



Sustainable Production of Aging-Resistant Bitumen: Waste Engine Oil Modification

İslam Gökalp¹ and Volkan Emre Uz²

Abstract: Using waste engine oil (WEOIL) within bituminous binders might be one of the most energy-efficient and sustainable means of producing aging-resistant bitumen due to the antioxidative properties of WEOIL. In this paper, the use of WEOIL to obtain aging-resistant bitumen and its optimum rates for short and long terms were investigated. In this regard, a base bitumen was modified with WEOIL in certain rates ranging from 1% to 5% by weight of bitumen. Then base and oil-modified bitumen samples were subjected to aging. To define the changes in rheological properties of bitumen based on rutting, fatigue, and thermal cracking resistance, dynamic shear and bending beam rheometer tests were performed on each sample. Furthermore, an aging index (AI) analysis was performed for both the short- and long-term aging conditions to express the effect of WEOIL on aging resistance of the bitumen. According to the AI analysis, short-term-aging-resistant bitumen is obtained by adding 3.5% WEOIL to the base bitumen, while a 5.8% contribution rate is required to avoid the long-term aging effect. Moreover, the increase in rate of WEOIL content improved the low-temperature cracking resistance. Consequently, utilizing WEOIL for production of aging-resistant bitumen can provide environmental and economic benefits based on conservation of natural resources and waste recycling. DOI: [10.1061/JPEODX.0000315](https://doi.org/10.1061/JPEODX.0000315). © 2021 American Society of Civil Engineers.

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Introduction

The demand for industrial products has reached a critical level and consumption of raw materials is growing dramatically while natural resources are becoming scarce. Efforts for improving properties of industrial products related to their service life and the activities for recovering waste of natural resources and industrial products are the main concepts of sustainable production (UCLA 2019). Sustainable practices should also encourage environmental health and all kinds of living creatures on the earth and economic activities (Hart 1997; Norton 1992; Sikdar 2003). About 30% of greenhouse gas is emerged, 20% of energy is consumed, and 90% of waste is released in the world caused by economic activities (Tonelli et al. 2013). The amount of gas emission and energy consumption may be decreased with technological developments, but it is most likely that waste will continue to grow due to the increase in world population (Singh et al. 2009; Tseng et al. 2018). Materials that lost their properties at the end of use are categorized as waste, but it is possible to recover some of these materials with physical or chemical processes (LaGrega et al. 2010; Reed et al. 1995). The automotive industry, which constitutes approximately 5% of the world economy, engages with industries such as iron and steel, petrochemistry, glass, plastics, textiles, and electronics and it is one of the major industries producing large quantities of waste (Yilmaz et al. 2017). About 95.5×10^6 motor vehicles were produced and approximately 85×10^6 of them have been sold all over the world in 2019 (Demandt 2019; OICA 2019).

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Oil is used to protect the engines of motor vehicles from wear. After use, engine oils lose their physical and chemical properties and therefore they need to be recycled in accordance with the requirements established by the state or local government (Ssempebwa and Carpenter 2009). Waste engine oil (WEOIL) may contain hazardous heavy metals such as arsenic, cadmium, zinc, and lead (Irwin et al. 1997; Singh et al. 2006; Bartz 1998) and can adversely affect the ecosystem if they are not disposed of properly (WHO 2017). In Turkey, 0.45×10^6 t of engine oil is produced annually, and after consumption about 0.25×10^6 t of it emerged as waste. Unfortunately, only about 20% can be collected (PETDER 2016). The recovery rate might be higher in developed countries, and lower for others (Chung and Lo 2003; Oliveira et al. 2008).

Petroleum is a nonrenewable natural resource and demand for it is increasing on a daily basis with increasing population and developing technology. A recently published report showed that global petroleum consumption has grown by 1.8% and reached 1.7×10^6 barrels per day (b/d), while global refinery during the year of 2019 was about 1.6×10^6 b/d (BP Energy 2019). Petroleum comes in the form of a mixture of gas, liquid, and solid hydrocarbons and it is separable into fractions such as natural gas, gasoline, naphtha, kerosene, lubricating oils, paraffin wax, and bitumen. These fractions are very important for various industries as raw materials or energy sources (Pickett et al. 2018).

Bitumen (a black viscous mixture of hydrocarbons), which can be obtained from natural sources or produced by petroleum distillation, is widely used to construct road pavements all over the world and its properties play an important role in pavement performance. Aging, which is identified as oxidation, changes the physical, rheological, and chemical properties of bitumen significantly. Both short- and long-term aging cause bitumen hardening and loss of elasticity, and consequently some mode of distresses occur over the pavement surface (Uz and Gökalp 2020).

Considerable research has been devoted to produce aging-resistant bitumen and reuse waste materials such as vegetable cooking oil (Al-Omari et al. 2018), engine oil (Nurul Hidayah et al. 2014), and recycled asphalt (Barral et al. 2003) to conserve or minimize the use of

Table 1. Basic properties of base bitumen

| Test | Standard | Unit | Results | Limitation | Standard deviation |
|--------------------|-------------|-----------|---------------|-------------|--------------------|
| Penetration | EN 1426 | 0.1 mm | 76.2 | 70–100 | 0.56 |
| Softening point | EN 1427 | °C | 49.0 | Minimum 46 | 0.35 |
| Ductility | EN 12589 | cm | 104.3 | — | 2.40 |
| Flashing point | ISO EN 2592 | °C | 259.0 | Minimum 230 | 1.41 |
| Viscosity at 135°C | EN 13302 | Pa·s (cP) | 0.518 (517.5) | — | 0.73 |
| Viscosity at 165°C | | | 0.139 (139.2) | — | 0.12 |

natural resources in the road paving industry (Behnood 2019). Fernandes et al. (2017) reported that hot mix asphalt (HMA) performance is improved with WEOIL (10%)/styrene-butadiene-styrene (SBS) (5%) modification, which can be a superior solution for paving works. Shoukat and Yoo (2018) highlighted that among the other oil types, WEOIL addition decreased the stiffness and viscosity of asphalt binder, and for this reason it showed better performance at low temperatures. El-Shorbagy et al. (2019) demonstrated that waste cooking oil (WCO) and WEOIL improved the low-temperature thermal cracking of reclaimed asphalt pavements. Liu et al. (2018a) indicated that the elastic properties, thermal susceptibility, viscosity, and rutting resistance of bitumen were decreased, while fatigue resistance and temperature sensitivity were improved by WEOIL modification. Liu et al. (2018b) examined the effects of WEOIL on chemical properties of different asphalt binders. They concluded that there is a significant decrease in percentage of large-sized molecules and presence of carbonyl functional groups by addition of WEOIL to the bitumen. Gökalp et al. (2019) investigated the physical properties of WEOIL-modified bitumen and highlighted that the viscosity of bitumen decreased with WEOIL increment. In their paper, DeDene and You (2014) found that a certain rate of WEOIL is able to rejuvenate aged bitumen. Ma et al. (2020) presented a laboratory study and observed the properties of rejuvenated asphalt binders blended with different rejuvenator application procedures. They concluded that asphalt binders exhibited different properties after short-term aging, but were similar after long-term aging. Ackbarali and Maharaj (2011) investigated the effect of WEOIL amount on rheological properties of Trinidad Lake bitumen and between 10% and 20% reduction was reported in rutting resistance and viscosity. Hesp and Shurvell (2013) highlighted that addition of WEOIL would change the performance grade (PG) of bitumen and this fluctuation may be more remarkable according to its origin. Taherkhani and Noorian (2020) compared the rejuvenation ability of WCO and WEOIL using 10% by weight of bitumen in HMA containing 25%, 50%, and 75% of recycled asphalt pavement (RAP). They reported that mixtures with WCO showed better creep stiffness, while WEOIL-rejuvenated mixtures had higher resistance to plastic deformation. In the light of the literature, almost all the earlier studies focused on rejuvenation effect of WEOIL in aged bitumen by evaluating their physical, rheological, and chemical properties, rather than production of aging-resistant bitumen. Therefore, this study established the usability of WEOIL as an aging-resistant additive to fill the gap in the literature.

The objective of this study is to investigate the usability of WEOIL for sustainable production of short- and long-term-aging-resistant bitumen. In pursuit of this objective, a base bitumen was modified with different rates of WEOIL and the bitumen aged for short and long terms. To analyze the rutting, fatigue, and thermal cracking resistance of base and modified bitumen, a series of rheological tests were conducted on each sample according to related European standards. To demonstrate the aging resistance of modified bitumen, its rheological properties were compared to base bitumen using aging index (AI) analysis and the optimum contribution rates

Table 2. Basic properties of WEOIL

| Test | Standard | Unit | Results | Standard deviation |
|--------------------|------------|-------------------|--------------|--------------------|
| Color | ASTM D1500 | — | Black | — |
| Density | ASTM D1298 | g/cm ³ | 49.0 | 0.35 |
| Evaporation loss | ASTM D5800 | % | 2.0 | 0.02 |
| Water content | ASTM D1744 | % | 2.5 | 0.30 |
| Flashing point | ASTM D92 | °C | 135 | 0.50 |
| Viscosity at 100°C | ASTM D4683 | Pa·s (cP) | 0.019 (19.5) | 0.03 |
| Viscosity at 150°C | ASTM D4741 | Pa·s (cP) | 0.008 (8.1) | 0.01 |

of WEOIL were determined for each test method. It is believed that the findings make an important contribution to the literature.

Materials

In this study, bitumen and WEOIL samples were supplied from bitumen reservoir of the Turkey General Directorate of Highways and from Adana Organized Industrial Zone, where is located within the boundaries of Sarçam, District of Adana, Turkey, respectively. WEOIL was used to modify bitumen to enhance its aging-resistant characteristics and to prevent or slow down the process of aging. The basic properties of the bitumen are presented in Table 1 [EN 1426 (CEN 2015a); EN 1427 (CEN 2015c); EN 12589 (CEN 2002); ISO EN 2592 (CEN 2017); EN 13302 (CEN 2018)], while those of WEOIL are in Table 2 [ASTM D1500 (ASTM 2017b); ASTM D1298 (ASTM 2017a); ASTM D5800 (ASTM 2020a); ASTM D1744 (ASTM 2013); ASTM D92 (ASTM 2018); ASTM D4683 (ASTM 2020b); ASTM D4741 (ASTM 2021)]. The methodology used for identifying the base and WEOIL-modified bitumen and their short- and long-term-aged forms are presented in Table 3.

Methods

In this section, the test methods, modification process, and aging resistance analysis are presented. Basic and advanced bitumen tests were performed to compare the properties of bitumen. Basic tests are the simplest methods, which have been used for evaluation of physical properties of bitumen all around the world. However, the rheological tests, which have been introduced by US Strategic Highway Research Program through research called the Superpave PG system (Hesp and Shurvell 2010), are the most recent and advanced bitumen test methods. Throughout this study, rheological tests were used to determine the properties of base, WEOIL-modified, and aged bitumen samples. The dynamic shear rheometer (DSR) test was used to characterize the rutting/fatigue resistance and the bending beam rheometer (BBR) test was utilized to determine thermal cracking resistance properties of bitumen samples. The rolling thin-film oven (RTFO) test was performed to obtain short-term-aged bitumen, while pressure aging vessel (PAV) apparatus was utilized to obtain long-term-aged bitumen. The tests were conducted for at least two repetitions and the methods are presented in the following sections in brief.

Table 3. Sample identification

| Sample | Bitumen composition | Form of bitumen |
|-----------|--------------------------------|-----------------|
| BB | Base bitumen | Base and |
| WEOIL-1 | Base bitumen + 1% WEOIL | WEOIL- |
| WEOIL-2 | Base bitumen + 2% WEOIL | modified |
| WEOIL-3 | Base bitumen + 3% WEOIL | bitumen |
| WEOIL-4 | Base bitumen + 4% WEOIL | |
| WEOIL-5 | Base bitumen + 5% WEOIL | |
| BB-R | Base bitumen + RTFO | Short-term |
| WEOIL-1-R | Base bitumen + 1% WEOIL + RTFO | aged bitumen |
| WEOIL-2-R | Base bitumen + 2% WEOIL + RTFO | using rolling |
| WEOIL-3-R | Base bitumen + 3% WEOIL + RTFO | thin-film oven |
| WEOIL-4-R | Base bitumen + 4% WEOIL + RTFO | test (RTFO) |
| WEOIL-5-R | Base bitumen + 5% WEOIL + RTFO | |
| BB-P | Base bitumen + PAV | Long-term |
| WEOIL-1-P | Base bitumen + 1% WEOIL + PAV | aged bitumen |
| WEOIL-2-P | Base bitumen + 2% WEOIL + PAV | using pressure |
| WEOIL-3-P | Base bitumen + 3% WEOIL + PAV | aging vessel |
| WEOIL-4-P | Base bitumen + 4% WEOIL + PAV | (PAV) |
| WEOIL-5-P | Base bitumen + 5% WEOIL + PAV | |

Rutting/Fatigue Resistance Tests

DSR, which includes computer software, a compressor, a cooling/heating system and a test frame, is one of the major pieces of equipment in the Superpave PG system and is utilized to determine the viscoelastic properties of bituminous binders. The PG system requires testing the base and short- and long-term-aged forms of bitumen samples. All of these forms of bitumen can be evaluated under elevated temperatures and specified loading frequencies with the DSR equipment. At the end of the test, the complex shear modulus (G^*) and phase angle (δ°) data of the bitumen sample can be determined for each test temperature. To evaluate rutting and fatigue resistance of bitumen samples, the PG system defines the limit values of G^* for base and short- and long-term-aged bitumen as 1, 2.2, and 5,000 kPa, respectively.

In the case of complex shear modulus, when the sample reaches the specified values, the device terminates the test. The failing temperature for the samples is defined as the test temperature at which G^* values reach the specified limits for each aging condition. Base and short-term-aged bitumen were tested at high temperatures higher than 46°C, whereas long-term-aged bitumen was tested at intermediate test temperatures in the range of 4°C–40°C. Phase angle, the time lag between response and applied force, is a parameter that gives an idea about viscoelastic properties of the samples and ranges between 0° and 90°. Based on this scale, a perfect elastic material has a phase angle of 0°, whereas 90° refers to perfect viscous material.

Rutting resistance is determined by G^* divided by $\sin \delta^\circ$ ($G^*/\sin \delta^\circ$) and it is used for identification of high-temperature stiffness of base and short-term-aged bitumen. The fatigue factor is calculated with multiplying G^* by $\sin \delta^\circ$ ($G^* \sin \delta^\circ$) and is utilized to assess intermediate temperature stiffness of long-term-aged bitumen. Base and short-term-aged bitumen samples were tested on 25-mm-diameter plates with 1-mm gap, while the long-term-aged samples were tested on 8-mm plates with 2-mm gap following EN 14770 (CEN 2012b).

Thermal Cracking Resistance Tests

The thermal cracking resistance and relaxation characteristics of the bitumen samples were evaluated by BBR test parameters, creep stiffness (St), and m-value. St is the quotient of bending stress and bending strain under constant load and m-value is the slope of the creep stiffness at 60 s. The limitations for St and m-value

of the sample are specified as maximum 300 MPa and minimum 0.300, respectively. The test temperature that satisfies both criteria is defined as the thermal cracking resistance temperature of the bitumen. The BBR test was applied on the long-term-aged bitumen and the duration of the test was 240 s after conditioning. In the present study, tests were performed on each sample at four temperatures, −4°C, −10°C, −16°C, and −22°C, by following EN 14771 (CEN 2012c).

Aging Processes

In this paper, in addition to the base bitumen, rutting and fatigue resistance and thermal cracking behavior of short- and long-term-aged bitumen samples were also tested. Short-term aging refers to the aging of bitumen during production until construction of asphalt pavements. Long-term aging covers the time period from the pavement construction to the end of its service life. It is possible to simulate the short- and long-term aging processes of bitumen in laboratory with RTFO and PAV, respectively. The RTFO test was conducted at 163°C for a duration of 75 min according to EN 12607 (CEN 2015b) and the PAV test can be conducted at 90°C, 100°C, and 110°C temperatures with 20-h test duration by following EN 14769 (CEN 2012a). The PAV test was performed on the residue of short-term-aged samples at 100°C throughout the current study.

Modification Process

After reviewing excessive literature, modification processes and WEOIL contents were optimized. To modify the bitumen, WEOIL was used at 1%–5% by weight of the base bitumen with 1% increment. The bitumen modification process used the following steps:

1. The base bitumen was first heated at 150°C for 2 h in an oven,
2. Then 500 g of bitumen was discharged to an empty metal container,
3. WEOIL in specified proportions was poured into bitumen, and
4. Bitumen and WEOIL were mixed for 45 min with a propeller mixer running at 1,000 rpm at 150°C ± 5°C.

Aging Resistance Analysis

In the context of this study, the base and modified bitumen were aged for short and long terms using RTFO and PAV test methods. To figure out the aging resistance of each sample, the AI was determined for both aging cases with Eq. (1), which was also used by Zhang et al. (2011), Gökalp and Uz (2019), and Li et al. (2019). Because of the different properties of bitumen, AI analysis was performed for all the test methods individually. According to the given equation, a zero value of AI refers to bitumen samples fully resistant to aging. AI values higher than zero indicate that the contribution rate of WEOIL is not sufficient, while negative values represents an excessive contribution rate

$$AI = [T(\text{WEOIL}) - T(\text{BASE})]/T(\text{BASE}) \quad (1)$$

where T = result of the related test used in AI analysis for aged and unaged samples. The unit of T is unique for all test methods, but AI is unitless because it is a ratio of two numbers, which have the same units.

Results and Discussions

In this section of the paper, the DSR and BBR test results, aging index analysis for each aging case, and the optimum rates of WEOIL are presented.

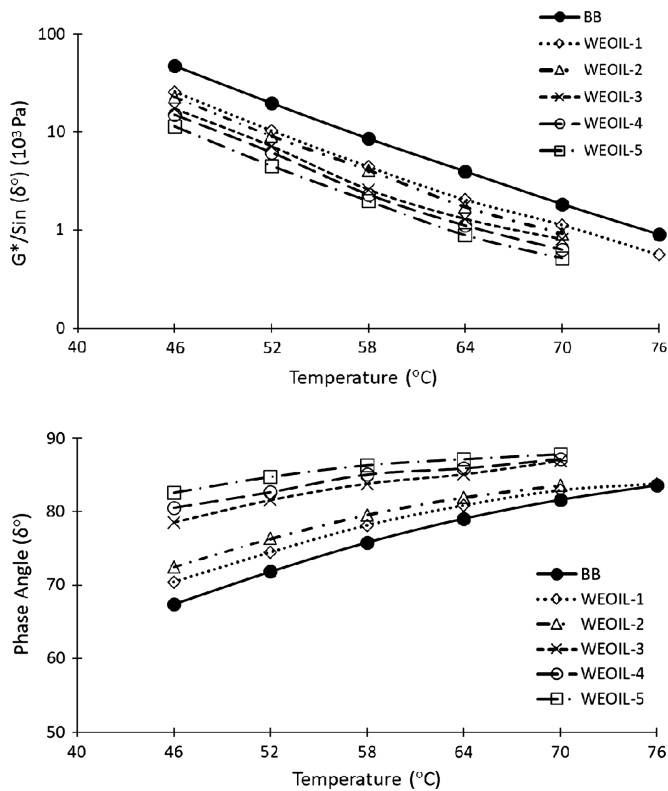


Fig. 1. DSR results for base and modified bitumen.

Rutting Resistance Test

The DSR test was implemented based on the performance grade determination of bitumen samples and elevated test temperatures from 46°C to 76°C. Rutting resistance analyses were performed for unaged and short-term-aged bitumen samples. The tester automatically calculated and checked if compliance is fulfilled and stops when the failure case is reached. For analyzing the rutting resistance of bitumen, Fig. 1 presents the unaged forms of bitumen. The experimental data obtained for short-term-aged base and WEOIL-modified bitumen samples are presented in Fig. 2.

It is apparent in Fig. 1 that the failure temperature decreases with increasing WEOIL content in the base bitumen. The failure temperature of base and WEOIL-1-modified bitumen is determined as 76°C, whereas WEOIL-2, WEOIL-3, and WEOIL-4 are determined as 70°C. WEOIL-5 has the lowest failure temperature, which is 64°C. The phase angle indicates the viscous or elastic behavior of each sample and it can be clearly seen that the addition of WEOIL decreases the viscosity of bitumen, which means the samples with a higher rate of WEOIL have lower rutting resistance. Therefore, the best rutting resistance is observed in the base bitumen. As can be also seen from Fig 1, the initial viscosity of base bitumen is higher than the WEOIL-modified bitumen. The base bitumen is more elastic than the WEOIL-modified bitumen. This discussion can be proven with phase angle of the bitumen samples. The phase angle approaches 90°, which means the bitumen is more elastic, while approaching 0° means the bitumen samples is more elastic.

According to the Superpave PG system, the PG grade is determined as the temperature below failure. In order to remark the PG temperature and the reliability of the two consecutive test results, some statistical information is given in Table 4 based on the related standard. The statistical data confirm that the test provided the specifications requirements and satisfied the reliability of the test results.

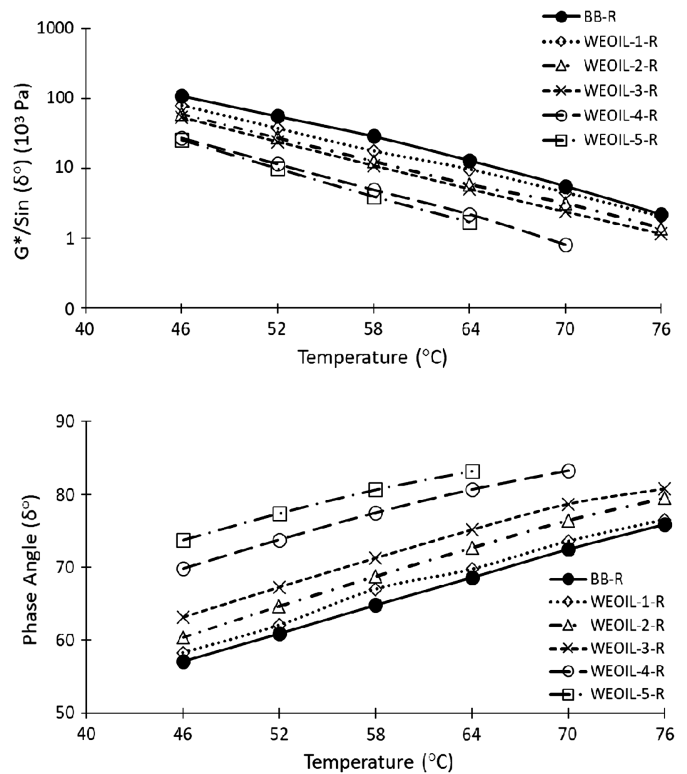


Fig. 2. DSR results for short-term-aged bitumen.

The data for short-term aged bitumen samples in Fig. 2 show a similar tendency as observed in Fig. 1. According to the Superpave PG system, the failure temperature for the base bitumen is expected to be less for the short-term aged samples. The temperature of bitumen below the failure temperature is considered as the highest-performing temperature. Overall, utilizing WEOIL for modification of base bitumen makes the bitumen less resistant to rutting and the rutting resistant decreases with increasing the rate of WEOIL content. The same performance grades are determined for the 1% to 4% WEOIL contents, but the rutting factors differ.

Two consecutive tests were performed with a single operator and the results and the repeatability criteria are given in Table 5. In view of the results presented in Table 5, it is apparent that WEOIL contribution significantly decreases the rutting resistance of bitumen, which was also highlighted by Ackbarali and Maharaj (2011) and Hesp and Shurvell (2013).

One of the objectives of this study is to investigate whether an aging-resistant bitumen can be obtained by using WEOIL without sacrificing the material performance. In this respect, aging index analysis was performed using the DSR and BBR test data obtained from short- and long-term-aged samples. In order to evaluate the short-term aging-resistance capability of WEOIL, AI values were calculated with Eq. (1) and the results are given in Table 6.

According to the AI data in Table 6, it can be concluded that the resistance to short-term aging increases with the increase of WEOIL rate. The effect of test temperature on AI analysis is obvious at lower contents of WEOIL, but the temperature sensitivity diminishes with the increase in WEOIL content. To determine the optimum WEOIL contribution rate for preventing short-term aging, the AI/WEOIL rate graph (Fig. 3) is drawn. The optimum WEOIL rate is calculated in conjunction with the zero value of AI using best-fit line.

Fig. 3 shows AI values for different test temperatures and it can be clearly observed that AI values are converging as the WEOIL

Table 4. Performance grade temperature of base and modified bitumen

| Test parameter | BB | WEOIL-1 | WEOIL-2 | WEOIL-3 | WEOIL-4 | WEOIL-5 |
|---|-----|---------|---------|---------|---------|---------|
| Performance grade (°C) | 70 | 70 | 64 | 64 | 64 | 58 |
| Difference between two tests for G^* (%) | 4.8 | 6.0 | 3.5 | 3.8 | 7.8 | 9.0 |
| Difference between two tests for δ° (%) | 1.6 | 2.0 | 0.9 | 1.0 | 2.1 | 2.9 |

Note: Differences between two tests should not be more than 15% for complex modulus and 3° for phase angle for the failure temperature.

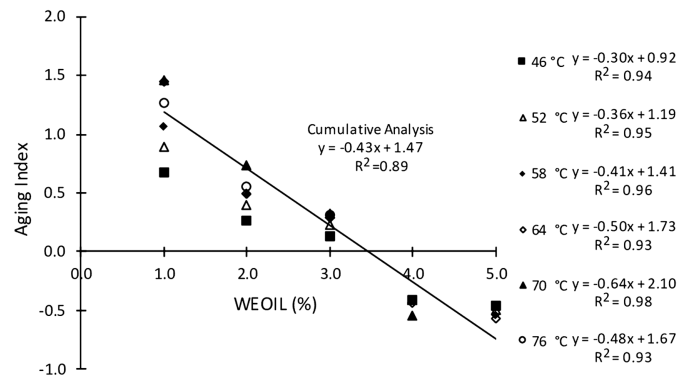
Table 5. Performance grade temperature of short-term-aged base and modified bitumen

| Test parameter | BB-R | WEOIL-1-R | WEOIL-2-R | WEOIL-3-R | WEOIL-4-R | WEOIL-5-R |
|---|------|-----------|-----------|-----------|-----------|-----------|
| Performance grade (°C) | 70 | 70 | 64 | 64 | 64 | 58 |
| Difference between two tests for G^* (%) | 8.2 | 4.5 | 6.5 | 6.8 | 1.8 | 3.0 |
| Difference between two tests for δ° (%) | 2.3 | 1.3 | 1.9 | 1.9 | 1.0 | 0.6 |

Note: Differences between two tests should not be more than 15% for complex modulus and 3° for phase angle for the failure temperature.

Table 6. Aging index values based on rutting resistance for short-term aging

| Temperature (°C) | Samples and aging index | | | | |
|------------------|-------------------------|-----------|-----------|-----------|-----------|
| | WEOIL-1-R | WEOIL-2-R | WEOIL-3-R | WEOIL-4-R | WEOIL-5-R |
| 46 | 0.68 | 0.26 | 0.13 | -0.42 | -0.46 |
| 52 | 0.89 | 0.39 | 0.22 | -0.41 | -0.50 |
| 58 | 1.07 | 0.48 | 0.29 | -0.42 | -0.54 |
| 64 | 1.45 | 0.49 | 0.29 | -0.44 | -0.57 |
| 70 | 1.46 | 0.73 | 0.31 | -0.55 | — |
| 76 | 1.27 | 0.55 | 0.30 | — | — |

**Fig. 3.** Best-fit line analysis according to aging index values for short-term aging.

content increases. For the studied bitumen, all the negative effects of short-term aging can be eliminated and short-term-aging-resistant bitumen can be provided with the addition of approximately 3.5% WEOIL.

Fatigue Resistance Test

Fatigue resistance of bitumen samples was evaluated on the long-term-aged form of bitumen, which was obtained by PAV test. The expected failure temperatures at fatigue test for bitumen graded with PG 70, PG 64, and PG 58 are 34°C–19°C, 31°C–16°C, and 25°C–13°C, respectively. According to this information, tests were performed at temperatures between 7°C and 34°C to determine the fatigue resistance performance of each sample. The DSR stops the

test when failure case is reached. The results of fatigue resistance test for long-term-aged samples are presented in Fig. 4.

It is obvious in Fig. 4 that the failing temperature for base bitumen is 22°C, whereas the failing criteria are reached at 19°C for WEOIL-1-P and WEOIL-2-P. On the other hand, WEOIL-3-P, WEOIL-4-P, and WEOIL-5-P fail at 13°C, 10°C, and 7°C, respectively. The samples with lower failure temperatures indicate better fatigue resistance of bitumen. The fatigue resistance of BB-P is the lowest because it has the highest failure temperature. The PG temperature for long-term-aged samples is determined as the temperature higher than that at which the sample fails. The contribution of WEOIL significantly increases the fatigue resistance of bitumen, which was also highlighted in the studies by Nurul Hidayah et al. (2014) and Liu et al. (2018a).

For reliability of the tests, at least two consecutive tests must be conducted and they must comply with the specified criteria in the standard. Two consecutive tests were performed with a single operator and the results and the repeatability criteria are given in Table 7. According to this information, the results are compatible with the specification requirements.

To evaluate the long-term-aging-resistant capability of WEOIL-modified bitumen, AI analyses were also performed according to the DSR test data obtained from the residue of PAV test samples for test temperatures ranging from 34°C to 7°C. The calculated AI values for different WEOIL contents are presented in Table 8.

It can be seen from Table 8 that long-term-aging resistance increases at higher contribution rates of WEOIL. The AI values increase gradually with the test temperature increase from 7°C to 34°C. However, the temperature sensitivity diminishes with the increasing rate for WEOIL, similar to the short-term-aging case.

According to the AASHTO MP 1 (AASHTO 1998) requirements, PG 70-4 bitumen, which is a Superpave performance grade of base bitumen, is expected to provide limit values (5,000 kPa)

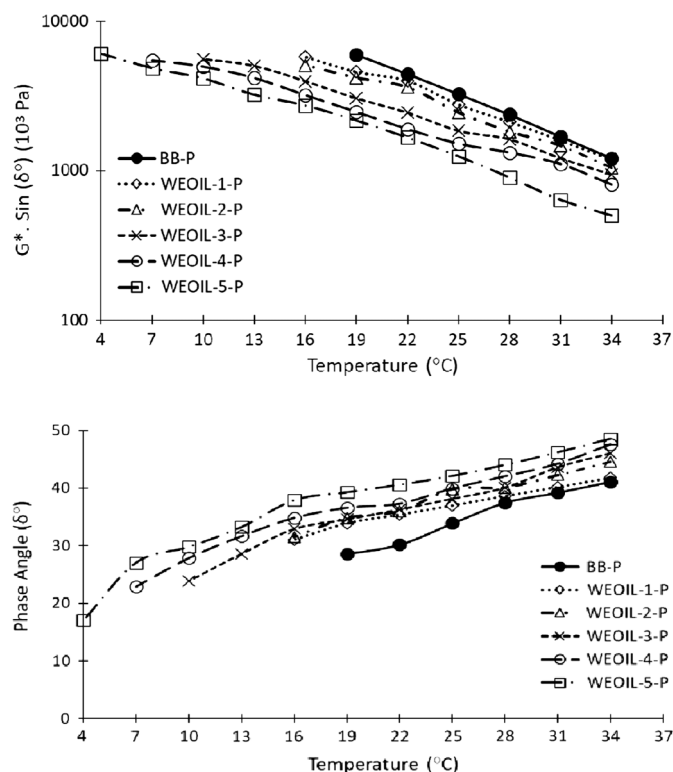


Fig. 4. DSR test results for long-term-aged bitumen.

between 34°C and 19°C for long-term-aged samples. To determine the optimum contribution rate for zero aging index value, the best-fit line analysis is given in Fig. 5 for long-term aging based on 34°C–19°C test temperatures.

From Fig. 5, WEOIL content for long-term-aging-resistant bitumen can be calculated as approximately 6.8% with 0.42 accuracy considering all the test temperatures. However, if the analysis is performed depending on testing temperature, the rates of WEOIL vary between 5.8% and 7.6%. The PG of bitumen used in this study

is 70-4. According to the PG system, the lowest failure temperature is expected to be 19°C. In this respect, the optimum contribution rate of WEOIL can be considered as 5.8%.

Thermal Cracking Resistance Test

Thermal cracking performance is an important parameter that must be determined for the bituminous binders. The base bitumen with PG 70 must be durable at temperatures between -10°C and -40°C considering the PG system. According to this information, the thermal cracking performance of the base and WEOIL-modified bitumen were analyzed at four different temperatures, -4°C , -10°C , -16°C , and -22°C , and the results are given in Fig. 6.

Fig. 6 shows that creep stiffness of base bitumen is higher than WEOIL-modified bitumen. Creep stiffness is decreasing with increasing WEOIL content. Higher creep stiffness indicates lower thermal cracking resistance so it is obvious that bitumen with the highest WEOIL content showed the best thermal cracking resistance. The m -values of all the bitumen samples decrease with reducing test temperatures, but increase with greater WEOIL rates.

The difference between two successive tests with the same operating conditions should not exceed the value of repeatability (r). In this respect, calculated r values for both St and m -value at each test temperature are presented in Table 9.

The data in Table 9 show the accuracy of test results compatible with the requirements of the specification. The grade of each bitumen (base and WEOIL-modified) sample is determined according to the Superpave PG system and is presented in Table 10. It can be seen that increase in contribution rate of WEOIL decreases the rutting resistance, but increases the low-temperature cracking resistance. Adding WEOIL to bitumen changes the performance grade of the bitumen significantly. Similar findings were reported by El-Shorbagy et al. (2019) and Taherkhani and Noorian (2020).

AI analysis was also performed according to the BBR test data obtained from the residue of PAV test samples in order to evaluate thermal cracking-resistant properties of WEOIL-modified bitumen at test temperatures ranging from -4°C to -22°C . As can be seen from Table 11, all the calculated aging index values are negative, which means the addition of WEOIL significantly improved the

Table 7. Performance grade for long-term aged base and modified bitumen

| Test parameter | BB-P | WEOIL-1-P | WEOIL-2-P | WEOIL-3-P | WEOIL-4-P | WEOIL-5-P |
|---|------|-----------|-----------|-----------|-----------|-----------|
| Performance grade ($^{\circ}\text{C}$) | 25 | 22 | 22 | 16 | 13 | 10 |
| Difference between two tests for G^* (%) | 9.2 | 7.5 | 3.1 | 2.8 | 2.8 | 10.0 |
| Difference between two tests for δ° (%) | 2.9 | 2.3 | 0.9 | 0.3 | 0.1 | 2.9 |

Note: Differences between two tests should not be more than 15% for complex modulus and 3° for phase angle for the failure temperature.

Table 8. Aging index values based on fatigue resistance for long-term aging

| Temperature ($^{\circ}\text{C}$) | Samples and aging index | | | | |
|------------------------------------|-------------------------|-----------|-----------|-----------|-----------|
| | WEOIL-1-P | WEOIL-2-P | WEOIL-3-P | WEOIL-4-P | WEOIL-5-P |
| 7 | — | — | — | -0.21 | -0.30 |
| 10 | — | — | 0.14 | 0.02 | -0.15 |
| 13 | — | — | 0.50 | 0.25 | -0.03 |
| 16 | 1.59 | 1.27 | 0.77 | 0.43 | 0.22 |
| 19 | 2.11 | 1.83 | 1.06 | 0.68 | 0.47 |
| 22 | 3.17 | 2.77 | 1.56 | 0.98 | 0.74 |
| 25 | 3.50 | 2.98 | 2.01 | 1.47 | 1.03 |
| 28 | 4.44 | 3.67 | 3.16 | 2.39 | 1.31 |
| 31 | 5.52 | 5.02 | 3.98 | 3.54 | 1.62 |
| 34 | 6.44 | 5.60 | 4.96 | 4.08 | 2.16 |

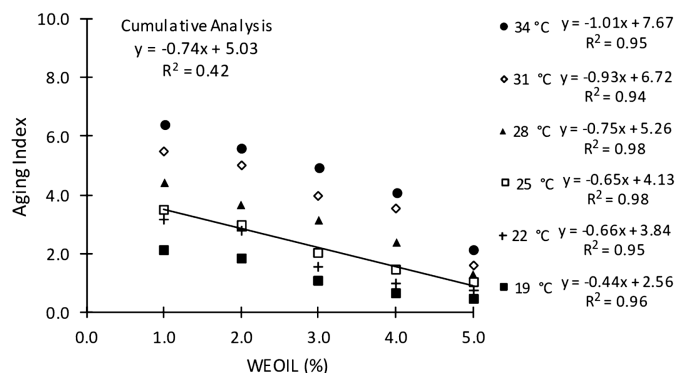


Fig. 5. Best-fit line analysis according to aging index values for long-term aging.

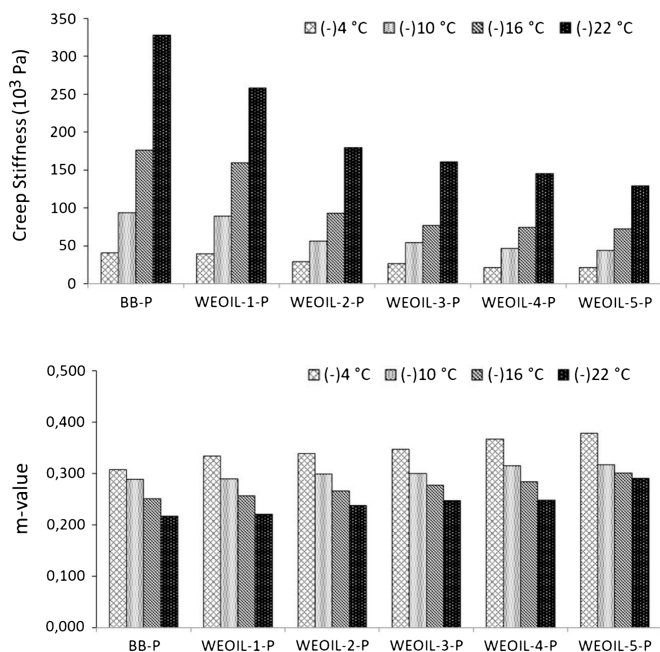


Fig. 6. BBR results for long-term-aged bitumen samples.

low-temperature cracking resistance and changed the performance grade of the bitumen even at the lowest contribution rate. In order to determine the optimum contribution rate for zero AI value, the best-fit line analysis was performed as presented in Fig. 7. It can be seen that, unlike the best-fit line used for analysis done by DSR test results for the two aging cases, exponential curve analysis was used because of higher root-square values.

Table 9. Calculated repeatability value of base and modified bitumen

| Samples | (-)4°C | | (-)10°C | | (-)16°C | | (-)22°C | |
|-----------|--------|---------|---------|---------|---------|---------|---------|---------|
| | St | m-value | St | m-value | St | m-value | St | m-value |
| BB-P | 6.8 | 2.3 | 5.1 | 2.0 | 3.6 | 1.6 | 3.1 | 1.1 |
| WEOIL-1-P | 7.1 | 2.6 | 5.2 | 2.0 | 3.0 | 1.5 | 2.0 | 1.3 |
| WEOIL-2-P | 6.6 | 2.9 | 6.2 | 2.6 | 3.3 | 1.9 | 2.8 | 1.6 |
| WEOIL-3-P | 6.9 | 3.0 | 6.2 | 2.5 | 3.1 | 2.1 | 2.2 | 1.9 |
| WEOIL-4-P | 7.0 | 3.0 | 7.2 | 2.9 | 6.9 | 2.9 | 3.1 | 2.8 |
| WEOIL-5-P | 8.3 | 3.4 | 8.4 | 3.4 | 5.3 | 2.9 | 5.0 | 2.6 |

Note: Estimated repeatability (*r*) is identified as 9 and 4 in percentage for St and m-value, respectively in the related standard.

Fig. 7 shows that optimum rate of WEOIL can be calculated as 0.6% with 0.81 accuracy in cumulative analysis. However, the optimum rate of WEOIL varies from between 0.8% and 0.4% with analysis done for individual test temperatures. Considering the grading temperature of the base bitumen, which is (-)4°C, the optimum WEOIL contribution rate can be calculated as 0.8%. This result proves that the thermal cracking characteristics of bitumen can be improved and the aging effect can be eliminated in terms of thermal cracking resistance with less than 1.0% WEOIL contribution.

Summary, Conclusions, and Recommendations for Future Studies

When the related literature about utilizing waste oils in pavement engineering field is evaluated, it can be easily seen that the interest of those past studies focused on the rejuvenation capability of waste oils in recycling asphalt pavements. The aim of this study is to investigate whether WEOIL can be used to enhance the aging resistance of bitumen. The presented study was designed to determine the optimum amount of WEOIL in bitumen to resist aging for both short- and long-term cases. To evaluate the characteristics of base and WEOIL-modified bitumen and to make a robust comparison between their performances, Superpave tests were conducted on each sample. RTFO and PAV tests were used to obtain short- and long-term-aged samples, respectively. The rutting, fatigue, and thermal cracking resistance performance of samples were presented. AI analysis was performed on the data obtained from DSR and BBR tests for short- and long-term-aging conditions. Based on the aging index analyses, the optimum WEOIL rates for each aging condition were determined.

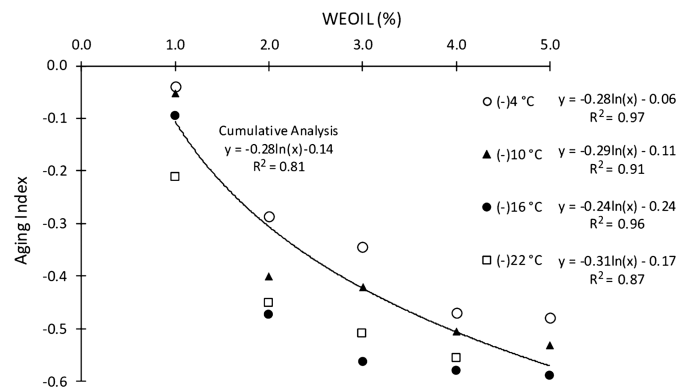
In conclusion, the effect of aging can be significantly decreased, and can even be removed fully with the addition of a certain amount of WEOIL into bitumen. By weight of bitumen, 3.5% WEOIL is required to make the bitumen short-term aging resistant, while 5.8% is required to eliminate the long-term aging effect for the studied bitumen. On the other hand, the rate of WEOIL for eliminating long-term-aging effect on thermal cracking is pointed out as 0.8%, which is significantly lower than the rate found for fatigue resistance. Parallel to the former studies, rheological properties of bitumen change significantly with the contribution of WEOIL, and rutting resistance decreases, while fatigue and thermal cracking resistances increases. These effects may change according to the bitumen type and origin, as well as the environment. In general, harder bitumen requires lower WEOIL rates, while higher rates of WEOIL are required for softer bitumen to recover from the aging effect. Test temperature is found to be an effective parameter to determine the optimum rate of WEOIL due to the thermoviscoelastic behavior of bitumen.

Table 10. Performance grade of base and WEOIL-modified bitumen

| Test parameter | BB | WEOIL-1 | WEOIL-2 | WEOIL-3 | WEOIL-4 | WEOIL-5 |
|-----------------------------|------|---------|---------|---------|---------|---------|
| Superpage performance grade | 70-4 | 70-4 | 64-10 | 64-10 | 64-10 | 58-16 |

Table 11. Aging index results based on BBR test for long-term aging

| Temperature (°C) | Samples and aging index | | | | |
|------------------|-------------------------|-----------|-----------|-----------|-----------|
| | WEOIL-1-P | WEOIL-2-P | WEOIL-3-P | WEOIL-4-P | WEOIL-5-P |
| -4 | -0.04 | -0.29 | -0.35 | -0.47 | -0.48 |
| -10 | -0.05 | -0.40 | -0.42 | -0.51 | -0.53 |
| -16 | -0.10 | -0.47 | -0.56 | -0.58 | -0.59 |
| -22 | -0.21 | -0.45 | -0.51 | -0.56 | -0.61 |

**Fig. 7.** Best-fit line analysis according to aging index values for long-term aging using BBR results.

The present study confirms the previous findings and gives additional evidence to the fact that WEOIL can be utilized for producing aging-resistant bitumen. Despite numerous methods conducted by recycling WEOIL, using it in bitumen modification in order to produce aging-resistant bitumen can be a sustainable alternative and effective method with ecological and economic benefits. Moreover, production of aging-resistant bitumen using WEOIL ensures protection of bitumen resources and environmental benefits for all creatures living on Earth and economical gains not only for individuals but also for governments.

This research has brought about some additional questions for further investigation. For example, different bitumen types, either in origin or in grade, should be studied because only one type of bitumen performance was evaluated in the current study. Similar to bitumen, we evaluated only one type of WEOIL and the results are limited to the studied WEOIL performance. Therefore, WEOIL in different sources should be examined further. Additionally, the effect of the environment can be studied in detail due to the temperature-sensitivity characteristics of bitumen. Investigating the effect of WEOIL modification on asphalt mixture performance, which is not included in this study, is also recommended.

Data Availability Statement

No data, models, or code were generated or used during the study.

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