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Rheological characteristics of bitumens prepared with process oil

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Research paper

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Depletion of natural and finite resources has necessitated the worldwide reuse of waste and residue materials from industrial production. Not only does the utilization of crude oil residue with bitumen allow supply of additives at a very low cost, but it also permits the use of waste and residue materials as rejuvenator in nature. The objective of the study is to evaluate conventional and rheological characteristics of original bitumen samples obtained from two different refineries involving different contents (1 %, 2 % and 3 %) of process oil (PO). The results show that PO can be used as an effective and environmentally friendly solution instead of commercially available expensive additives that are used in modification process.

Key words:
crude oil residue, rejuvenation, process oil, rheological characterization, Superpave performance, rutting parameter

Prethodno priopćenje

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Reološka svojstva bitumena s procesnim uljem kao aditivom

Smanjenje prirodnih, ograničenih resursa u svijetu potiče ponovnu uporabu otpadih materijala kao i ostataka industrijske prozvodnje. Upotreba ostataka destilacije sirove nafte kao dodatka bitumenu nije samo izvor jeftinog aditiva, nego ona znači da otpadni materijal može djelovati kao prirodni poticatelj. U radu je provedena ocjena standardnih i reoloških svojstava uzoraka bitumena dobivenih iz dvije rafinerije s različitim udjelom (1 %, 2 % i 3 %) procesnog ulja. Rezultati su pokazali da se procesno ulje može upotrebljavati kao učinkovit i ekološki prihvatljiv izbor u odnosu na komercijalno skupe dodatke koji se koriste tijekom procesa modifikacije.

Ključne riječi:
naftni ostaci, prirodni poticaj, procesno ulje, reološka karakterizacija, svojstva superpave, parametar kolotraga

Vorherige Mitteilung

Julide Oner

Rheologische Eigenschaften von Bitumen mit Prozessöl als Additiv

Die Reduzierung der natürlichen, begrenzten Ressourcen auf der Welt fördert die Wiederverwendung von Abfallstoffen sowie von Rückständen der industriellen Produktion. Die Verwendung von Rohöldestillationsrückständen als Bitumenzusatz ist nicht nur die Quelle eines billigen Zusatzstoffes, sondern bedeutet auch, dass Abfallmaterial als natürliches Antriebselement wirken kann. Die Arbeit bewertet die Standard- und rheologischen Eigenschaften von Bitumenproben aus zwei Raffinerien mit einem unterschiedlichen Anteil (1 %, 2 % und 3 %) an Prozessöl. Die Ergebnisse haben gezeigt, dass Prozessöl als wirksame und umweltverträgliche Wahl gegenüber kommerziell teuren Additiven verwendet werden kann, die während des Modifizierungsprozesses verwendet werden.

Schlüsselwörter:
Ölrückstände, natürliches Antriebselement, Prozessöl, rheologische Charakterisierung, Superpava-Eigenschaften, Vorspurparameter

1. Introduction

Crude oil is a material naturally occurring in liquid state in sedimentary r ocks, with colour varying from brown to black. It is a complex mixture of hydr ocarbons. While the light fraction of crude oil contains ass ociated gases and low molecular weight hydr ocarbons, the heavy fraction of crude oil includes a wide variety of naphthenic and aromatic compounds [1]. When temperature decreases, the heavy fraction part precipitates in the form of liquid oil mass, which may be deposited in the pipe walls or processing equipment. This precipitation has economic implications of significant interest to petroleum industry [2].

Consumption of natural resource is a dangerous and highly topical issue of this century. Limitation of crude oil reserves makes the products expensive. The price of bitumen is directly based on the price of crude oil since the bitumen is a byproduct of crude oil [3]. The development of bitumen containing a new additive is of great importance as a result of rapid price changes and for achieving sustainability of natural resources [4].

A residue is produced after hydr ocracking, thermal cracking and catalytic conversion [5]. Bitumen is the name given to the residue that is actually a viscous petroleum distillate remaining after atmospheric distillation of crude oil, further refined under vacuum. Bitumen is used in the production of asphalt mixture, where it essentially acts as a binder for mineral aggregates to form asphalt mixes, also called bituminous mixes, asphalt concrete, or bituminous concrete [6, 7].

Bitumen that contains saturates, aromatics, resins and asphaltenes is a colloidal dispersion of asphaltene into the maltene matrix [3]. The variability and chemical composition of bitumen are related to the source and type of crude oil, and to the manufacturing process. Therefore, bitumen performance is directly related to manufacturing conditions. One of the most frequently used methods to overcome this burning issue is the physical or chemical modification of bitumens by various additives [8]. With an increase in traffic volume and axle loads, the demand placed by communities for safe and better quality travel has imposed the need to build durable asphalt pavements [9]. The comfort and durability parameters that asphalt pavements can provide over the service life depend on the properties of bitumen used in asphalt mixtures [9, 10].

The utilization of additives in bitumen improves stiffness properties at high temperatures, and resistance against moisture and cracking at low temperatures [11-13]. This has encouraged researchers and engineers to investigate the effective and environmentally friendly additives for bitumen modification, aimed at improving performance characteristics of bitumen [12].

Many sectors consume large quantities of engine oil. Engine oil is also referred to as process oil, oil lubricant, and motor oil [14]. Several attempts have been made to use engine oil in conjunction with bitumen due to similarity in their molecular structure. The utilization of engine oil can provide advantages such as money savings, preservation of nature and natural resources, and reduction in environmental pollution [14, 15].

Process oil is defined as a special engine oil that is used in a wide variety of chemical and technical industries either as a raw material component or as an aid to processing [14, 16]. Process oil may also be used as bitumen extender, but addition rates of process oil are generally low in order to guarantee adequate performance of asphalt mixtures. On the other hand, process oil, which should be used as a partial substitute of bitumen, has a higher commercial value for pavement industry. It is used as an additive to bitumen in the United States and Canada because of its low cost and high availability [17]. Several researches have placed emphasis on the utilization of process oil, engine oil and motor oil as extenders or modifiers [18]. Many of them are used to decrease the softening point and viscosity values and to increase the penetration point. Yet, the elastic recovery and permanent deformation resistance may not be appropriate [19]. Considering the utilization of commercial polymers that provide solution to susceptibility to fatigue cracking, thermal cracking, and pothole formation issues in asphalt mixtures, bitumen modification with process oil products can be a promising, environmentally friendly, and economical solution for pavement industry [17].

In recent years, many studies have been published on the modification of bitumen with engine oil, process oil and various types of waste oil. Lei et al. (2017) determined that the utilization of lubricating oil may soften bitumen while decreasing the softening point and increasing the penetration value [15]. Fernandes et al. (2018) reported that high performance at elevated temperature and low non-recoverable creep compliance values were achieved by the use of high amounts of waste materials [17]. Liu et al. (2018) found that utilization of waste process oil decreases rutting resistance while increasing fatigue resistance of asphalt pavements [20]. Qurashi and Swamy (2018) prepared several asphalt blends using varying percentages of process oil. They concluded that process oil can act as a rejuvenator while providing elasticity and flexibility to bitumen [21]. Shafiq et al. (2018) reported that low content of process oil causes a substantial reduction in the coefficient of oxygen permeability and porosity of all mixes [18].

The primary objective of this study is to evaluate the effects of different quantities (1 %, 2 % and 3 %) of process oil (PO) in various original bitumen samples obtained from two different refineries. Considering the recommended dosages and addition methods given in previous literature, the values of 1 %, 2 % and 3 % of PO additive by weight of bitumen were chosen [17, 20, 22]. Laboratory tests including conventional bitumen tests, rotational viscosity (RV) test, Superpave performance grading (PG), dynamic shear rheometer (DSR), zero shear viscosity (ZSV) and multiple stress creep recovery (MSCR), were used to explore rheological characteristics of bitumen samples with different amounts of PO. Unaged and aged specimens were subjected to oscillating shear at a frequency of 1.59 Hz at different temperatures starting from 52 oc for unaged and 64 oc for aged samples with 6 oc increments in order to determine the upper critical temperature used in the PG system. ZSV and MSCR tests were also performed at different stress levels in creep mode. Besides, oscillation tests were performed to evaluate rutting parameters at low (0.01 Hz) and high (10 Hz) frequency levels at five different temperatures by means of a dynamic shear rheometer (DSR). Furthermore, the aim is to find an environmentally friendly alternative bitumen modifier instead of bitumen polymers, and to fulfil all bitumen modification requirements. The objective is also to define whether PO can be used as an alternative to commercially available additives for bitumen modification.

2. Materials and preparation of samples

In this study, two bitumen samples from different refineries were selected as original (base) bitumens. The original bitumens (OB) that are extensively utilized in Turkey for flexible pavement construction were obtained from the Turkish Petroleum Refineries Corporation (TUPRAS®). The official label is "TÜPRAŞ® Bitüm 50/70" for both original bitumen samples. The bitumen samples were classified as OB-1 and OB-2, respectively. OB-1 was produced from crude oil deposited in the east part of Turkey and OB-2 was manufactured by blending crude oil from various sources in Middle East countries.
The additive used in this study is process oil (PO), obtained as a byproduct during distillation of crude oil. PO, which is a commercial product supplied by TUPRAS®, is widely used in chemical and technical industries either as a raw material component or as an aid in processing substances such as brake fluids, paints, lubricants, coatings, cold resistant plastics, dyes, inks, pharmaceuticals, nylons, and so on. The main aim of this study is to evaluate the effects of adding 1 %, 2 % and 3 % of PO in two different types of original bitumen samples. The main properties of the PO are presented in Table 1.
The preparation of bitumen samples was basically realized in accordance with the Turkish Highway Construction Specifications. A rotational viscometer was employed to determine production temperature and periods of bitumens containing different PO contents. In preparation, an appropriate amount of original bitumen samples (250 g) in a 250 ml graduated beaker was heated in the stove to 120 oc and then placed under a laboratory type mixer (800 rpm). Different dosages of PO additive by weight of bitumen were gradually added to the beaker and the mixing action started at 140 oc. The temperature was kept constant and the viscosities were evaluated using a rotational viscometer for every five-minute increment. The viscosities were evaluated for 5 different mixing periods (5, 10, 15, 20 and 25 minutes) but no constant viscosity value was reached. The same pr ocedure was repeated for other samples but the production temperature was kept constant at 150 oc. It was identified that the viscosity values of the sample were kept at a constant value after stirring for 20 minutes. The production temperature and the period for adding each proportion of PO additive into original bitumen samples were eventually identified to be 150 °C and 20 minutes. Sample descriptions are presented in Table 2.

Table 1. Technical properties of process oil

Properties	Specification	Results
Density at 15.6 °C [kg/m³]	ASTM D1298 - ASTM D4052	849.4 - 1013.7
Viscosity at 100 °C [cst]	ASTM D445	13.5
Sulphur [mas. %]	ASTM D129	0.5
Aniline point [°C]	ASTM D611	35
Color [% 10 dilute]	ASTM D1500	4.5
Flash point [°C]	ASTM D92	240
Water [mas. %]	ASTM D95	0.1
Pour point [°C]	ASTM D97 - ASTM D5950	12

Table 2. Descriptions of samples

Terminology	Abbreviation
Original Bitumen -1	OB -1
Original Bitumen -1 + 1 % Process Oil	OB -1 + 1 % PO
Original Bitumen -1 + 2 % Process Oil	OB -1 + 2 % PO
Original Bitumen -1 + 3 % Process Oil	OB -1 + 3 % PO
Original Bitumen -2	OB -2
Original Bitumen -2 + 1 % Process Oil	OB -2 + 1 % PO
Original Bitumen -2 + 2 % Process Oil	OB -2 + 2 % PO
Original Bitumen -2 + 3 % Process Oil	OB -2 + 3 % PO

3. Experimental methods

3.1. Conventional bitumen tests

Two different original bitumen samples and bitumen samples containing 1 %, 2 % and 3 % of process oil (PO) were initially subjected to penetration, softening point, rolling thin film oven (RTFOT), penetration and softening point after RTFOT tests (ASTM D5-06, ASTM D36-06, ASTM D2872-12) [23-25]. On the other hand, the temperature susceptibility of bitumen samples was evaluated in terms of Penetration Index (PI) according to the results obtained from penetration and softening point tests.

3.2. Rotational viscosity tests

Rotational viscosity tests were conducted in accordance with standards described in ASTM D4402-06 [26]. This testing was used to determine flow properties with regard to workability of bituminous mixtures. The rotational viscosity testing was conducted by measuring torsion that maintained rotational speed of a cylindrical rod in bitumen samples around its own axis constant after attaining the steady state condition [19, 27, 28].

In this study, rotational viscosities of bitumen samples were tested at a temperature range of 135 °C and 165 °C. A Brookfield Viscometer DV-II Pro EXTRA was used at a fixed rotation speed of 20 rpm at a torque of 45 %. The temperatures corresponding to bitumen viscosities of 170 ± 20 mPa·s and 280 ± 30 mPa·s were chosen as mixing and compaction temperatures, respectively [27].

3.3. Rheological tests

This part of study concerns rheological test procedures conducted on original bitumen samples obtained from two different refineries involving different contents (1 %, 2 % and 3 %) of PO. Rheological tests were applied using three groups of Dynamic Shear Rheometer (DSR), Zero Shear Viscosity (ZSV), Multiple Stress Creep Recovery (MSCR) tests.

Performance grade tests were conducted on the eight different bitumen samples using DSR in order to evaluate the upper critical temperature. Original bitumen samples and samples containing different content of PO additive were subjected to oscillating shear by the use of DSR at the frequency of 10 rad/s (1.59 Hz). The temperature cycles were set to start at 52 oc for un-aged and 64 oc for RTFOT aged samples and then to run up in 6 oc increments.

In spite of the fact that the PG specifications require conduct of tests at a single frequency, the test can be repeated for a range of frequencies and is termed the "frequency sweep test". The frequency sweep test yields a number of parameters related to the bitumen being tested: complex modulus, viscous modulus, elastic modulus, and phase angle [16]. The operation of DSR is fairly basic during the frequency sweep test. As the plate oscillates, the centreline of the plate at a point moves to other point. This oscillation is one cycle and is repeated for a specified number of cycles. The number of cycles completed in one second is the loading frequency [16]. In this study, shear stresses were applied on the specimen at different frequencies (10 Hz and 0.01 Hz) over a range of temperatures (from 40 °C to 80 °C, with an increment of 10 °C). On the other hand, the rutting parameter ($G^*/\ \delta$) was obtained as the output of the frequency sweep test. The test results indicate the change of rutting parameters at different frequencies and temperatures.

Zero Shear Viscosity (ZSV) is defined as the viscosity value at a shear rate approaching zero during shear deformation. This parameter can be a predictor for rutting related bitumen characteristics. Many researchers indicated that $G^*/\sin\delta$ is not very effective or that it fails to predict the rutting performance of bitumen [19, 28]. In ZSV test, short-term aged samples were utilized based on CEN/TS 15325 [29]. The ZSV of samples was predicted by means of the creep test conducted at 60 °C. The test geometry for creep tests consisted of 25 mm parallel plates and the gap between the plates was 1 mm. The stress level was 10 Pa for bitumen samples, and each creep test lasted 30 minutes.

The Multiple Stress Creep Recovery (MSCR) test was introduced [30] during revision of bitumen testing procedures. The MSCR test was performed in accordance with ASTM D7405-08 using the DSR at 0.1 and 3.2 kPa stress levels on RTFOT aged specimens 25 mm in diameter, with 1 mm gap, at the temperature of 60 °C [31]. Ten cycles were applied at each stress level, with 1 s creep and 9 s recovery time. The percentage of recoverable and non-recoverable components of creep compliance was determined at the end of the test.

4. Results and discussions

4.1. Conventional bitumen test results

Following preparation of bitumen samples, conventional bitumen tests were performed on each of the samples presented in Table 3.

Conventional properties of bitumen prepared with different contents of PO are presented in Table 3 as an increase in penetration and decrease in softening point. The consistency of bitumen samples is determined by the penetration test. Penetration value is inversely proportional to viscosity and eventually bitumen softens as the penetration values increase. When the penetration values of original bitumen samples and bitumen samples containing three different contents of PO were compared, it was observed that the consistency decreased with the addition of PO. Bitumens produced from different crude oils demonstrate different levels of susceptibility to temperature.

The simplest method for measuring sensitivities is the softening point test. As depicted in Table 3, when softening point values are taken into consideration after the aging process, the amount of volatile matter in the structure of the bitumen samples decreases during aging, and so the content of asphaltene increases. Accordingly, due to increment in the rate of the crystallization, bitumen samples become harder and the softening point values increase as compared to the values before the aging process.

The temperature susceptibility of bitumen samples was calculated in terms of penetration index (PI) using the results obtained from penetration and softening point tests. Temperature susceptibility is defined as the change in consistency as a function of temperature. The increase in penetration indexes indicates that the sensitivity of bitumen samples to temperature is significantly reduced. The results of the experimental study indicate that the OB-1 samples containing three different proportions of PO have low temperature sensitivity and are more resistant to formation of rutting as compared to OB-2 and OB-2 samples including three different percentages of PO. As to OB-2 and OB-2 samples including different contents of PO, they exhibit lower temperature susceptibility when the PO content is increased.

All bitumen samples containing different contents of PO that are listed in Table 3 exhibit lower values in terms of viscosity at 135 °C and 165 °C compared to original bitumen samples. The utilization of PO additive in original bitumen samples shows positive effects in decreasing the viscosity values at 135 oc and 165 oc at all three concentrations of PO samples. As compared to bitumen samples containing PO additive, the lowest viscosity value was achieved at OB-2 + 3 % PO sample. Hence, conventional bitumen test results indicated that the addition of PO in OB-2 bitumen increases resistance to permanent deformations.

4.2. Rotational viscosity test results

The rotational viscosity values of samples involving PO are also shown in Table 3. The coefficient of variation (which is calculated as the ratio of standard deviation to mean value) related to bitumen tests such as Brookfield viscosity at 135 °C and 165 °C, varies between 0.15 % and 0.52 % indicating a reasonable consistency. As presented in Table 3, the utilization of PO additive in original bitumen samples gives a desirable effect in decreasing viscosity at 135 °C and 165 °C for all PO concentrations. The lowest value in terms of viscosities at 135 °C and 165 °C was achieved in OB-2 including 3 % PO. This indicates that the utilization of PO in bitumen increases the workability and reduces mixing and compaction temperatures.

Table 3. Conventional properties of the bitumen samples

Test	Specification	OB-1	OB-1 + 1 % PO	OB-1 + 2 % PO	OB-1 + 3 % PO	OB-2	OB-2 + 1 % PO	OB-2 + 2 % PO	OB-2 + 3 % PO
Penetration (25°C; 100 g; 5s), 0.1 mm	ASTM D5-06	50	56	72	75	48	68	76	88
Softening point [°C]	ASTM D 36-06	54	52	48.4	47	49.7	51	46.8	44.6
Penetration index [PI]		-0.25	-0.45	-0.73	-1.02	-1.37	-1.02	-1.04	-1.30
Viscosity at 135 °C [Pa·s]	ASTM D 4402-06	0.613	0.425	0.388	0.363	0.463	0.413	0.350	0.325
Viscosity at 165°C [Pa·s]	ASTM D 4402-06	0.150	0.135	0.113	0.113	0.125	0.113	0.113	0.100
Rolling thin film oven test (RTFOT). ASTM D 2872-12									
Change of mass after RTFOT [%]		0.07	0.15	0.15	0.16	0.07	0.16	0.16	0.17
Retained penetration after RTFOT		62	58.93	75	69.33	58	82.4	85.53	72.27
Increase in softening point RTFOT		8.8	7.9	6.6	6.3	4.7	4.8	5	3.7

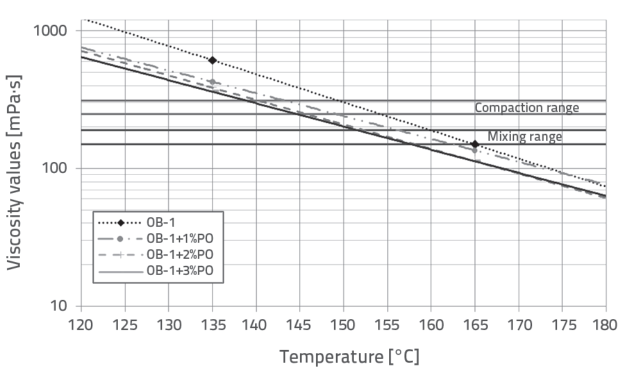

Figure 1. Mixing and compaction ranges for OB-1 and OB-1 containing PO additive

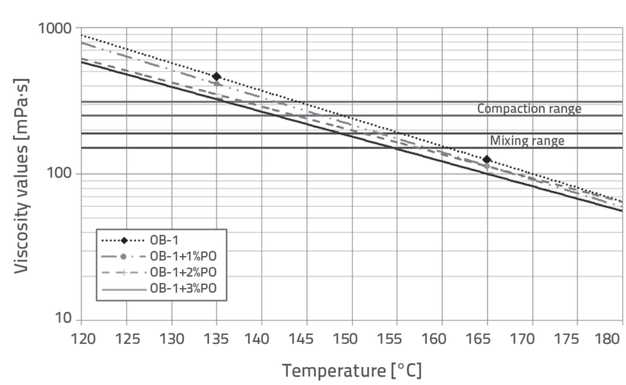

Figure 2. Mixing and compaction ranges for OB-2 and OB-2 containing PO additive

Table 4. Mixing and compaction temperatures

	PO Content [%] / Bitumen type	0	1	2	3
Mixing temperatures [°C]	**OB-1**	160-165	155-163	152-157	152-157
Compaction temperatures [°C]		150-154	143-147	140-145	139-145
Mixing temperatures [°C]	**OB-2**	156-161	153-157	151-156	148-154
Compaction temperatures [°C]		144-149	142-146	137-143	135-141

The Asphalt Institute Equi-Viscous Method was implied in determining mixing and compaction temperatures of asphalt pavements. Viscosity values of each sample at 135 °C and 165 °C were plotted on semi logarithmic viscosity-temperature charts. Mixing and compaction temperatures are illustrated for all samples in Figure 1 and Figure 2. 0.17±0.02 Pa·s and 0.28 ± 0.03 Pa·s are fixed as acceptable viscosity ranges related to mixing and compaction temperatures of samples, respectively. Mixing and compaction temperature ranges for all samples, including original bitumen samples and bitumen samples containing different contents of PO additive, are listed in Table 4.

As can be seen in Table 4, the utilization of PO additive in the two types of original bitumen samples obviously decreases mixing and compaction ranges of the mixtures. It is evident that the addition of 1 %, 2 %, and 3 % PO additive reduces the mixing temperature by 4 °C, 7 °C, and 8 °C, respectively. Besides, the addition of 1 %, 2 %, and 3 % PO additive decreases the compaction temperatures by 5 °C, 8 °C, and 10 °C, respectively.

4.3. Rheological test results

4.3.1. Superpave performance grading

The Superpave performance grading test was performed on the eight different bitumen samples using the Dynamic Shear Rheometer (DSR) to evaluate the upper critical temperature of samples. The coefficient of variation (which is calculated as the ratio of standard deviation to mean value) related to rheological bitumen tests varies between 0.78 % and 2.12 %, indicating a reasonable consistency. The calculated variances show that the rheological test results vary in acceptable ranges. In the Superpave performance grading system specification, $G^*/\sin\delta$ must be no less than 1 kPa for the unaged bitumen sample, and 2.2 kPa for the RTFO aged bitumen sample [16, 19, 28]. The high temperature performance grade of each bitumen sample is given in the Table 5.

As can be seen in Table 5, the utilization of 2 % and 3 % PO with OB-1 bitumen sample increases the upper critical temperature from 70 °C to 76 °C. Higher upper critical temperature is an indicator of higher resistance to rutting deformation [15, 16]. However, an addition of 1 and 2 % of PO to OB-2 bitumen sample does not have any effect on the upper critical temperature of samples. As the upper critical temperature for unaged and aged OB-2+3 % PO sample does not match at the same temperature, a lower temperature (52 °C) was chosen to be on the safe side in terms of the upper critical temperature. This situation is due to the fact that OB-2+3 % PO sample hardens and oxidizes more as compared to other bitumen samples because of the aging process.

4.3.2. Frequency sweep test

The bitumen samples were assessed by means of the DSR test to evaluate rutting parameter ($G^*/\sin\delta$) at five various temperatures from 40 °C to 80 °C with 10 °C increment at low and high frequencies. The calculated $G^*/\sin\delta$ parameters for 0.01 Hz and 10 Hz frequency levels are shown in Figure 3 and Figure 4, respectively.

As presented in Figure 3 and Figure 4, G*/ δ parameters related to all bitumen samples increase with a decrease in temperature at both low and high frequencies. High G*/ δ parameter indicates higher resistance to permanent deformations. On the other hand, G*/ δ parameters increase with an increase in frequency for all bitumen samples due to rheological behaviour of bitumen. Bitumen samples exhibit elastic behaviour under shorter loading times (high frequency).

As depicted in Figure 3 and Figure 4, OB-1 sample and the samples including three different contents of PO exhibit higher G*/ δ parameters as compared to OB-2 and OB-2 including 1 %, 2 % and 3 % PO samples at all temperatures and at both frequencies. For OB-1 and all concentrations of PO, G*/ δ values increase with an increase in the content of PO at all temperatures and at both frequencies.

As to OB-2 and OB-2 including different concentrations of PO samples, G*/ δ value increase with an addition of 1 % PO additive as compared to OB-2 sample at all temperatures and at both frequencies. This implies that the utilization of 1 % PO additive in OB-2 sample yields a positive effect in terms of rutting resistance. However, this case is not valid for the utilization of 2 % and 3 % PO additive in OB-2 bitumen samples. The utilization of PO additive of more than 1 % in the OB-2 bitumen sample decreases G*/ δ values at all temperatures and at both frequencies.

Table 5. High temperature performance grade of bitumen samples

Bitumen sample	Temperature [°C]	Rheometer test with dynamic smoothing - DSR G*/sinδ [Pa]		Performance grade
		Original*	RTFO Aged**	
OB-1	52	1.25E+04		PG70
	58	5918		
	64	2820	5816	
	70	**1384**	**2868**	
	76	702	1462	
OB-1+ 1 % PO	52	1.18E+04		PG70
	58	5553		
	64	2629	6433	
	70	**1295**	**3218**	
	76	666.6	1674	
OB-1+ 2 % PO	52	1.14E+04		PG76
	58	6385		
	64	3551	1.02E+04	
	70	2245	5022	
	76	**1025**	**2471**	
	82	623.8	1272	
OB-1+ 3 % PO	52	9494		PG76
	58	5687		
	64	4441	9785	
	70	2160	4807	
	76	**1006**	**2355**	
	82	978.6	1186	
OB-2	52	8047		PG 64
	58	3493		
	64	**1592**	**3356**	
	70	779	1493	
OB-2+ 1 % PO	52	5760		PG 64
	58	2498		
	64	**1124**	**2202**	
	70	529.2	1021	
OB-2+ 2 % PO	52	7306		PG 64
	58	3148		
	64	**1379**	**2293**	
	70	634.8	1063	
OB-2+ 3 % PO	52	4162		PG 58
	58	3641	**4466**	
	64	**1891**	2003	
	70	877.7		

* - Samples that were not exposed to aging
** - Samples that were exposed to aging by RTFO method (hardening in thin film with vertical rotation)

Consequently, based on oscillating test, it is clear that the utilization of PO additive in OB-1 bitumen sample results in an improved permanent deformation. Moreover, the addition of PO in OB-1 bitumen sample exerts a more profound effect compared to addition to OB-2 in terms of rutting resistance.

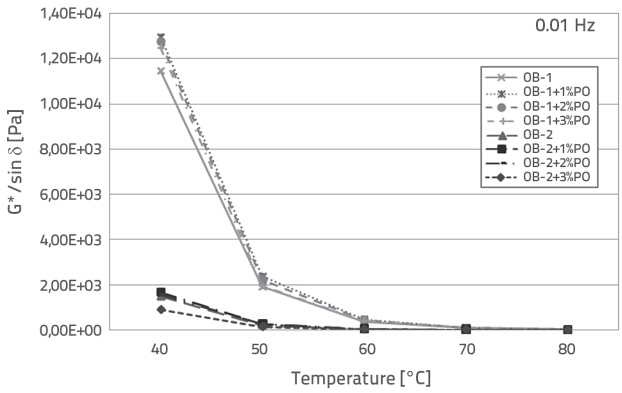

Figure 3. G*/sinδ values of samples at 0.01 Hz

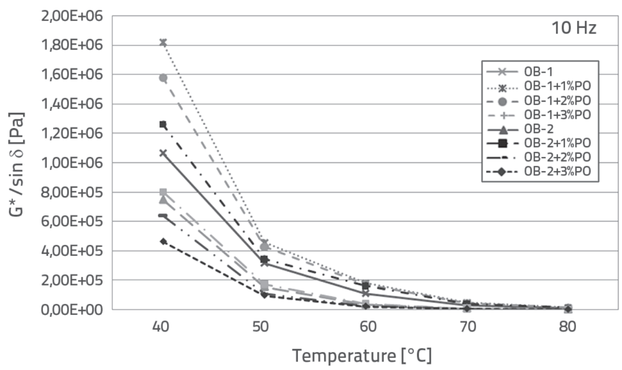

Figure 4. G*/sinδ values of samples at 10 Hz

4.3.3. Zero shear viscosity test

Zero shear viscosity (ZSV) values for all bitumen samples were evaluated using DSR machine at 60 °C in creep mode. ZSV test results related to all bitumen samples are presented in Figure 5.

As indicated before, this value can be a predictor for rutting related bitumen characteristics. As presented in Figure 5, OB-2 sample exhibits lower ZSV value compared to OB-1 sample since OB-2 was produced by the blending of crude oil from various sources. OB-1 sample and the samples including three different contents of PO yielded higher ZSV value as compared to OB-2 and OB-2 including 1 %, 2 % and 3 % PO samples. ZSV values increase with an increase in the content of PO in the OB-1 bitumen samples. Utilization of PO yields the peak ZSV value at 3 % concentration of PO in the OB-1 bitumen sample.

1 % and 2 % contents of PO in the OB-2 provide higher ZSV values compared to OB-2 bitumen samples. However, ZSV values decrease with the addition of 3 % PO to the OB-2 bitumen sample. Hence, the rutting performance is adversely affected by the utilization of 3 % PO in OB-2 bitumen samples.

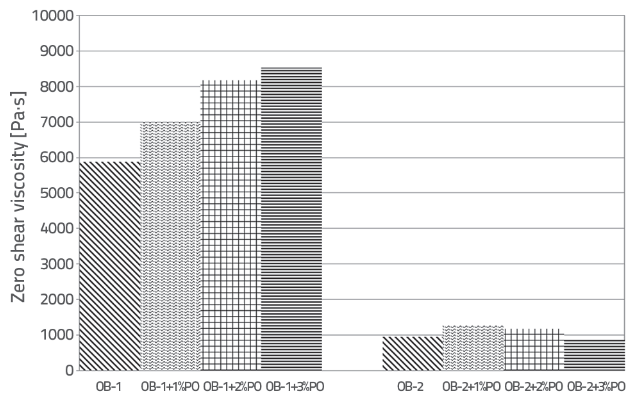

Figure 5. ZSV values for original bitumen samples and samples containing different contents of PO additive

4.3.4. Multiple stress creep recovery test

The multiple stress creep recovery (MSCR) test was performed by DSR in creep mode on eight various bitumen samples. The percent recovery, creep compliance, creep compliance difference between 100 Pa and 3200 Pa, and stress sensibility values are presented in Table 6.

As presented in Table 6, for all bitumen samples the percent recovery values at 3200 Pa are lower compared to those obtained at 100 Pa. Lower percent recoveries indicate that the samples may recover a lower portion at the end of cycles. On the other hand, higher recovery values indicate that samples may resist formation of rutting deformations. The utilization of PO additive in the OB-1 bitumen sample reveals higher percent recoveries compared to OB-2 and OB-2 including various percentages of PO. While the concentrations of PO additive in the OB-1 sample increase, the percent of recoveries increase at both 100 Pa and 3200 Pa loading conditions. This means that utilization of PO additive in OB-1 bitumen sample is favourable in terms of resistance of the material to permanent deformation such as rutting. However, the addition of more than 1 % PO in the OB-2 bitumen sample undesirably reduces the percent of recoveries. Similar evaluation may also be made for creep compliance (Jnr) values. Higher Jnr values define higher susceptibility of pavement to rutting deformations. In OB-1 and all concentrations of PO, Jnr values decrease with an increase in the content of PO at both 100 Pa and 3200 Pa loading conditions.

The percentage of difference in creep compliance (Jnr-diff) and stress sensibility of samples are also given in Table 6. Since the percent difference is a measure of sample sensitivity to an increase in stress level, lower values are associated with a less stress-sensitive material. The Jnr-diff and stress sensitivity are low for OB-2 bitumen samples, which indicates that a sample can be considered less stress-

Table 6. MSCR test results for bitumen samples at different stress levels in creep mode

Bitumen	R at 100 Pa [%]	R at 3200 Pa [%]	Jnr at 100 Pa [1/kPa]	Jnr at 3200 Pa [1/kPa]	Jnr-diff [%]	Stress sensitivity
OB-1	35.030	29.862	0.211	0.231	9.522	0.095
OB-1 + 1 % PO	36.744	31.109	0.210	0.230	10.382	0.103
OB-1 + 2 % PO	45.367	41.452	0.179	0.210	17.460	0.175
OB-1 + 3 % PO	58.544	54.874	0.138	0.166	21.062	0.211
OB-2	5.148	2.688	1.305	1.404	7.596	0.076
OB-2 + 1 % PO	8.662	6.520	0.943	1.094	15.960	0.160
OB-2 + 2 % PO	4.457	0.953	2.105	2.290	8.577	0.088
OB-2 + 3 % PO	3.821	0.660	3.027	3.340	10.201	0.102

Table 7. MSCR letter grades of all bitumen samples

Bitumen	Superpave performance grade	Jnr at 3200 Pa [1/kPa]	PG Plus grade
OB-1	PG 70	0.231	PG 64E
OB-1 + 1 % PO	PG 70	0.230	PG 64E
OB-1 + 2 % PO	PG 76	0.210	PG 64E
OB-1 + 3 % PO	PG 76	0.166	PG 64E
OB-2	PG 64	1.404	PG 64H
OB-2 + 1 % PO	PG 64	1.094	PG 64H
OB-2 + 2 % PO	PG 64	2.290	PG 64S
OB-2 + 3 % PO	PG 58	3.340	PG 64S

sensitive with a 3200 Pa in all bitumen samples. Jnr values at 100 Pa and 3200 Pa can be used to define the grading of bitumen samples. In addition to the Superpave performance grading, the Jnr value at 3200 Pa can be used to designate one of four traffic conditions, namely S (Standard: < 4.0 kPa^{-1} and standard traffic loading), H (Heavy: < 2.0 kPa^{-1} or slow moving traffic loading), V (Very Heavy: < 1.0 kPa^{-1} or standing traffic loading) and E (Extreme Heavy: < 0.5 kPa^{-1} ESALs and standing traffic loading) [32]. The mentioned MSCR letter grades (S, H, V, and E) of bitumen samples with respect to temperature of 64 oc are presented in Table 7.

As presented in Table 7, OB-1 and the samples containing 1 %, 2 % and 3 % PO additive belong to E: Extreme Heavy PG Plus Grade. This MSCR letter implies that OB-1 and the samples containing 1 %, 2 % and 3 % PO additive can withstand deformations even under extremely heavy traffic conditions. However, OB-2 bitumen samples including more than 1 % PO additive belong to S: Standard PG Plus Grade. This PG Plus Grade once again proved that OB-2 + 2 % PO and OB-2 + 3 % PO bitumens are the most sensitive to rutting deformation. When all test results are evaluated together, it can be seen that the results are affected by chemical composition of PO. Besides, the use of high percentages of PO was limited by blending crude oil from various sources in the Middle East countries.

5. Conclusion

One of the most hazardous issues of this century is the consumption of natural resources. The rapid depletion of petroleum reserves makes the products expensive. Therefore, the search for new additives is steadily intensifying due to rapid increase in the price of bitumen and to to achieve sustainability of natural resources. In spite of the fact that the literature shows that PO can be used as an alternative additive for modified asphalt pavements, detailed studies are needed from the point of view of chemical and rheological characteristics due to diversity in sources and processing techniques. The present study provides an evaluation of rheological characteristics of bitumen samples using different dosages of process oil. Original bitumen samples used in this study are extensively utilized in Turkey for flexible pavement construction.

Conventional bitumen tests indicate that the consistency decreases with the use of PO. OB-2 samples containing three different proportions of PO have low temperature sensitivity and are more resistant to the formation of rutting as compared to the OB-1 and OB-1 sample including three different percentages of PO. The utilization of PO in OB-2 bitumen increases resistance to permanent deformations in terms of conventional bitumen test results.

The asphalt industry aims to substantially decrease application temperatures of asphalt pavements by means of a variety of methods and additives. The utilization of PO additive in the two types of original bitumen samples obviously decreases mixing and compaction ranges of the mixtures. Hence, PO additive in bitumen increases the workability and makes relative reductions in mixing and compaction temperatures.

In the light of the frequency sweep test, in the case of OB-1 and all concentrations of PO, G^*/δ values increase with an increase in the content of PO at all temperatures and at both frequencies. However, it has been established that the use of PO additive in the quantities of more than 1 % in the OB-2 bitumen sample decreases G^*/δ values at all temperatures and at both frequencies.

In terms of ZSV values, the utilization of PO yields the peak ZSV value at 3 % concentration of PO in the OB-1 bitumen sample. However, the ZSV values decrease if 3 % PO is added to the OB-2 bitumen sample. Hence, the rutting performance is adversely affected if 3 % PO is added to OB-2 bitumen samples.

According to MSCR test results, the MSCR letter implies that OB-1 and the samples containing 1 %, 2 % and 3 % PO additive could remain resistant to deformations even under extreme heavy traffic conditions. However, OB-2 bitumen samples with more than 1 % of PO additive belong to grade S: Standard PG Plus Grade.

When all test results are evaluated together, 3 % and 1 % can be accepted as optimum PO contents in OB-1 and OB-2 bitumen samples, respectively. Besides, the use of PO in OB-1 is more effective since it significantly improves rheological properties of OB-1. It can also be stated that the use of process oil is justified from environmental, economic and rheological points of view. In addition, it should be noted that an efficient and environmentally friendly PO can be used as an alternative to commercial expensive additives for modification process. It is recommended to conduct a long term aging performance testing campaign by using the standardized method PAV (Pressure Aging Vessel) and the Bending Beam Rheometer (BBR) test for bitumen samples containing different contents of PO additive, so as to further evaluate low temperature performance of bitumen.

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