

Investigation of the rheological properties of bitumen modified with different waste oils under various frequencies and temperatures

Farklı atık yağ modifiyeli bitümlerin farklı frekans ve sıcaklıklardaki reolojik özelliklerinin incelenmesi

Erkut YALÇIN^{1*}, Mehmet YILMAZ²

^{1,2}Department of Civil Engineering, Engineering Faculty, Fırat University, Elazığ, Turkey.
erkutyalcin@firat.edu.tr, mehmetyilmaz@firat.edu.tr

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Abstract

In this study, the rheological properties of pure binders and modified bitumen that were prepared with these binders were investigated under various temperatures and frequencies. In the study, the B 50/70 class pure bitumen, which was obtained from the TUPRAS refinery in Batman, was used as the main binder. Waste oils were used as additives at 2%, 4%, 6%, 8%, 10%, 12% and 14% ratios. Pure and modified bitumen were subjected to dynamic shear rheometer (DSR) tests at 4 different temperatures (40 °C, 50 °C, 60 °C, and 70 °C) and 10 different frequencies (between 0.01 Hz and 10 Hz). As a result of the DSR tests, it was determined that the modified bitumen that was prepared with waste engine oil had higher complex shear modulus and lower phase angle values compared to the modified bitumen prepared with waste vegetable oil, which pointed to elastic behaviors at higher levels in favor of engine oil modification. It was also determined that waste vegetable oil proved more effective at 2% and 3% additive ratios in terms of the complex shear modulus values while waste engine oil modification was more effective at 2% and 4% additive ratios. In conclusion, it was discovered that waste engine oil was more effective in preparing modified bitumen compared to waste vegetable oil.

Keywords: Bitumen, Rheology, Waste engine oil, Waste vegetable oil, Mechanical analysis, Modification.

Öz

Çalışmada, saf bağlayıcı ve bu bağlayıcılarla hazırlanan modifiye bitümlerin farklı sıcaklık ve frekanslardaki reolojik özellikleri belirlenmiştir. Çalışmada ana bağlayıcı olarak Batman TÜPRAŞ rafinerilerinden temin edilen B 50/70 sınıfı saf bitüm kullanılmıştır. Katkı maddesi atık yağlar kullanılmış olup katkı oranları %2, %4, %6, %8, %10, %12 ve %14 olarak seçilmiştir. Saf ve modifiye bitümlere 4 farklı (40, 50, 60 ve 70 °C) sıcaklıkta ve 10 farklı frekansta (0.01 ile 10 Hz aralığında) dinamik kayma reometresi (DSR) deneyi uygulanmıştır. Uygulanan DSR deneyleri sonucunda atık madeni yağ kullanılarak hazırlanan modifiye bitümlerin atık bitkisel yağ kullanılarak hazırlanan modifiye bitümlerden daha yüksek kompleks kayma modülü ve daha düşük faz açısı değerlerine sahip olduğu dolayısıyla daha elastik davranış sergilediği belirlenmiştir. Kompleks kayma modülü değerleri üzerinde %2 ve %3 katkı içeriklerinde atık bitkisel yağ %2 ve %4 katkı içeriğinde ise atık madeni yağ modifikasyonunun daha etkin olduğu belirlenmiştir. Modifiye bitüm hazırlamada atık madeni yağın, atık bitkisel yağa göre daha etkin olduğu belirlenmiştir.

Anahtar kelimeler: Bitüm, Reoloji, Atık madeni yağ, Atık bitkisel yağ, Mekanik analiz, Modifikasyon.

1 Introduction

Flexible pavements in highways consist of aggregates and bituminous materials, which are used as binders. Hot mix asphalt (HMA) contains 93-95% aggregates and 5-7% bituminous materials as the weight of the mixture [1]. In Turkey, almost all of the roads that were constructed under the supervision of the General Directorate of Highways of Turkey were flexible pavements, which included 25.470 km of HMA and 38.817 km of chip seal [2]. Although bituminous binders, which are obtained by refining petroleum, provide sufficient levels of performance for most roads, bituminous binders that can demonstrate sufficient performance, especially under increased traffic volumes, numerous heavy vehicles, and various climatic conditions, are required.

Bituminous binders consist of asphaltenes and maltenes and they are regarded as colloidal complex compound systems with various levels of polarity and molar mass [3]. The level of distribution in asphaltenes and maltenes and the characteristics of hydrocarbons affect the physical features of asphalts, such as fatigue cracks, rutting resistance, low-

temperature cracks, and oxidation resistance [4]. Therefore, the most commonly used method to improve the performance of hot mix asphalt is using additive materials. Among the bituminous additive materials, the most frequently used ones are polymer-type materials [5]. During the modification processes, employing alternative waste materials to improve or support traditional asphalt binders also provides environmental benefits. Some of the commonly used waste materials for these purposes cover rubber [3],[4], recycled polyethylene (PE) [6], bio-oil derived from waste wood [7], waste vegetable oil (WVO) [8], electronic-waste plastics [9], and waste engine oil (WEO) [10]. Waste engine oils are refined petroleum products, such as engine oils extracted from motorized vehicles, and possess physical and chemical characteristics that are similar to petroleum asphalt.

On the other hand, waste vegetable oils constitute a significant amount of oils, which are collected after being used mainly in food industries, restaurants, hotels, and households for cooking practices such as frying and broiling.

Today, significant increases occur in the amount of WVO and WEO that are produced, which are due to reasons such as

*Corresponding author/Yazışılan Yazar

increased activity in food industries and increases in the number of vehicles in the world [11]. In today's numbers, the population of the world is 7.8 billion and it is estimated that this number will reach approximately 12 billion at the end of the next century. The population of the world is expected to reach 8 billion, an increase of 200 million, by the end of 2020 [12]. For example, in China, 5 million tons of waste vegetable oils are stored every year while in Turkey; 350 thousand tons of WVO are generated every year. With the rapid increase in the number of vehicles, significant amounts of WEO in the maintenance and repair process were generated. For example, in Turkey, 22 thousand tons of WEO are stored every year [13]. Unless WVO and WEO are eliminated properly, they threaten the environment and human health [14]. Thus, it is required to recycle, recover, and dispose of WVO and WEO safely for suitable purposes. Because eliminating WVO and WEO by storing is rather expensive, it is a difficult method to practice. In such a case, using WVO and WEO in bitumen enables environmental and economic benefits.

In the last decade, numerous studies have been conducted on bitumen modified with WVO and WEO. In such a study, Qui et al. (2018) compared the damage characteristics of aged bitumen with WEO and reported that WEO could improve the performance of aged bitumen [15]. In a study conducted by Jia et al., it was reported that asphalt and WEO had similar functional groups and molecular structures [16]. Thomas et al., determined that asphalt mixtures that were modified with engine oil had permanent deformation and dynamic modulus values that were similar to pure asphalt mixtures [17]. In a study conducted by Cao et al., waste vegetable oils were used for rejuvenating asphalts. Additionally, the researchers investigated the effects of waste vegetable oils on the rheological and chemical properties of aged asphalt binders. As a result of the tests, it was concluded that the workability and fatigue resistance of aged asphalts improved with waste vegetable additives [18]. In a study conducted by Koudelka et al., it was determined that the optimum ratios of rejuvenating agents depended on their origin. In the study, it was determined that the optimum ratios for the agents were 5% and 10% by the weight of the mixtures [19]. Zargar et al., investigated the possibility of using waste vegetable cooking oils as additive materials for rejuvenating aged bitumen in the laboratory with non-standard tests. The researchers revealed that including approximately 4% of waste vegetable cooking oil (by the weight of bitumen) in aged bitumen rendered it in a way that was similar to unaged bitumen [20]. In a study conducted by Chen et al., the effects of waste vegetable cooking oils on rejuvenating asphalt binders were investigated in terms of physical, chemical, and rheological properties. In conclusion, the researchers reported that waste vegetable cooking oil could effectively soften aged asphalt [21].

Although little amounts of binders are used in hot mix asphalt mixtures, they play significant roles in the performance of asphalt mixtures. Bituminous binders behave as Newtonian materials at high temperatures while behaving as viscoelastic solids at normal temperatures in addition to behaving similar to fragile viscoelastic materials even at low temperatures and moderate loads [16]. Therefore, conducting tests at various temperatures and various loading rates, instead of constant temperatures and loading rates, to evaluate binder performance yields more realistic results.

In this study, two distinct waste oils were included in pure bitumen at seven different ratios (2%, 4%, 6%, 8%, 10%, 12%,

and 14%) to prepare modified bitumen. Then, the rheological properties of the binders were evaluated by conducting dynamic shear rheometer (DSR) tests at 4 different temperatures (40°C, 50°C, 60°C, and 70°C) and 10 different frequencies (between 0.01 Hz and 10 Hz) on pure and 14 distinct modified bitumen.

2 Materials and experimental methods

2.1 Materials

In this study, the B 50/70 class bitumen with 1.015 g/cm³ density was used as the pure binder. The bitumen that was used in the study was obtained from the TUPRAS Batman refinery. Waste engine oils (WEO) were obtained from a local mechanic shop while waste vegetable oils (WVO) were obtained from the Municipality of Elazığ. The elemental compositions and physical properties of WVO and WEO were presented in Table 1. Additionally, the waste oils were demonstrated in Figure 1.

Table 1. Elemental composition and physical properties of WVO and WEO.

Items	Nitrogen (%)	Carbon (%)	Hydrogen (%)	Sulfur (%)	Flash Point (°C)	Density (g/cm ³)
WVO	-	76.42	11.00	-	310	0.896
WEO	-	83.18	13.91	0.408	210	0.8816



(a)



(b)

Figure 1(a): Waste engine oil and (b): Waste cooking oil.

In the study, 2 different waste oils were included in pure bitumen at 7 different ratios (2%, 4%, 6%, 8%, 10%, 12% and 14%) to prepare the targeted binders. To prepare the modified bitumen, bitumen and additive materials were mixed in a stable container at 180 °C and 1000 RPM for 1 hour [22]. The

abbreviations for the pure and binders that were modified with waste oils in the study were presented in Table 2. Additionally, a schematic for the preparation of the modified bitumen with waste oils was demonstrated in Figure 2.

Table 2. Abbreviations used for the bitumen in the study.

Waste Oils	Waste Oil Contents (%)							
	0	2	4	6	8	10	12	14
Waste engine oil	C	E2	E4	E6	E8	E10	E12	E14
Waste vegetable oil		V2	V4	V6	V8	V10	V12	V14

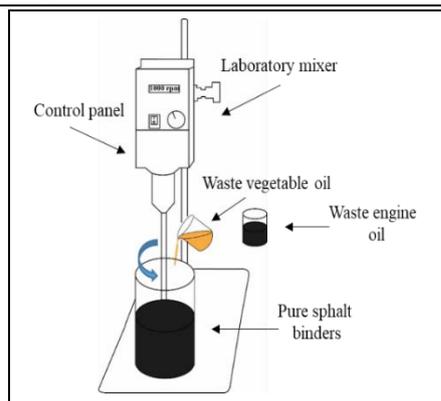


Figure 2. The preparation process of asphalt binders with WVO and WEO modifications.

The results of penetration, softening point, flash point, and elastic recovery tests, which were conducted on pure bitumen and bitumen with waste oil modifications, were presented in Table 3.

Table 3. Results of conventional binder tests conducted on pure bitumen and bitumen with waste oil modification.

Type of Binder	Penetration (mm ⁻¹)	Softening Point (°C)	Flash Point (°C)
C	62	53.3	245
V2	65	52.9	252
V4	92	51.0	254
V6	118	47.8	260
V8	136	45.9	265
V10	174	42.6	288
V12	192	40.0	298
V14	231	37.9	300
E2	64	53.25	242
E4	69	52.2	240
E6	77	51.1	238
E8	88	49.6	232
E10	105	48.15	228
E12	121	46.95	224
E14	136	44.65	217

E and V additives increased the penetration values of pure binders while decreasing softening point values. E additive decreased the flash point values compared to pure binder while V additive increased the flash point. As can be seen in Table 3, in the use of both waste oils, the penetration values were increased as the additive contents were increased in addition to decreased softening point values. As the content of the E additive was increased, the flash point values were decreased while increased V additive content decreased flash point values. It was determined that the binders with E additive had higher penetration and softening point values compared to binders with V additive. The flash point values of binders with E additive were lower than the flash point values of the binders

with V additive. The use of waste oils caused pure bitumen to demonstrate a softer behavior.

2.2. Dynamic shear rheometer test

Today, mechanical analysis is one of the most commonly used methods to determine the rheological properties of bituminous binders. In this analysis, the dynamic shear rheometer device, which was demonstrated in Figure 3, was used.



Figure 3. The dynamic shear rheometer test device.

As can be seen in Figure 4(a), the behaviors of bituminous binder samples are evaluated with the oscillation (sinusoidal) applied on bituminous binders. In the test, a thin bituminous binder sample is placed between two parallel metal plates, which are kept at a constant temperature. One of the plates moves sinusoidally while the other is kept stable. A complete load rotation of DSR was demonstrated in Figure 4(a). When torque is applied, the DSR motor goes from point A to point b, returns to point A, and moves to point C. Then, it reaches back to point A. This process constitutes a single rotation and it is repeated throughout the test [23].

The main viscoelastic parameters that are obtained from DSR tests are complex shear modulus (G^*) and phase angles (δ). G^* is defined as the ratio of maximum (shear) tension's rate to maximum transformation and represents the measurement of a total resistance against deformation when bitumen is exposed to shear load [24]. The complex shear modulus contains elastic and viscous components, which are defined as storage (elastic) modulus (G') and loss modulus (G''). These two components are related to complex (shear) modulus and phase angle (δ). Phase angle, which is defined as the delay between tension and transformation in an oscillation test, is a measurement of viscoelastic balance in behaviors of materials. If δ is equal to 90, then, it can be accepted that the bituminous material is completely viscous. On the other hand, if the phase angle is 0, the material corresponds to complete elastic behavior. Between these two peak points, the behavior of the material is regarded as a combination of viscous and elastic responses Figure 4(b) [25]. In the study, the sample geometry was chosen as 25 mm for diameter and 1 mm for height. Then, the DSR tests were conducted with the binders at 4 different temperatures (40 °C, 50 °C, 60 °C, and 70 °C) and 10 different frequencies (between 0.01 Hz and 10 Hz).

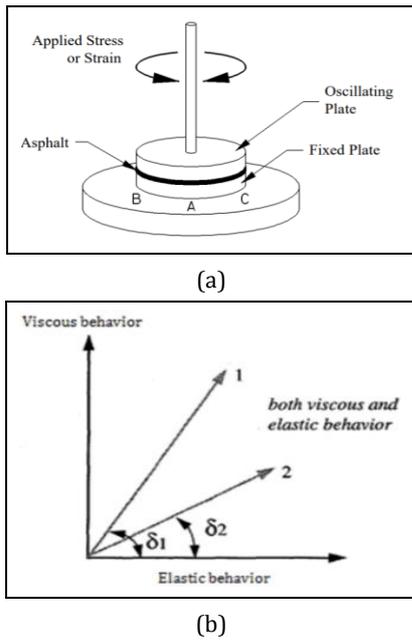


Figure 4(a): Directions of deformations applied to the samples in the DSR test and (b): Demonstration of elastic and viscous behaviors according to DSR test [23].

3 Results and discussion

In the study, the rheological behaviors of bituminous binders were investigated by conducting dynamic shear rheometer tests on pure and modified bitumen at 40 °C, 50 °C, 60 °C, and 70 °C, and 10 different frequencies between 0.01 Hz and 10 Hz. The results of the tests were superposed to draw the master curves by using Malvern Bohlin DSR software. The master curves of the complex modulus, which were obtained from modified binders with waste oils, and black diagrams were presented in Figure 5.

As can be seen in Figure 5, as the frequency values (loading rates) increased, the complex shear modulus values were increased. As the content of waste oils, the additive materials, were increased, the complex modulus values of the binders were decreased. The effects of the additive materials were more apparent at low frequencies while the effects of the waste oils were decreased at high frequencies. In the graphs, although the differences between the binders were apparent because the data were presented both horizontally and vertically, the values seem close to each other. Furthermore, as the waste oil contents were increased, the differences between the complex modulus values became apparent. It was determined that the complex modulus values of binders with waste engine oil modification were lower compared to the complex modulus values of binders with waste vegetable oil modification.

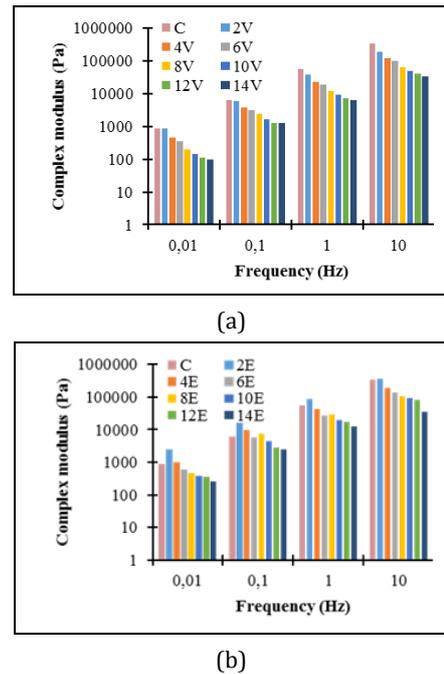


Figure 5. Complex modulus values of the binders at 4 different frequencies.

As can be seen in Figure 5, as the frequencies were increased, the complex modulus values were increased. On the other hand, as the additive contents were increased, the complex modulus values were decreased. Accordingly, it was determined that the complex shear modulus values of modified bitumen with waste engine oils were higher compared to the binders that were prepared with waste vegetable oils. Furthermore, as the waste vegetable oil contents were increased, it was determined that the complex modulus values were decreased. The pure binders yielded higher complex modulus values at every frequency compared to the binders prepared with waste vegetable oils. All the binders, except for 2E and 4E binders, provided lower complex values compared to pure binders. Similar results were determined in the study of the Chen team. Chen and his team used waste vegetable oil in bitumen modification [26]. The complex shear modulus values of the modified bitumen at 4 different frequencies, in comparison with the complex modulus values of pure binders, were presented in Table 4.

As can be seen in Table 4, more effective modifications were obtained in bitumen that was modified with waste vegetable oils 2V and 3V. It was also determined that the modified bitumen was more effective at 2% and 4% waste engine oil contents.

Table 4. The ratio of G^* values of the modified binders to G^* values of the pure binders.

Frequency (Hz)	G_{2V}^*/G_c^*	G_{4V}^*/G_c^*	G_{6V}^*/G_c^*	G_{8V}^*/G_c^*	G_{10V}^*/G_c^*	G_{12V}^*/G_c^*	G_{14V}^*/G_c^*
0.01	0.98	0.53	0.42	0.22	0.17	0.13	0.12
0.1	0.98	0.60	0.51	0.39	0.27	0.21	0.20
1	0.68	0.42	0.33	0.22	0.16	0.13	0.12
10	0.55	0.35	0.30	0.19	0.14	0.12	0.10
Frequency (Hz)	G_{2E}^*/G_c^*	G_{4E}^*/G_c^*	G_{6E}^*/G_c^*	G_{8E}^*/G_c^*	G_{10E}^*/G_c^*	G_{12E}^*/G_c^*	G_{14E}^*/G_c^*
0.01	2.78	1.14	0.67	0.54	0.45	0.40	0.30
0.1	2.68	1.58	0.93	1.19	0.73	0.44	0.40
1	1.59	0.77	0.50	0.53	0.36	0.30	0.23
10	1.10	0.57	0.40	0.31	0.28	0.23	0.10

Accordingly, it was determined that waste engine oil was more effective in preparing the modified bitumen compared to waste vegetable oil. As the contents of both waste oils were increased, the G^* values of the modified binders were increased compared to the G^* values of pure binders. Furthermore, as the frequency values were increased, the G^* values of the modified binders with both oils were decreased compared to the G^* values of pure binders. The ratio of G^* values of modified binders with waste engine oils to the G^* values of pure binders was higher compared to the ratio of G^* values of modified binders with waste vegetable oils to the G^* values of pure binders. The black diagrams of pure and modified binders were presented in Figure 6.

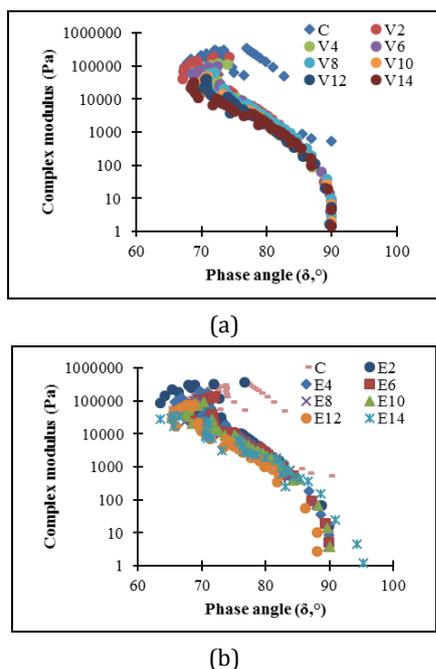


Figure 6. Relationship between complex shear modulus - phase angle (black diagrams) in binders prepared with modified bitumen that contained waste vegetable oils (a) and waste engine oils (b).

The low phase angle values and high complex shear modulus values indicated that the binders demonstrated more elastic behaviors. As can be seen in Figure 6, as the complex modulus values of pure binders were increased, the phase angle values were decreased. In terms of the modified binders, as the additive contents were increased, the phase angle values reached the lowest levels, especially at approximately $1.0E+5$ Pa complex modulus values. Additionally, as the additive contents were increased, the phase angle values were regularly decreased. It was observed that the lowest phase angle values for both waste oils were obtained at 2% modified bitumen content and between 65°C and 70°C . The fact that the phase angle values of both V and E modified bitumen were decreased until G^* values of $1.0E+5$ Pa and increased afterward indicated that the limit shear tension values of modified bitumen with both waste oils were similar to each other. The phase angle values of the E binder decreased until 60°C while it was determined that the binders with the best result had a phase angle value of approximately 63.46° . This indicated that the binders obtained from the modification with waste engine oil yielded better results in terms of elastic behaviors compared to

the binders obtained from waste vegetable oil modification. In addition, as the content of waste oil used in the study conducted by Li et al Increases, the G^* values of the modified binders decrease. In addition, G^* values decrease with the increase in temperature [27]. The changes in the elastic modulus of the binders with the viscous modulus (cole cole diagrams) were presented in Figure 7.

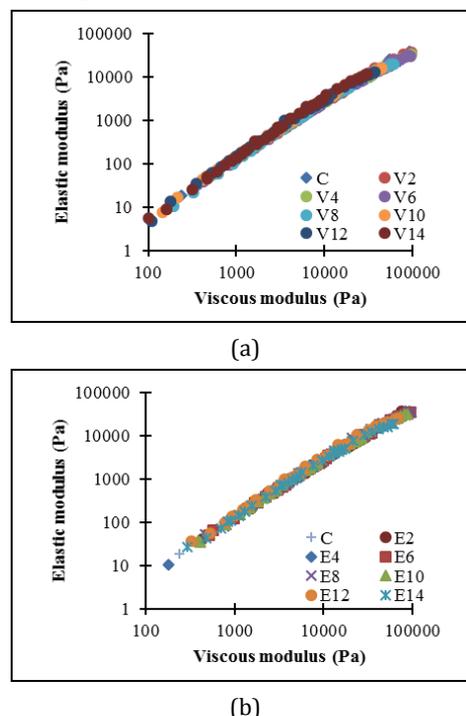


Figure 7. Relationship of elastic modulus - viscous modulus in binders prepared with modified bitumen that contained waste vegetable oils (a) and waste engine oils (b).

As can be seen in Figure 7, it was determined that the gradient in the elastic modulus-viscous modulus graph of modified bitumen with waste engine oil (E) was higher compared to the modified bitumen that was prepared with waste vegetable oil (V). This indicated that using modified bitumen that was prepared with waste engine oils yielded behaviors that were more elastic compared to using modified bitumen prepared with waste vegetable oils. For both the binders with waste vegetable oils and the binders with waste engine oils, it was determined that the diagrams became similar to each other as the oil contents were increased.

4 Conclusions

In the study, the B 50/70 class bitumen, which was obtained from the TUPRAS refinery in Batman, was used to create modified bitumen with 2 different waste oils at 7 different ratios (2%, 4%, 6%, 8%, 10%, 12%, and 14%). The rheological properties and the changes in the rheological properties of the binders were investigated by conducting dynamic shear rheometer tests at 4 different temperatures and 10 different frequencies. As a result of the tests, it was determined that the complex shear modulus and phase angle values of the binders that were prepared with waste vegetable oils and waste engine oils were different in terms of their additive content ratios. Furthermore, it was determined that the binders that were prepared with waste engine oils had higher complex modulus

values. The pure binders yielded higher complex modulus values at every frequency compared to the binders prepared with waste vegetable oils. All the binders, except for 2E and 4E binders, yielded lower complex modulus values compared to pure binders. In the black diagrams of the binders, it was determined that the modified bitumen with waste engine oil demonstrated behaviors that were more elastic compared to the modified bitumen with waste vegetable oils.

The fact that the phase angle values of modified bitumen with both waste oils were decreased until G^* values of $1.0E+5$ Pa and increased afterward indicated that the limit shear stress values of the modified binders prepared with both waste oils were similar to each other. The phase angle values of binders with waste engine oils were lower compared to binders with waste vegetable oils. This indicated that binders with waste engine oils had more elastic properties.

Generally, the viscosity of bituminous binders is determined with a static test conducted at a single temperature in penetration classification, while in the Superpave method, the rutting and fatigue parameters of the binders are determined via a DSR test conducted at a single frequency. In this study, it was observed that bituminous binders could demonstrate various rheological behaviors at various temperatures and various loading rates. This could be related to the chemical structures of waste oils. Therefore, it was concluded that conducting rheological tests at a wide range could be especially more effective while investigating the viscosities and rheological behaviors of modified bitumen.

5 Author contribution statements

In the work carried out, Erkut YALÇIN under the titles of forming the idea, making the design, supplying the materials, collecting data, performing the analysis, writing the article and literature review; Mehmet YILMAZ contributed to spelling, checking the article in terms of content, and critical review.

6 Ethics committee approval and conflict of interest statement

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

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