

# Article Evaluation of Low- and Intermediate-Temperature Performance of Bio Oil-Modified Asphalt Binders

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Abstract: Fatigue cracking and low-temperature cracking are two major distresses that occur in asphalt pavements. Fatigue cracking is a load-associated distress caused by the tensile stresses at the bottom/top of the asphalt concrete (AC) layer due to repeated traffic loading. On the other hand, low-temperature cracking occurs when tensile stresses built up with in the AC layer at low temperatures exceed the tensile strength of that layer. In this study, the performance of date seeds oil bio-modified asphalt binders (DSO-BMB) is evaluated against fatigue and low-temperature cracking. The DSO-BMBs are prepared using volume ratios of 1.5, 2.5, 3.5, 4.5, and 5.5% date seeds oil-to-asphalt binder. The base asphalt binder used in the study is a 60/70-penetration grade with a Superpave performance grade (PG) of PG 64-16. The dynamic shear rheometer (DSR) standard test was used to assess the fatigue performance of the bio-modified binders (BMBs), while the bending beam rheometer (BBR) test was used to test the BMBs for low-temperature performance. In addition, the DSR linear amplitude sweep (LAS) test was used to evaluate the fatigue tolerance behavior of the DSO-BMBs. The analysis and results of the study showed that the bio-oil enhanced the lowtemperature performance. The low PG grade improved from -16 °C for the control asphalt binder to -28 °C for the BMB. Additionally, the fatigue resistance of the BMBs was improved as illustrated by the damage-characteristic curves of the modified asphalt binders from the visco-elastic continuum damage (VECD) analysis and the increase in the number of cycles to fatigue failure (Nf).

**Keywords:** bio oil; bio binders; date seeds oil; fatigue cracking; low-temperature cracking; BBR; DSR; LAS; RTFO; PAV; VECD; asphalt binders

# 1. Introduction

Cracking is one of the major distress modes for asphalt pavements and is categorized in two main groups: (1) load-associated (fatigue cracking) and it occurs when tensile stresses due to repetitive traffic loading exceed the tensile strength of the material, creating microcracks that grow and coalesce into macrocracks that lower pavement smoothness and the structural integrity [1]. However, there are two types of fatigue cracking: top–down and bottom–up cracking. The top–down cracking is most common in thick pavements as reported by Canestrari & Ingrassia [2]. (2) Non-load-associated (thermal cracking) which is common in the cold climate regions [3], and it occurs when the thermal stress that builds up during cooling events in the pavement exceeds the tensile strength of the AC layer. Cracked pavements allow water to infiltrate underlying pavement layers, further weakening the pavement structure and leading to a rougher ride and shorter service life [4]. The need to ensure the safety of the roads and pavement without compromising costs and environmental effects is attracting great interest in the asphalt research community.



Citation: Alattieh, S.A.; Al-Khateeb, G.G.; Zeiada, W. Evaluation of Lowand Intermediate-Temperature Performance of Bio Oil-Modified Asphalt Binders. *Sustainability* **2021**, *13*, 4039. https://doi.org/10.3390/ su13074039

Academic Editor: Nuha Mashaan

Received: 15 February 2021 Accepted: 20 March 2021 Published: 5 April 2021

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**Copyright:** © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Multiple sustainable techniques have been implemented to reduce the use of petroleumbased asphalt or enhance the performance of the pavement. Bio binders have extensively become research alternatives to the conventional asphalt binder as a sustainable alternative and have proved to be effective in minimizing long-term aging related distresses and reducing the need of petroleum asphalt binder [5,6]. The bio agent was found to reduce the air void content in the compacted mixture, especially when blended with reclaimed asphalt pavement (RAP) binder due to the change in the visco-elastic properties of asphalt binder and the improvement in the compaction process of asphalt mixtures [7].

Bio-oil was used in different forms in the literature and was classified as per its function [6]. The bio-oil was considered as a direct alternative binder when the asphalt binder is composed of 100% bio binder as in [8,9], an extender when it is more than 25% of total weight of the modified binder [10,11], and as a modifier when the bio-oil content is less than 10%, as reported in [12–15].

Asphalt binder blended with bio-oil extracted from tall oil had similar fatigue performance compared to the conventional binder with the same consistency (i.e., penetration). In addition, the bio binder was able to improve the self-healing potential compared to the conventional binder, especially in the aged conditions [5]. In another study [16], waste cooking oil (WCO) was used as a modifier for asphaltic materials indigenous to Trinidad (Trinidad lake asphalt (TLA) and Trinidad petroleum bitumen (TPB)) were used to assess the fatigue and rutting resistance behavior of the modified asphalt binder. This blend results in changes in the rheological properties as demonstrated by the changes in the phase angle and complex modulus. The results demonstrate that the values of  $G^*sin\delta$  for the blends decreased as the testing temperature and oil content increased and this indicates that the fatigue cracking resistance was enhanced as temperature increased, based on their study. As a result, at lower temperatures the modified binder exhibits better thermal cracking performance with a higher waste cooking oil (WCO) concentration.

A soybean-derived rejuvenator was used [17] to modify asphalt binder (PG 58–28), blended with RAP binder. Results showed an improvement in the fatigue life of the pressure aging vessel (PAV)-aged binders with the addition of the soybean rejuvenator at temperatures below 25 °C. A temperature-frequency sweep was performed at temperature and frequency ranges of 10 °C to 34 °C and 0.6 to 100 rad/sec, respectively. The low temperature grade was determined through the bending beam rheometer (BBR) test and was found to be reduced with the addition of the modified binder to the PAV binder, from -10.8 °C to -22.3 in the case of RAP+12% modified PG 58–28. The number of cycles to failure from the linear amplitude sweep (LAS) test for the modified binder was increased dramatically compared to the control RAP binder. The impact of rejuvenation on fatigue life was more pronounced at lower temperatures. It is evident that the improvement attained with the addition of the rejuvenator is more prominent with decreasing temperatures. As the temperature decreased from 31 °C to 25 °C, the RAP binder showed a considerable reduction in fatigue performance compared to the rejuvenated RAP binders. These results provide evidence of the improved fatigue resistance of the rejuvenated binders.

The linear amplitude sweep test (LAS) was conducted to assess the fatigue tolerance of date seed oil (DSO) bio-modified asphalt binder at intermediate temperatures [18]. The results indicated that after the addition of 1.5% DSO to the asphalt binder,  $\alpha$  was reduced, which is an indicator to the enhanced fatigue resistance of the bio-modified binder. The visco-elastic continuum damage (VECD) curve shows that the damage parameter, C, was reducing at higher damage intensity which means that the bio-binder with higher DSO content has higher fatigue tolerance compared to the control binder. In addition, the number of cycles to failure was increased by 25% compared to the control binder. The BBR results of the bio-modified asphalt binder in [19] showed an improvement in the critical failure temperature compared to the control binder, but was not significant enough to change the low temperature grade as the content is low. Another research study compared the effects of bio-oil as a partial replacement for asphalt binder and as a rejuvenating agent [20]. The viscosity of the blends was reduced by 33.5% and 44.6% by adding 5% and 10% DSO, respectively. The ultimate results of the LAS test showed that the N<sub>f</sub> value was found to be 9200 cycles as a maximum value when the DSO content was 10% compared to the other tested blends, whether the blend is rejuvenated or modified with DSO only. The increase in the RAP content, i.e., 30% and more, should be carefully utilized. It was also found that the 5% or 10% DSO with 10% or 20% RAP had an improved fatigue tolerance and had similar values to the base binder. The fatigue performance determined from the four-point beam fatigue test of another blend sourced from waste wood was found to be improved significantly when compared to the control binder [15]. The fatigue life was enhanced by 85.4% compared to the control binder by adding 5% bio-oil under 200 microstrain. Further statistical analysis showed that bio-oil content had a significant effect on the indirect tensile strength and fatigue lives of the modified binder.

The fatigue parameter obtained from dynamic shear rheometer (DSR) testing in a study by Ingrassia et al., [21] was reduced significantly by more than 90% just with the addition of 10% wood-based bio-oil to the asphalt binder. The aging behavior of the tested blends were explored as well. It was concluded that blends with bio binder did undergo a lower aging rate than the corresponding conventional binder, which in return lowered the cracking susceptibility.

Waste vegetable oil (WVO) was used in another study [22] to evaluate the characteristics of the bio-modified asphalt binder, including DSR and BBR tests. According to the DSR results at intermediate temperatures,  $G^* \sin \delta$  was reduced by 8% with the addition of 1% WVO at 28 °C. In addition, with the increase in temperature from 28 °C to 31 °C, the  $G^* \sin \delta$  for the 1% modified binder was reduced by 30%. However, all tested modified binders at the intermediate temperature testing range (28–34 °C) had  $G^* \sin \delta$  values less than 5000 kPa. The addition of 1% WVO resulted in decreasing the creep stiffness and increasing the m value by 30% and 18%, respectively. Increasing the percentage of added WVO had a significant effect on the selected low temperature performance.

An amount of 5% waste cooking oil (WCO) was added to asphalt binder mixed with different rates of fillers in a study [23]. The fatigue resistance behavior for the modified binder obtained from the DSR testing at the intermediate temperature range (10–22 °C) showed that  $G^* \sin \delta$  decreased by 12%. The BBR results showed a reduced stiffness at the same testing temperature. The control binder resists low temperature cracking up to -6 °C, while modified sample was able to resist low temperature cracking up to -22 °C. The incorporation of bio-oil derived from corn stover improved the strength of the asphalt binder modified with different ratios of filler at -10 °C in a study [24]. Bio-oil has an evident influence on the mechanical property of asphalt mastic at low temperature condition. The maximum loads of the modified asphalt binder were larger (~300%) than those of neat asphalt mastic at the same filler-to-binder ratio. The obtained energy required (E<sub>S</sub>) to break the bio-modified material was double than that without the bio-oil, due to the direct relation of energy to deflection and maximum load, in which the modified binder had higher maximum load and lower deflection, at the same filler to binder ratio.

Based on the reviewed literature, the evaluation of the fatigue and thermal behavior of bio-modified asphalt binder was limited. Therefore, this research aimed at a comprehensive evaluation of fatigue and thermal cracking performance of date seeds oil bio-modified asphalt binder (DSO-BMB) through multiple tests including DSR, BBR and LAS tests. The main objectives of this study were to investigate the fatigue behavior of DSO-BMB at different oil contents and temperatures; assess the significance of DSO on the fatigue behavior of the bio-modified binder; and conduct sensitivity analysis on the different fatigue resistance parameters and the correlation between these parameters.

## 2. Methodology

The present study provides a comprehensive fatigue and thermal cracking assessment of DSO-BMB. The methodology discussed in this section includes the materials, preparation of bio-modified binder (BMB) procedure, aging conditions, followed by the laboratory tests for the characterization of asphalt binders.

## 2.1. Preparation of the BMB Blends

The DSO-BMBs were prepared at 1.5%, 2.5%, 3.5%, 4.5% and 5.5% volume ratio of DSO following a prior study using 60/70-penetration-grade asphalt binder [18,25].

The DSO was extracted from waste date seeds collected from local dates factories (Dubai Dates Factory, Sharjah, United Arab Emirates). The seeds were cleaned and dried prior to the grinding process. DSO was extracted using the Soxhlet apparatus shown in Figure 1 through the Goldfisch/Randall technique. The neat asphalt binder is a commonly used material for the construction of highway asphalt pavements in the United Arab Emirates. The asphalt binder was obtained from MENA Energy, Dubai. The physical characteristics of the asphalt binder were tested in the Transportation and Pavement Laboratory at the University of Sharjah. The values of 62, 49.10 °C, and 404.38 cP were obtained for the penetration, softening point, and rotational viscosity for the control (neat) asphalt binder, respectively. The original performance grade (PG) for the neat asphalt binder was PG 64–16. The DSO-BMB blends underwent a short-term aging (RTFO) followed by a long-term aging (PAV) process as per the American Society for Testing and Materials (ASTM) D2872 [26] and ASTM D6521 [27], respectively. For the long-term aging, the conditioning temperature was selected as per the Superpave that specified a 110 °C temperature for desert climate regions.



Figure 1. Fat extraction apparatus (Soxhlet apparatus).

### 2.2. Characterization of Aged DSO-BMB

Three tests were conducted on the aged DSO-BMBs at different temperatures to evaluate the fatigue and thermal behavior of the DSO-BMB. Table 1 shows the testing matrix used in the assessment of the DSO-BMB.

Test	Standard Test Method	Temperatures (°C)	Obtained Parameter
DSR	ASTM D7175 [28]	13–31	$ G^* \sin\delta$
BBR	ASTM D6648 [29]	0, -6, -12, -18, -24	Stiffness, m-value
LAS	AASHTO TP101 [30]	Intermediate temperature and 20	D <sub>f</sub> , N <sub>f</sub>

Table 1. Testing matrix used in the assessment of date seeds oil bio-modified asphalt binder (DSO-BMB).

## 2.2.1. Dynamic Shear Rheometer (DSR) Test

The dynamic shear rheometer (DSR) test was used to assess the fatigue performance of the aged DSO-BMB following the ASTM D7175 using the DSR device using an oscillatory shear loading frequency of 1.59 HZ (10 rad/s). DSO-BMB were sampled using a disk of  $8 \pm 0.1$  mm diameter and 2 mm thickness (Figure 2) and tested at 31, 28, 25, 22, 19, 16, and  $13 \pm 0.1$  °C which corresponds to the intermediate temperature range. The fatigue performance parameter ( $|G^*|\sin\delta$ ) was then determined. The Superpave limits the fatigue parameter to 5000 kPa at the intermediate temperatures.



Figure 2. Dynamic shear rheometer (DSR) 8 mm sample disk.

2.2.2. Bending Beam Rheometer (BBR) Test

The bending beam rheometer (BBR) provides a measure of low temperature flexuralcreep stiffness and relaxation properties of asphalt binder. The test was conducted following the ASTM D6648 using samples preprepared as per the ASTM D6521 (Figure 3). A constant load of 980  $\pm$  50 mN was applied to for 240 s at the minimum testing temperature (0, -6, -12, -18, and -24 °C).  $\pm$  0.1 °C Superpave specified a maximum stiffness value of 300 MPa and a minimum m-value of 0.3 for the selection of the low temperature performance grade (PG).



Figure 3. Bending beam rheometer (BBR) (a) beam sample (b) beam mold.

### 2.2.3. Linear Amplitude Sweep (LAS) Test

The linear amplitude sweep (LAS) test was conducted to evaluate the fatigue behavior of the tested asphalt binders under cycling loading using the DSR following the AASHTO TP 101. LAS tests were conducted at the intermediate temperature determined earlier from the high and low performance grades and 20 °C as well for direct comparison purposes. To quantify the damage tolerance, a rigorous viscoelastic continuum damage approach was used to calculate fatigue resistance from rheological properties and amplitude sweep results. This test consisted of two phases, the frequency and strain amplitude sweeps. The frequency sweep test was used to determine the alpha parameter ( $\alpha$ ). The test was performed at the selected temperature, where an oscillatory shear loading at constant strain (0.1%) was applied over 12 frequency ( $\omega$ ) values that varied from 0.2 to 30 Hz. The complex shear modulus ( $|G^*|$ , Pa) and phase angle ( $\delta$ , degrees) were recorded at each frequency level. The sample was then tested using a series of oscillatory load cycles at systematically increasing strain amplitudes (0.1 to 30%) at a constant frequency (10 Hz); this is known as the amplitude sweep test. The strain ( $\gamma$ ) was increased from 0.1% to 30% over 3100 loading cycles for a total time of 310 s, and the peak shear strain and peak shear stress (35%) were recorded every 10 cycles (1 s), along with the complex shear modulus ( $|G^*|$ , Pa) and phase angle ( $\delta$ , degrees). The procedure for computing the outputs of the LAS test followed the visco-elastic continuum damage (VECD) theory. The analysis of the results was carried on an Excel sheet developed by the Modified Asphalt Research Center and WISCONSIN [31]. Key parameters and their equation are summarized in Table 2.

Table 2. Summary of equations used in the analysis of LAS results.

Test Protocol	Parameter	Notation	Equation
Frequency Sweep Test	Storage Modulus	$G'(\omega)$	$\log G'(\omega) = m \left(\log \omega\right) + b$
1 June June J	Alpha Parameter	α	$\frac{1}{m}$
	Accumulated Damage	D(t)	$D(t) = \cong \sum_{i=1}^{N} [\pi \gamma_0^2 (C_{i-1} - C_i)]^{\frac{\alpha}{1+\alpha}} (t_i - t_{i-1})^{\frac{1}{1+\alpha}}$
Amplitude Sweep Test	Complex Modulus Ratio	<i>C</i> ( <i>t</i> )	$C(t)=rac{ G^* (t)}{ G^* _{initial}}$ ,
	Damage at Failure	$D_f$	$D_f = \left(\frac{C \ at \ peak}{C_1}\right)^{1/C_2}$
	Fatigue Model Parameter 1	Α	$A = \frac{f(D_f)^k}{k(\pi I_D C_1 C_2)^{\alpha}}$
	Fatigue Model Parameter 2	В	$B=2\alpha$
	Number of Cycles to Failure	$N_f$	$N_f = A_{35}(\gamma_{max})^{-B}$

# 3. Results and Discussion

The rheological properties of the unaged DSO-BMB were represented in terms of penetration, softening point and rotational viscosity, as seen in Table 3. The significance change in these parameters were due to the low density of the DSO (0.88 g/mL) mixed with asphalt binder [18], which resulted in a reduction in the penetration and the viscosity of the DSO-BMB. Additionally, the pre-determined continuous high temperature PG was determined from the DSR testing at different temperatures ranging from 52 to 70 °C.

The results showed that the PG was reducing with the increase in the DSO content. This reduction was attributed to the loss in the  $|G^*|$  of the bio-modified asphalt binder at higher oil contents. In fact, the 5.5% BMB was almost flowable at room temperature!

BMB Blend	Penetration @25 °C (dmm)	Softening Point (°C)	Rotational Viscosity @135 °C (Pa.s)	PG (°C)	Continuous PG (°C)
0.0%	54	49.93	0.583	64	67.40
1.5%	66	49.75	0.381	64	64.83
2.5%	84	45.03	0.323	58	63.16
3.5%	102	43.5	0.323	58	61.54
4.5%	117	42.38	0.298	58	59.95
5.5%	139	41.03	0.263	58	58.41

Table 3. Consistency and viscosity results of the unaged bio-modified binder (BMB) blends.

The continuous PG was determined from plotting the DSO content and rutting factor  $(|G^*|/\sin\delta)$  at different temperature to satisfy the American Association of State Highway and Transportation Officials (AASHTO) M320 requirements at both aging levels, unaged and short-term aged binders, where  $|G^*|/\sin\delta$  should be more than 1 and 2.2 kPa for unaged and RTFO asphalt samples. The equations of lines were set to find the maximum temperature ( $T_{max}$ ) that satisfies both requirements. This is also similar to Foroutan Mirhosseini et al. [20] where the continued PG was at least decreased by 6 °C compared to the base binder.

Sun el al., [14] had close results to the obtained results, the only difference being that the binder used was stiffer as compared to this research (asphalt binder with a penetration grade of 40/60). Both binders jumped at least one penetration grade with the addition of the same amount of bio-oil.

# 3.1. Temperature Sweep Test Using DSR

# 3.1.1. Fatigue Parameter (G\*sinδ)

The DSR was used to estimate the viscoelastic behavior of the DSO-BMB at the intermediate temperature range from 13 °C to 31 °C. The fatigue parameter (G\*sin $\delta$ ) shown in Figure 4 indicates that the fatigue parameter was decreasing with the increase in the oil content and temperature. The Superpave limits the fatigue parameter to 5000 kPa at the intermediate temperature. It can be observed that the after adding 1.5% DSO the intermediate temperature range was reduced to beneath 22 °C. This reduction is attributed to the reduction in the complex modulus at the testing temperature caused by the softness of the binder. This concludes that the low temperature grade (PG) is reducing as well.



Figure 4. Fatigue parameter ( $|G^*|\sin\delta$ ) results for PAV-aged BMB blends.

## 3.1.2. Significance of Temperature and Oil Content on the Fatigue Parameter (G\*sinδ)

Asphalt is a viscoelastoplastic material [32] which is affected by the temperature and oil content. Specifically, the fatigue factor was decreasing when both parameters are increased. In order to analyze the significance of both parameters on the asphalt binder, regression analysis and ANOVA tests were conducted. Multiple regression types were conducted to find best fit including linear and nonlinear regression. As it can be seen from Table 4, the temperature (*T*) was more significant than the oil content (*OC*) as the *p*-value from the ANOVA test is lower than that of the oil content in both regression models, however, both *x* and *T* are significant parameters in changing the fatigue parameters. Therefore, as a result, the fatigue factor logarithm of the matrix BMB has good linear correlation ( $\mathbb{R}^2$  of 0.97) in the case of the logarithmic values of G\*sin $\delta$  and temperature.

<b>Table 4.</b> Fitting equations of fatigue parameter ( $G^*\sin\delta$ ) using DSR results.

Model	Equation	R <sup>2</sup>	ANOVA Test (p-Value)
Linear	$G^* \sin \delta = 16,129.35 - 85,897.40C - 423.441T$	0.91	$OC: 5.74  imes 10^{-10} \ T: 4.31  imes 10^{-15}$
Nonlinear	$G^* \sin \delta = -20,685 \log(T) - 85,897.4OC + 342,23.37$	0.93	$OC: 7.69 \times 10^{-15}$ Log $T: 4.54 \times 10^{-22}$
	$\log(G^* \sin \delta) = -2.33 \log(T) - 9.610C + 6.87$	0.97	$OC: 6.57 \times 10^{-21}$ LogT: 7.62 × 10 <sup>-29</sup>

# 3.2. Bending Beam Rheometer Test Results

3.2.1. Stiffness, and Low Temperature PG

The BBR test was conducted to estimate the stiffness of the asphalt binder at different low temperature ranges varying from zero to -30 °C. Figure 5 shows the measured stiffness (at 60 sec) of BMB at different testing temperatures. The results indicated that the stiffness of the binder increases with the decrease in the testing temperature, however, the modified binder has a lower stiffness than the control binders. However, adding 1.5% bio-oil reduced the stiffness of the binder by 33.39%, 36.90%, and 19.44% compared to the 0.0% BMB at of 0, -6, and -12 °C, respectively. On the other hand, adding 5.5% oil to the binder reduced the stiffness by more than 70% at the test temperature of -6 compared to the 0.0% BMB. Figure 6 shows the results of the m-value of the binder increased the mvalue. However, the reduction in the temperature induced a reduction in the m-value. The 1.5% BMB had an increased m-value by 10.30%, 13.50% and 19.85% at the temperatures of 0, -6, and -12 °C, respectively, compared to the 0.0% BMB. In addition, the corporation of 5.5% DSO increased the m-value by 43.09% and 43.07% at the temperatures of -6 and -12 °C compared to the 0.0% BMB.

As for the stiffness requirement for the low PG, the stiffness at testing temperature shall be under 300 MPa and m value above 0.300, to address the low PG of tested asphalt binders, both criteria should be valid. Table 5 highlighted the temperatures where at least one of the two parameters or both parameters were not satisfied in addition to the corresponding PG, intermediate temperature, and the fatigue parameter obtained from DSR earlier. It can be obtained that the low temperature PG was reduced with the addition of the DSO to the asphalt binder; the obtained low temperature PG of the 5.5% BMB was -28 compared to -16 for the control binder. In summary, the DSO content of 1.5% performed the best in terms of maintaining the high temperature of 64 °C PG and extending the low temperature PG to -22 °C. further addition of DSO reduced both low and high temperature PGs of the BMB.



Figure 5. Stiffness results from BBR test.



Figure 6. The m-value results from BBR test.

Table 5. PG results with associated G\*sin  $\delta$  parameter.

BMB Blend	Failed Criteria	PG (°C)	Intermediate Temperature (°C)	G* sinδ (kPa)
0.0%	Temp: -12 m-value: 0.267	64–16	28	3226.25
1.5%	Temp: —18 Stiffness: 377.10 MPa m-value: 0.274	64–22	25	3330.10
2.5%	Temp: -18 m-value: 0.279	58–22	22	3915.77
3.5%	Temp:	58–22	22	3095.91
4.5%	Temp: -24 Stiffness: 429.28 MPa m-value: 0.253	58–28	19	4732.19
5.5%	Stiffness: 381.08 MPa m-value: 0.289	58–28	19	2604.02

The low PG grade as per [20] was reduced from -22 to -28 °C for the bio binder containing 5% DSO; similarly, our PG was reduced to -28 instead of -16 °C with the addition of 5.5% DSO.

## 3.2.2. Significance of Temperature and Oil Content on BBR Results

The stiffness values of the DSO- BMB were found to decrease at higher DSO contents and temperature. To assess the significance of each parameter on the stiffness of the BMB at low temperature, an ANOVA test was conducted. Table 6 shows that the temperature (T) was more significant than the oil content (*OC*) as the *p*-value from the ANOVA test was lower than that of the DSO content in both regression models, however, both *OC* and *T* are significant parameters in changing the stiffness parameter. Multiple regressions were adopted to estimate the best fit of the stiffness as linear or nonlinear models, and it was obtained that the stiffness factor logarithm of the matrix BMB had good correlation ( $R^2$  of 0.98) in case of the normal values of DSO content and temperature. However, the m-value models did not have different  $R^2$  values in both regression models.

Parameter	Model	Equation	R <sup>2</sup>	ANOVA Test ( <i>p</i> -Value)
Stiffness	Linear	Stiffness = -2484.3OC - 18.7T + 37.27	0.94	$OC: 3.4  imes 10^{-6} \ T: 4.5  imes 10^{-13}$
	Nonlinear	log(Stiffness) = -8.02OC - 0.05T - 1.78	0.98	$OC: 3.97  imes 10^{-10} \ T: 1.21  imes 10^{-16}$
m-value	Linear	$m \ value = 2.18OC + 0.0085T + 0.37$	0.94	$OC: 2.87 \times 10^{-10} \ T: 7.24 \times 10^{-13}$
	Nonlinear	$log(m \ value) = 2.74OC + 0.011T - 0.43$	0.94	$OC: 6.1  imes 10^{-10} \ T: 1.07  imes 10^{-12}$

Table 6. Fitting equations of stiffness parameter using BBR results.

### 3.3. Linear Amplitude Sweep (LAS) Test Results

#### 3.3.1. Shear Strain and Stresses

The shear stresses and strains obtained from the raw results of the LAS test are shown in Figure 7a,b at the intermediate temperatures and at a fixed temperature of 20 °C. At 20 °C, the results indicated that the effective shear stress for the BMBs was reduced with the addition of the DSO to the asphalt binder. The maximum shear stress occurred at higher shear strains for the asphalt binders with higher DSO content. Based on Figure 7b, it is apparent that the energy required for the modified asphalt binders to resist fatigue cracking is smaller. For instance, the 5.5% DSO-BMB had the lowest peak effective shear stress compared to the other asphalt binders. The maximum effective shear strain at 20 °C was approximately in the range of 5% to 10% (Figure 7b).

However, when the LAS test was conducted at the intermediate temperature for each asphalt binder based on the PG grade, the results were slightly different. Based on Figure 7a, at very low DSO contents such as 1.5% and 2.5%, the peak effective shear stress was higher than that of the control asphalt binder, which indicates that these two-bio-oil-modified asphalt binders had lower fatigue resistance than the control asphalt binder. On the other hand, at higher DSO contents including 3.5%, 4.5%, and 5.5%, the peak effective shear stress value was lower than that of the control asphalt binder, which indicates that these modified asphalt binders had improved fatigue resistance compared to the control asphalt binder. The maximum effective shear strain at the intermediate temperature was approximately in the range of 10% to 15% (Figure 7a).



Figure 7. Effective shear strain vs. stress at (a) intermediate temperature, and (b) 20 °C.

# 3.3.2. VECD Curves and Fatigue Behavior

The fatigue behavior of the DSO-BMBs was estimated through the LAS test at the intermediate test temperature. The VECD graph (Figure 8) illustrates the damage intensity versus C parameter at the intermediate temperatures. The C damage parameter is defined as the ratio of the complex modulus value at any time to the initial complex modulus value of the binder (the value of G\*sin $\delta$  at any time/initial value of G\*sin $\delta$ ).

Tables 7 and 8 shows the LAS results tested at the intermediate temperature and at a fixed temperature of 20 °C. The  $\alpha$  is defined as the slope of applied strain, which means that higher values of  $\alpha$  indicates more sensitivity to fatigue damage. The results show that the DSO-BMB that contains higher DSO content had lower  $\alpha$ , this reduction was due to two reasons; (1) the DSO-BMB is softer than the control asphalt binder and it is expected to perform better in terms of fatigue resistance, (2) the intermediate testing temperature for the BMB was lower than the control asphalt binder. From these two tables, the N<sub>f</sub> values generally increased with the increase in the DSO content. This trend was very obvious when the temperature was fixed at 20 °C for all tested asphalt binders. However, when the asphalt binders were tested at the intermediate temperature (which is different from

binder to binder based on the PG grade), the N<sub>f</sub> values also increased with the increase in DSO content but the variability is relatively higher. The percentage increase in the N<sub>f</sub> values compared to that of the control asphalt binder ranges from about 7.4% to 27.2% at the fixed temperature 20 °C.



Figure 8. VECD damage curve from amplitude for BMBs at the intermediate temperature.

BMB Blend	α	N <sub>f</sub> @5.0%	$D_{\mathrm{f}}$	
0.0%	2.04	1361	33	
1.5%	1.94	1224	33	
2.5%	2.02	1137	32	
3.5%	1.81	1441	38	
4.5%	1.98	1342	35	
5.5%	1.97	1478	36	

Table 7. LAS results at intermediate temperature.

**Table 8.** LAS results at test temperature of 20 °C.

BMB Blend	α	N <sub>f</sub> @5.0%	D <sub>f</sub>
0.0%	2.45	1079	28
1.5%	2.17	1284	32
2.5%	2.10	1159	31
3.5%	1.99	1333	35
4.5%	1.95	1351	35
5.5%	1.93	1373	35

3.3.3. Significance of Temperature and Oil Content on the LAS Parameters

The N<sub>f</sub> was found to be more affected by the DSO content in the BMB blend rather than the test temperature. The significance of each parameter was obtained through the *p*-value from the ANOVA test in Table 9, which shows that the oil content (*OC*) is more significant than the temperature (T) as the *p*-value is lower than that of the temperature in both regression models. Multiple regression models were used to estimate the best fit of the N<sub>f</sub> as linear or nonlinear models; it was obtained that the linear N<sub>f</sub> of the matrix BMB has a satisfactory correlation ( $R^2$  of 0.69) in the case of the logarithmic values of temperature.

Model	Equation	R <sup>2</sup>	ANOVA Test ( <i>p</i> -Value)
Linear	$N_{\rm f} = -2 \times 10^5 OC - 1266T + 55,083$	0.65	OC: 0.0013 T: 0.006
Nonlinear	$N_{\rm f} = -2 \times 10^5 OC - 69,974 \log T + 121,009$	0.69	OC: 0.0007 T: 0.0034

Table 9. Equations of N<sub>f</sub> parameter using LAS results.

## 4. Conclusions

This study was conducted to evaluate the low-temperature and fatigue performances of asphalt binders modified with DSO. Based on the analysis and results of the study, the following conclusions were drawn:

- 1. The results from the conventional tests showed that the penetration values increased, the softening point and the rotational viscosity both decreased with the addition of the bio-oil.
- 2. The increase in the bio-oil content resulted in a decrease in the continuous PG. A PG of 58 was obtained when 2.5% DSO was added to the control asphalt binder.
- 3. The fatigue parameter (G\*sinδ) of the bio-oil-modified binder decreased at higher oil contents and temperatures, and this is considered an improvement since the maximum value in the Superpave system is 5000 kPa at the intermediate temperature range.
- 4. The BBR results showed that the addition of bio-oil enhanced the low temperature performance, which leads to a better performance in cold climatic regions. The low PG of the bio-oil-modified asphalt binder at 5.5% DSO was -28 compared to -16 for the control asphalt binder.
- 5. The fatigue resistance of the modified asphalt binder has been enhanced; this was explained by the increase in the N<sub>f</sub> values obtained from the VECD theory.
- 6. The temperature was more significant in affecting the different assessed parameters such as the fatigue parameter, stiffness, PG, and stresses compared to the oil content. However, the oil content was more significant in the results of the LAS test as obtained from the ANOVA tests.
- 7. A DSO content of 1.5–2.5% was selected as the optimum value of bio-oil content to get the optimum fatigue and thermal cracking resistance while maintaining the high-temperature properties' stability without adversely affecting them.
- 8. The enhanced low-temperature and fatigue properties of the DSO-BMB obtained in this study are expected to enhance the performance of asphalt concrete in terms of low-temperature cracking and fatigue cracking as well.
- 9. Additional DSR LAS tests and VECD analyses are recommended to be performed for the DSO-BMBs at different strain levels with multiple replicates.

Few recommendations can be provided for future research based on the results and findings of this study such as the use of DSO as a rejuvenator agent for aged asphalt binder (RAP) instead of a replacement for asphalt binder.

Author Contributions: Conceptualization, G.G.A.-K., W.Z. and S.A.A.; methodology, G.G.A.-K., W.Z. and S.A.A.; formal analysis, G.G.A.-K., S.A.A., W.Z.; resources, W.Z., G.G.A.-K. and S.A.A.; data curation, S.A.A., G.G.A.-K., W.Z.; writing—original draft preparation, S.A.A., G.G.A.-K., W.Z.; writing—review and editing, G.G.A.-K., W.Z. and S.A.A.; supervision, G.G.A.-K., W.Z.; project administration, G.G.A.-K., W.Z. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not Applicable.

Informed Consent Statement: Not Applicable.

**Data Availability Statement:** The data related to the results and findings of this paper are available. At any time, the authors can provide them on request.

**Acknowledgments:** The authors of this paper would like to thank the Research Institute of Sciences and Engineering (RISE)'s Sustainable Construction Materials and Structural Systems (SCMSS) Research Group for their financial support.

Conflicts of Interest: The authors declare no conflict of interest.

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