumen consistency under constant load, time and temperature, the test method is applied on the samples. In this study, ASTM D5/D5M-20 (2020) standard was conducted.

Softening test method: The test is used to determine the heating temperature of bitumen and to allow bitumen to flow without compromising its chemistry. ASTM D36/D36M-14 (2020) standard was conducted.

Penetration index: As it is known that bitumen is sensitive to heat and heat sensitivity can be calculated in numerous ways. One method is to calculate the heat sensitivity index bitumen permeability (Penetration index, PI). What this index means, in summary, is that larger PI values are correlated with smaller degrees of temperature sensitivity of the bitumen. The determination of PI is a function of penetration and softening measurements and obtained from the following equations (1 and 2).

$$PI = \frac{20 - 500A}{1 + 50A} \tag{1}$$

$$A = \frac{log800 - \log{(Pen@25°C)}}{T_{R\&B} - 25\ °C} \tag{2}$$

Where;

Pen @ 25 °C is penetration values at 25 °C,

$T_{R\&B}$ is softening point temperature

Viscosity test method: It is conducted so as to evaluate the adequate heat temperature for mixing and compaction processes in both the lab and field condition. ASTM D4402/D4402M-23 (2023) standard was conducted at 135 °C and 165 °C temperatures.

2.2.2. Rheological Test Methods

Three rheological test methods were applied on the samples. These are: Dynamic shear rheometer, rolling thin film oven, and pressure aging vessel. At the following the test methods were briefly indicated.

Dynamic shear rheometer test (DSR): The DSR makes it possible to obtain deep insights under different temperatures and loading conditions in case of bitumen in different aged state. The results give an insight about rutting and fatigue resistance of the samples. ASTM D7175-23 (2024) standard was conducted.

Aging methods: Short-term process represents the aging period from production to the compaction of the pavement, while long-term process represents aging period until the end of the service life of the pavement. It is feasible to model both aging processes of bitumen under laboratory conditions. Rolling thin film oven (RTFO) is the test method to obtain short term aged samples. ASTM D2872-22 (2022) standard was conducted for RTFO test. Pressure aging vessel (PAV) is the way to obtain long term aged samples. ASTM D6521-22 (2022) standard was conducted for PAV test.

2.2.3. Bitumen Modification Procedure

The samples used in the study have been prepared by taking into consideration both the preliminary studies of the laboratory, the studies in the existing literature and the production / processing conditions of the manufacturer. The following procedure was conducted to modify bitumen modify with Elvaloy®).

The bitumen with additive polymer codded with “E” in different rate by mass, 1 kg base bitumen heated at 160ºC for 2-2.5, 1-1.5 and 1 hours according to BB1, BB2, and BB3, respectively, in order to make them fluidized. During heating base bitumen, the different proportions of “E” polymers were weighed, labeled and packaged. In order to ensure homogeneity in terms of heat and molecular distribution in bitumen at the specified temperatures, the bitumen was mixed at 100 rpm for 30 minutes with propeller mixer. The “E” polymer additive was poured into the hot bitumen at the mentioned rates and mixed at 1000 rpm for the specified time periods as 6, 5, and 4 hours according to BB1, BB2, and BB3, respectively. Before testing, one more stirring was done at the determined heat temperature with 100 rpm for nearly 30 min to make homogeneous. Therefore, the MBX-EY samples were performed on at least two samples.

3. RESULTS AND DISCUSSION

The results were given for base and modified samples in each aging condition in case of physical and rheological tests at the following Tables.

3.1. Physical Test Results

The result for unaged form of bitumen samples will be seen in Table 4. There is a significant reduction in penetration, rising in softening point, and PI values with the increasing rate of additive. The higher PI values observed in bitumen modified with the higher additive content may be linked to reduction in the temperature sensitivity of the bitumen. In line with these results, an increase in viscosity values is observed.

The result for short-term aging form of bitumen samples will be seen in Table 5. There is a significant reduction in penetration, rising in softening point and in PI values while additive rates increases. The aging effect together with the additive resulted in higher PI values, provoking a further reduction in the temperature sensitivity of the bitumen. In line with these results and the short term and modified based hardening of the bitumen, an increase in viscosity values is observed. Moreover, the mass loss can be evaluated after short-term aging, and the result showed that there is a decrease in the mass losses as additive rate increases.

The result for long-term aging form of bitumen samples will be seen in Table 6. there is a significant reduction in penetration, rising in softening point and PI values as rate of additive increases. Similar to the other data, the result showed that the higher PI values in positive manner observed in bitumen modified with the higher additive content were linked to certain reduction in the temperature sensitivity of the bitumen. The long-term aging effect together with the additive resulted in higher PI values making a further reduction in the temperature sensitivity. In line with these results and the hardening of the bitumen, higher viscosity values are observed.

Table 4. *Results of bitumen samples in unaged form*

Sample ID	Penetration (dmm)	Softening Point (°C)	Penetration Index	Viscosity (cP)	
				@ 135 °C	@ 165 °C
BB1	**56.4**	**46.8**	**-1.79**	**698.3**	**180.0**
MB1-E0.5	55.6	48.0	-1.47	803.3	195.0
MB1-E1	55.0	49.4	-1.13	908.3	217.5
MB1-E1.5	54.5	51.2	-0.70	1017	255.0
MB1-E2	53.0	51.4	-0.72	1237	549.2
BB2	**73.8**	**44.3**	**-1.85**	**563.3**	**160.0**
MB2-E0.5	69.0	47.0	-1.22	662.5	170.0
MB2-E1	68.4	47.7	-1.05	664.2	192.5
MB2-E1.5	67.9	48.4	-0.88	666.7	197.5
MB2-E2	67.1	48.8	-0.80	717.5	286.7
BB3	**129.0**	**39.0**	**-2.17**	**307.5**	**95.83**
MB3-E0.5	121.1	37.8	-2.83	297.5	92.5
MB3-E1	119.5	39.0	-2.40	288.3	87.5
MB3-E1.5	118.3	41.8	-1.36	383.3	120.0
MB3-E2	116.5	42.5	-1.15	395.0	150.0

Table 5. *Results of bitumen samples in short-term aged form*

Sample ID	Penetration (dmm)	Softening Point (°C)	Penetration Index	Mass Loss (%)	Viscosity (cP)	
					@ 135 °C	@ 165 °C
S-BB1	**32.3**	**57.0**	**-0.56**	**0.68**	**1495.0**	**300.0**
S-MB1-E0.5	30.0	58.6	-0.39	0.70	1770.7	398.0
S-MB1-E1	28.1	60.1	-0.23	0.72	2189.4	560.7
S-MB1-E1.5	26.4	61.0	-0.19	0.77	2780.0	730.0
S-MB1-E2	25.9	62.0	-0.04	0.88	3590.0	1079.0
S-BB2	**36.3**	**55.5**	-0.63	**0.57**	**1328**	**287.5**
S-MB2-E0.5	35.8	57.2	-0.31	0.60	1604.6	356.5
S-MB2-E1	34.2	57.8	-0.28	0.63	1845.0	407.8
S-MB2-E1.5	32.9	59.0	-0.13	0.67	2398.0	505.0
S-MB2-E2	31.7	60.5	0.09	0.87	3139.0	617.5
S-BB3	**64.8**	**44.8**	-2.01	**0.51**	**505**	**212.5**
S-MB3-E0.5	63.7	46.3	-1.62	0.55	792.6	276.8
S-MB3-E1	62.1	47.5	-1.35	0.59	908.1	331.3
S-MB3-E1.5	59.8	50.1	-0.75	0.65	1225.8	408.4
S-MB3-E2	59.5	51.0	-0.54	0.78	1419.9	473.6

Table 6. *Results of bitumen samples in long-term aged form*

Sample ID	Penetration (dmm)	Softening Point (°C)	Penetration Index	Viscosity (cP)	
				@ 135 °C	@ 165 °C
L-BB1	**22.7**	**61**	-0.47	**5999**	**811.7**
L-MB1-E0.5	22	62.4	-0.28	6360	860.4
L-MB1-E1	20.6	63.2	-0.26	7250.4	980.9
L-MB1-E1.5	17.1	65.3	-0.23	8555	1157.5
L-MB1-E2	16.3	66.1	-0.19	10000.9	1357.7
L-BB2	37.1	51.3	-1.51	**4080**	**747.5**
L-MB2-E0.5	36.9	52.3	-1.30	4435.6	899.3
L-MB2-E1	34.6	54.1	-1.03	4698.1	929.2
L-MB2-E1.5	32.2	55.3	-0.92	5723.8	998.9
L-MB2-E2	30.4	56.8	-0.72	7074.2	1256.2
L-BB3	**42.1**	**53**	-0.86	**1076.7**	**313.0**
L-MB3-E0.5	41.5	53.6	-0.76	1425.3	375.5
L-MB3-E1	40.1	54.3	-0.68	1581.3	423.3
L-MB3-E1.5	37.5	55.6	-0.54	1624.4	513.6
L-MB3-E2	36.8	56.3	-0.43	1877.6	566.7

3.2. Rheological Test Results

The result for unaged form of bitumen samples will be seen in Figure 1. It is shown that the failure temperature value (where G*/Sin δ° is below 1000 Pa) of the BB1 sample with low penetration is the same as BB2, but it is superior in value, but it is considerably higher than the BB3 value. The increase in the failure temperature values of the modified bitumen formed by adding polymer additives to the bitumen at increasing rates is seen in all three bitumen types. The failure temperatures of the unaged samples between 71.51 and 81.92 °C, 68.88 and 75.91 °C, and 63.02 and 67.80 ° for BB1, BB2 and BB3-based samples, respectively.

The result for short-term aging form of bitumen samples will be seen in Figure 2. There is a normal increase in the values due to the hardness that occurs with aging. In addition to short-term aging, there was a change in the failure temperatures according to the compatibility of each bitumen sample according to the additive rate. The increase in failure temperatures (where G*/Sin δ° is below 2200 Pa) is an indication that the resistance of the bituminous binder to the rutting increases with the addition of additives in increasing rates. The failure temperatures of the short-term aged samples are between 75.84 and 84.94 °C; 75.71 and 79.31 °C, and 64.7 and 68.46 °C for S-BB1, S-BB2 and S-BB3-based samples, respectively.

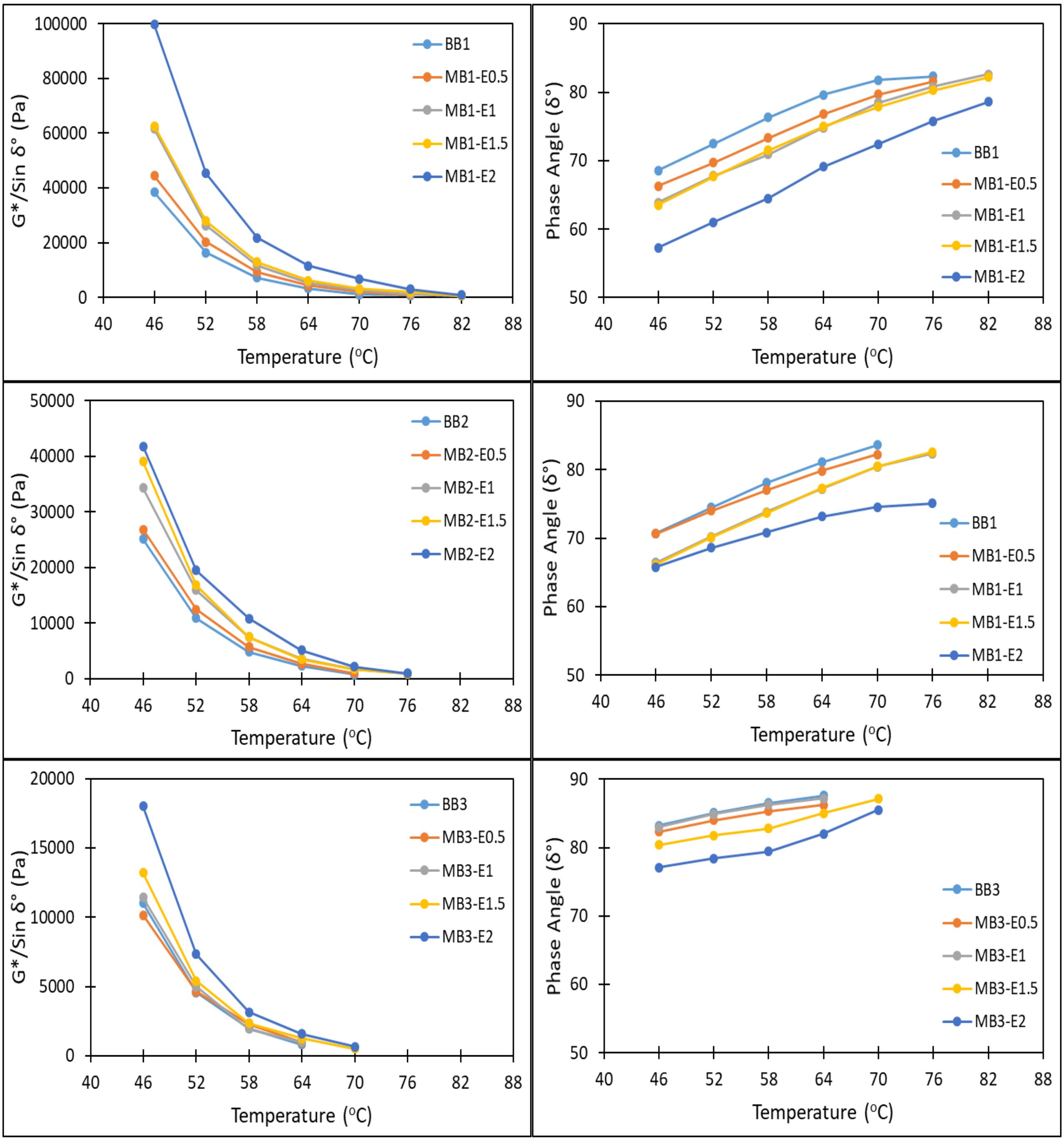

Figure 1. *DSR results of bitumen samples in unaged form*

The result for long-term aging form of bitumen samples will be seen in Figure 3. There is a normal increase in the values due to the hardness that occurs with aging as temperature decreases. There were significant decrease failure temperatures according to the compatibility of each bitumen sample according to the additive rate. The decrease in failure temperatures (where G*. Sin δ° is above 5000 kPa) indicates that the resistance of the bituminous binder to the fatigue increases with the addition of additives in increasing rates. The failure temperatures of the long-term aged samples are between 22.12 and 24.82 °C; 21.16 and 26.97 °C, and 18.91 and 24.01 °C. for L-BB1, L-BB2 and L-BB3-based samples, respectively.

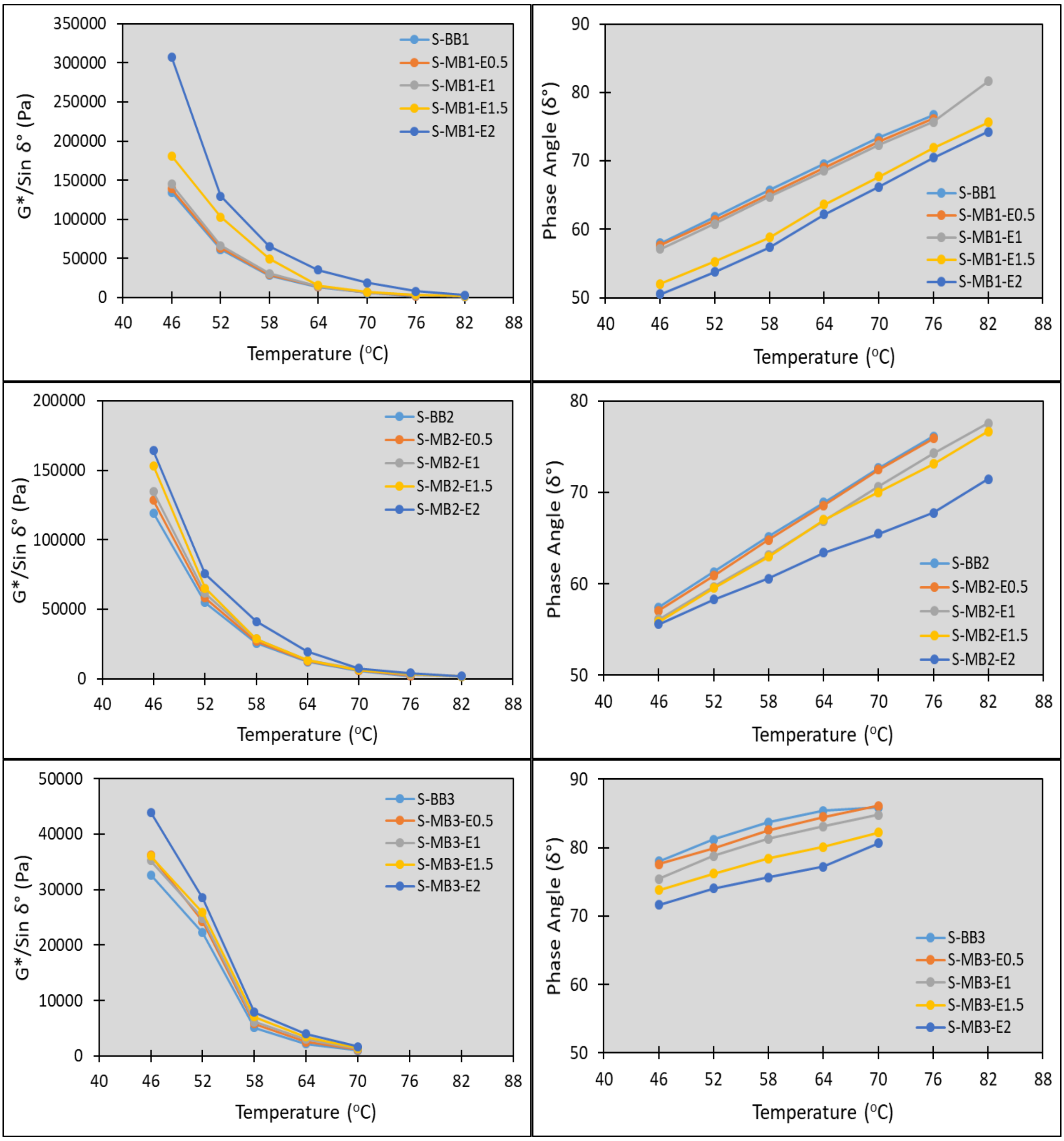

Figure 2. *DSR results of bitumen samples in short-term aging form*

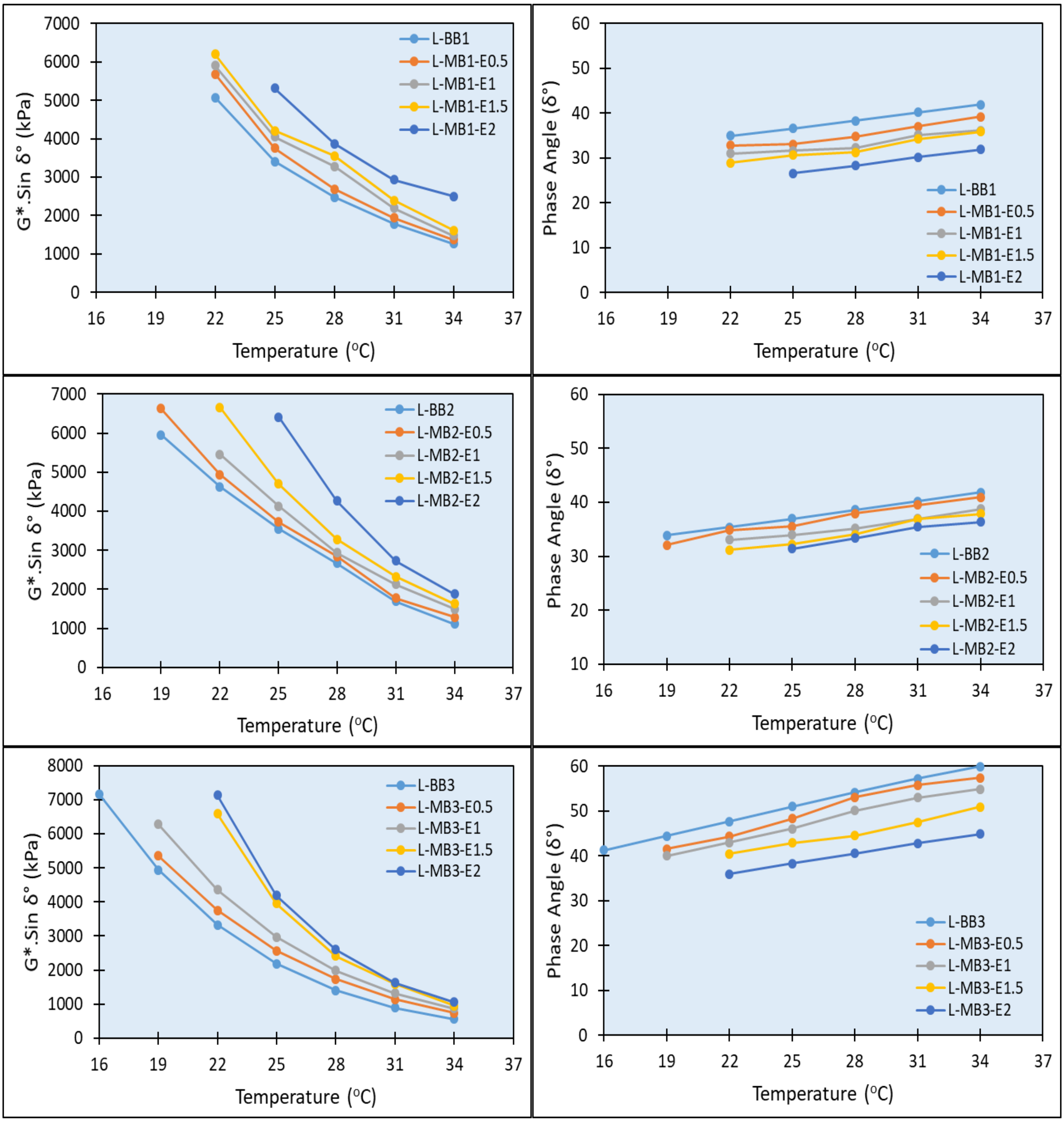

Figure 3. *DSR results of bitumen samples in long-term aging form*

4. CONCLUSION

To investigate the effect of Elvaloy®, one of thermos plastic polymer, on bitumen having different Pen-Grade for modification done with the rate 0.5 to 2.0 % by mass with 0.5% increments, this study was established. Numerous physical and rheological tests and analyses were conducted. Consequently, the followings can be highlighted.

• The penetration test result showed that strength of the bitumen increases with modification.

• The softening point test result showed that heating temperature of the bitumen increases with modification.

• Penetration index indicated that temperature susceptibility of the bitumen decreases with modification.

• Viscosity test results showed that the bitumen's resistance to flow decreases with modification.

• Mass loss results indicated that the bitumen becomes less flexible with modification.

• DSR result determined on unaged and short-term aging samples showed that rutting resistance of modified bitumen samples increases as the rate of additive increases.

• DSR result determined on long-term aging samples showed that fatigue resistance of modified bitumen samples decreases as the rate of additive increases.

The assessments given above vary according to the type of bitumen. Elvaloy® additive used in modification with bitumen makes significant improvements to the bitumen, turning it into a harder and more rigid structure. In this case, it is thought that its use in regions where hot weather conditions prevail will enable the construction of a more sustainable road superstructure. This shows that it will provide significant benefits in both environmental and economic aspects.

CONFLICT OF INTEREST

The author declares no conflict of interest.

ACKNOWLEDGMENT

I would like to thank the Highways 9th Regional Directorate (Diyarbakir) and 97th Branch Chiefdom (Batman), Türkiye and its staff for providing the samples and support to transfer them to the Batman University transportation lab and their personals for helping. I would also like to thank KOMSA Engineering Construction Industry Trade Inc. for donating the polymer to be used in the present and future studies.

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