Journal of Research in Science and Engineering (JRSE)
ISSN: 1656-1996 Volume-8, Issue-1, January 2026
DOI: 10.53469/jrse.2026.08(01).05

Rheological Characteristics and Micro-mechanism of Regenerated Asphalt from Different Viscosity Furfural Extract Oils

Wenxue Luan, Wenting Dai*

College of Construction Engineering, Jilin University, Changchun, China
**Correspondence Author, daiwt@jlu.edu.cn*

Abstract: ***Existing research indicates that furfural extracted oil (FEO) can regenerate aged asphalt. However, previous studies have lacked an investigation into the impact of the viscosity of FEO on the regeneration effect of asphalt. Therefore, this paper proposes the use of two viscosities of FEO to regenerate asphalt and compares the effects of different viscosities of FEO on the regeneration of aged asphalt. The basic physical properties of regenerated asphalt containing 10% and 15% of FEO with viscosities of 3.9 and 17-23 were compared using penetration, softening point, and ductility tests. Rheological characteristics were examined through dynamic shear rheometry and bending beam rheometry. Finally, the regeneration mechanism was analyzed using Fourier transform infrared spectroscopy. The findings revealed that FEO can effectively regenerate aged asphalt. As the viscosity of the extracted oil increased, the high-temperature deformation resistance of the regenerated asphalt improved, while its medium-temperature fatigue resistance and low-temperature cracking resistance decreased. Ultimately, it was determined that the ability of FEO to regenerate aged asphalt is due to its addition of small molecular components that are deficient in the aged asphalt. The lower polarity of the low-viscosity FEO allows for better reduction of the solubility of the aged asphalt compared to the higher viscosity oil, thus enhancing the solubility that decreases during the aging process due to increased polarity.***

Keywords: Asphalt, Furfural Extracted Oil, Regenerated Asphalt, FEO regenerated asphalt.

1. Introduction

Asphalt pavement has become the predominant surfacing material in transportation infrastructure owing to its superior ride quality [1]. Nevertheless, escalating traffic volumes impose heightened requirements on pavement structural capacity. Conventional asphalt surfaces are increasingly susceptible to distress under heavy traffic loading, manifesting as rutting, cracking, potholing, and permanent deformation [2-3]. Numerous pavements constructed in prior decades now necessitate intermediate and major rehabilitation. Current rehabilitation methodologies typically employ cold milling of deteriorated asphalt followed by overlay placement [4-5], generating substantial volumes of reclaimed asphalt pavement (RAP). The disposal of RAP presents significant environmental challenges, as landfilling contributes to ecological degradation and represents a substantial waste of finite resources. Asphalt recycling technology addresses this concern through reprocessing aged asphalt for reuse [6-9], offering an economically viable and environmentally sustainable approach aligned with circular economy principles.

FEO, a complex organic byproduct derived from lubricant manufacturing, comprises abundant polycyclic aromatic hydrocarbons (PAHs) alongside resin and asphaltene fractions [10-13]. Its chemical similarity to asphalt renders it suitable for asphalt modification [13-16]. The PAHs in FEO compensate for the maltene depletion in aged asphalt, improving the solvation capacity for aromatic constituents and facilitating the peptization of asphaltenic aggregates, thereby reducing average molecular weight [17]. Consequently, FEO application in asphalt recycling has garnered significant interest. For instance, Si L [18] reported that 4% FEO effectively regenerated lightly aged asphalt. While FEO utilization offers economic and sustainability benefits, extant research remains nascent, particularly regarding the influence of FEO viscosity on regeneration efficacy.

This investigation therefore evaluates the regeneration performance of FEO with distinct viscosity grades (3.9 vs. 17-23). The study comparatively analyzes fundamental physical parameters and rheological properties of regenerated asphalt, supplemented by Fourier transform infrared spectroscopy (FTIR) to elucidate microstructural evolution and rejuvenation mechanisms. The findings establish a theoretical framework for optimizing FEO-based asphalt regeneration protocols.

2. Experiment

2.1 Materials

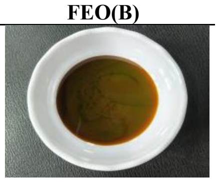
2.1.1 Asphalt

This investigation employs 70# paving-grade asphalt supplied by Hebei Xinhai Chemical Group Co., Ltd. The material's fundamental physical properties were characterized in accordance with the Chinese standard "Test Methods for Asphalt and Asphalt Mixtures in Highway Engineering" (JTG E20-2011). Penetration at 25 ℃ of base asphalt is 7.09 cm; ductility at 15 ℃ of base asphalt is > 100 cm; softening point of base asphalt is 47.5 cm.

2.1.2 Furfural extract oil (FEO)

FEO utilized in this research was sourced from Hengshui Diyi Petroleum Chemical Co., Ltd. The fundamental physicochemical properties of the FEO are comprehensively detailed in Table 2.

Table 2: Basic physical properties of FEO

Test	FEO(A)	FEO(B)
Appearance		
Kinematic viscosity at 100 ℃ /($mm^2 \cdot s^{-1}$)	3.9	17-23
Density at 20 ℃ /($g \cdot cm^{-3}$)	1.039	0.98-1.01
Flash point /(℃)	180	220
Pour point /(℃)	-15	-5

2.2 Aging of Asphalt

Aged asphalt was synthesized in this investigation through the combined application of the Rolling Thin Film Oven (RTFOT) and Pressure Aging Vessel (PAV) tests, adhering to the Chinese specification JTG E20-2011. Penetration at 25 ℃ of aged asphalt is 2.42 cm; ductility at 15 ℃ of aged asphalt is 3.7 cm; softening point of aged asphalt is 64.1 cm.

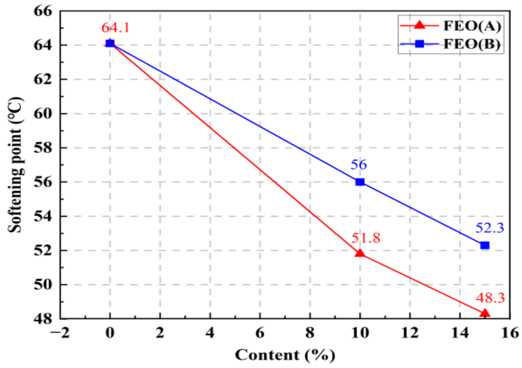
2.3 Preparation of Regenerated Asphalt

FEO was introduced into the aged asphalt matrix. The blending process was conducted at 150 °C, employing a high-speed asphalt shearing apparatus operating at 4000 revolutions per minute (rpm) for a duration of 15 minutes to ensure homogeneous dispersion of FEO within the asphalt binder.

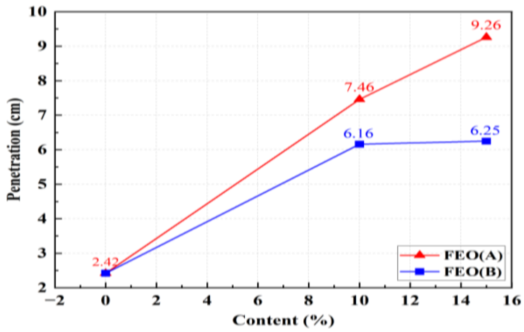
3. Results and Discussion

3.1 Basic Physical Properties

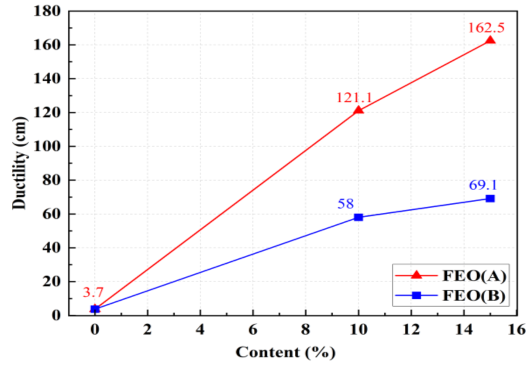
The softening point, penetration, and ductility tests are conducted to assess the basic physical properties of the asphalt. The test results are presented in Figure 1.



(a) Softenning point



(b) Penetration



(c) Ductility

Figure 1: The basic physical properties of FEO regenerated asphalt

According to Figure 1, the addition of FEO to aged asphalt results in regenerated asphalt whose softening point, penetration, and ductility at 5 °C all meet the standards of 70# matrix asphalt. Notably, the softening point and ductility of the regenerated asphalt using FEO are superior to those of the matrix asphalt before aging.

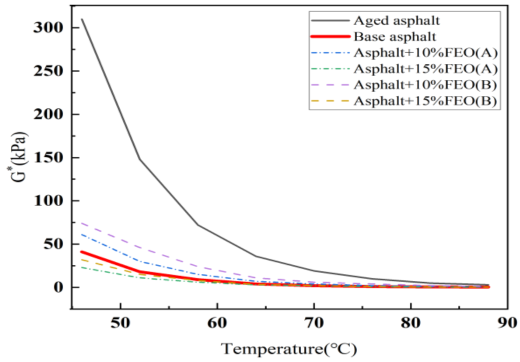
As the amount of FEO increases, the softening point of the regenerated asphalt gradually decreases, while the penetration and ductility at 5 °C increase. This indicates that with the increasing dosage of FEO, the regenerated asphalt becomes progressively softer, leading to a reduction in high-temperature deformation resistance, while the low-temperature cracking resistance improves.

Comparing the regenerated asphalt made with different viscosities of FEO, it is observed that the regenerated asphalt using FEO(A) has a lower softening point, higher penetration, and greater ductility at 5 °C than that made with FEO(B). This suggests that the regenerated asphalt using FEO(A) is softer, has weaker high-temperature deformation resistance, and exhibits stronger low-temperature cracking resistance compared to that made with FEO(B).

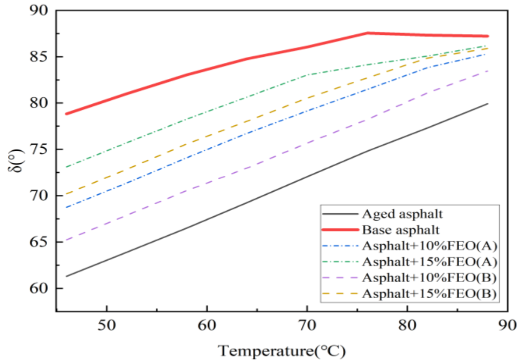
3.2 Rheological Characteristics

3.2.1 High-temperature deformation resistance

Dynamic shear rheometer tests are conducted on the regenerated asphalt using various furfural extracted oils to perform temperature scans, yielding results for the complex shear modulus (G*) and phase angle (δ). At the same temperature, a larger G* value and a smaller δ value indicate stronger high-temperature deformation resistance of the asphalt. The test results are presented in Figure 2.



(a) Complex shear modulus (G*)



(b) Phase angle (δ)

Figure 2: Complex shear modulus (G*) and Phase angle (δ) of FEO regenerated asphalt

From Figure (a), it can be observed that the G* of the aged asphalt is significantly greater than that of the matrix asphalt. This indicates that the aging effect on the asphalt is quite pronounced, providing a reliable basis for subsequent assessments of the regeneration effect. As the temperature increases, G* decreases while the δ increases. This behavior is attributed to the enhanced fluidity of the asphalt with rising temperature, leading to a deterioration in high-temperature deformation resistance. With the increasing dosage of FEO, the G* of the regenerated asphalt gradually decreases, and δ gradually increases. This suggests that the flowability of the regenerated asphalt improves as more FEO is added. When using 15% of FEO A for regeneration, the G* of the regenerated asphalt is greater than that of the matrix asphalt, indicating that its flowability is superior compared to the matrix asphalt. In Figure (b), it is evident that at the same temperature, the δ values of the regenerated asphalt with FEO are lower than those of the matrix asphalt. This is because the addition of FEO alters the ratio of viscoelastic components in the aged asphalt, resulting in an increase in the small molecular components of the regenerated asphalt compared to the aged asphalt. This change enhances the viscous components of the asphalt, although it does not fully restore to the level of the matrix asphalt. Nonetheless, this is beneficial for the high-temperature deformation resistance of the regenerated asphalt. Comparing the regenerated asphalt made with different viscosities of FEO, it is found that as the viscosity of the FEO increases, the G* of the regenerated asphalt increases while δ decreases. This indicates that the high-temperature performance of the regenerated asphalt improves with the increased viscosity of the FEO.

3.2.2 Medium-temperature fatigue resistance

The fatigue factor (G*sinδ) is determined using the linear amplitude sweep (LAS) mode of a dynamic shear rheometer, representing the product of the complex shear modulus (G*) and the sine of the phase angle (δ). This factor reflects the ability of asphalt to resist cracking under repeated stress or strain conditions. A larger G*sinδ indicates that the asphalt experiences a faster performance loss under load, leading to poorer medium-temperature fatigue performance. The variation of the fatigue factor with temperature is illustrated in Figure 3.

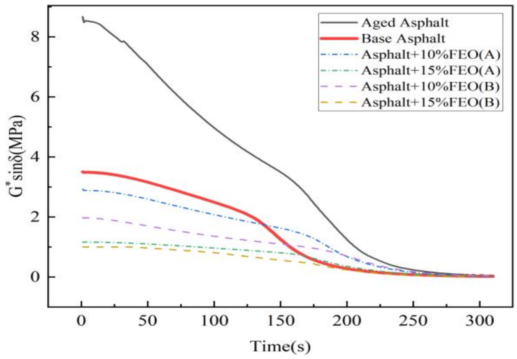


Figure 3: The fatigue factor(G*sinδ) of FEO regenerated asphalt

The G*sinδ values of aged asphalt, matrix asphalt, and regenerated asphalt gradually decrease over time. This indicates that the medium-temperature fatigue resistance of each type of asphalt is increasing. As the content of FEO increases, the G*sinδ of the regenerated asphalt decreases, suggesting that the medium-temperature fatigue resistance improves with a higher dosage of FEO. Furthermore, the G*sinδ values of the regenerated asphalt using FEO A are greater than those using FEO(B) at all dosages. This indicates that as the viscosity of the FEO increases, the medium-temperature fatigue resistance of the regenerated asphalt is enhanced.

3.2.3 Low-temperature crack resistance capability

The low-temperature creep stiffness (S) and low-temperature creep rate (m) of asphalt were measured using a bending beam rheometer at temperatures of -18 °C, -12 °C, and -6 °C. A larger S value indicates greater stiffness of the asphalt, which corresponds to poorer low-temperature cracking resistance. Conversely, a larger m value signifies enhanced stress relaxation ability of the asphalt at low temperatures, resulting in better low-temperature cracking resistance. The test results are presented in Figure 4.

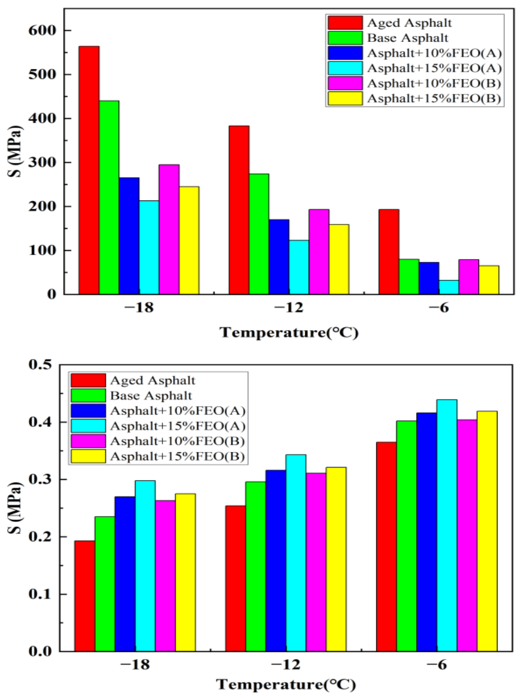
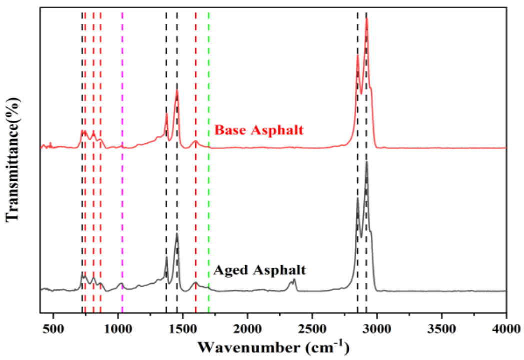


Figure 4: the low-temperature creep stiffness(S) and low-temperature creep rate(m) of asphalt

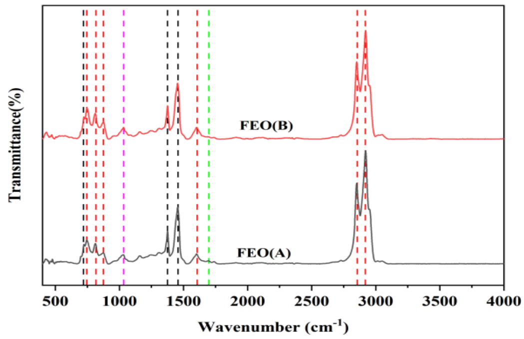
As shown in Figure 4, as the temperature decreases, The S of the regenerated asphalt with FEO gradually increases, while the m gradually decreases. This indicates that as the temperature drops, the stiffness of the asphalt increases, leading to poorer low-temperature cracking resistance. At different temperatures, the regenerated asphalt exhibits lower S and higher m values compared to aged asphalt, and it also shows lower S and higher m values compared to matrix asphalt. This demonstrates that the addition of FEO significantly improves the low-temperature cracking resistance of aged asphalt, making the regenerated asphalt superior to the matrix asphalt. With the increasing dosage of FEO, the S of the regenerated asphalt decreases while the m increases. This suggests that as more FEO is added, the asphalt becomes progressively softer, enhancing its low-temperature cracking resistance. Additionally, the regenerated asphalt made with FEO(A) has a smaller S and a larger m compared to that made with FEO(B), indicating that as the viscosity of the FEO increases, the low-temperature cracking resistance of the regenerated asphalt decreases.

3.3 FTIR Test Results

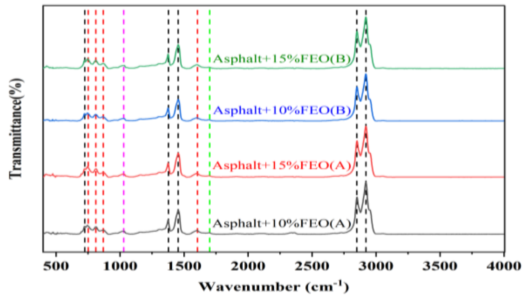
FTIR testing was conducted on FEO and various asphalt samples to assess their chemical characteristics. The test results are presented in Figure 5.



(a) Aged asphalt and base asphalt



(b) FEO



(c) FEO regenerated asphalt

Figure 5: FTIR test results

As shown in Figure (b), the FTIR spectra of the two types of FEO are identical, indicating that the substances contained in the oils of different viscosities are the same. Comparing the FTIR spectra of various regenerated asphalts from different FEOs in Figure (c), it can be observed that the absorption peaks appear at essentially the same positions, demonstrating that the functional groups in each regenerated asphalt are the same. When comparing the FTIR spectra of the regenerated asphalts with matrix asphalt, it is evident that both the FEO regenerated asphalt and the matrix asphalt exhibit absorption peaks at the same positions, indicating that the addition of FEO to the aged asphalt does not introduce any additional functional groups.

Table 4: Infrared spectroscopy absorption peak values

Absorption peak	Functional group	Aged Asphalt	Base Asphalt	FEO (A)	FEO (B)	Asphalt+ 10% FEO(A)	Asphalt+ 15% FEO(A)	Asphalt+ 10% FEO(B)	Asphalt+ 15% FEO(B)
3645	**O-H**	-	-	-	-	-	-	-	-
2924	**C-H**	0.2972	0.2981	0.2631	0.2524	0.2646	0.2457	0.1691	0.2711
2852	**C-H**	0.2143	0.2141	0.1872	0.1796	0.1907	0.1773	0.1297	0.1932
1750	**C=O**	-	-	-	-	-	-	-	-
1700	**C=O**	0.0090	0.0032	-	-	0.0103	0.0072	0.0058	0.0070
1600	**-**	0.0207	0.0170	0.0228	0.0271	0.0238	0.0226	0.0154	0.0204
1460	**$-CH_2-$**	0.1332	0.1340	0.1310	0.1299	0.1286	0.1208	0.0852	0.1255
1377	**$-CH_3$**	0.0793	0.0796	0.0752	0.0771	0.0736	0.0680	0.0485	0.0699
1030	**S=O**	0.0183	0.0061	0.0213	0.0271	0.0397	0.1090	0.0634	0.0665
865	**-**	0.0189	0.0207	0.0251	0.0398	0.0207	0.0194	0.0122	0.0182
810	**-**	0.0306	0.0341	0.0472	0.0596	0.0415	0.0372	0.0263	0.0388
750	**-**	0.0352	0.0393	0.0551	0.0718	0.0504	0.0454	0.0329	0.0479
722	**$(CH_2)_n$**	0.0394	0.0412	0.0450	0.0518	0.0438	0.0406	0.0309	0.0428

Previous research has indicated that asphalt aging results from a reduction in small molecular structures and an increase in large molecular structures within the asphalt, as well as a rise in the content of carbonyl and sulfoxide groups, leading to increased viscosity and subsequent aging of the asphalt. From Table 4, it can be observed that the aged asphalt exhibits significantly higher levels of carbonyl groups at 1700 cm^{-1} and sulfoxide groups at 1030 cm^{-1}. Additionally, at 722 cm^{-1}, the peak value for aged asphalt is noticeably lower than that of the matrix asphalt, indicating that the aging effect of the asphalt used in this experiment is quite pronounced.

When comparing the two types of FEOs, we find that both contain three methylene absorption peaks at 2924 cm^{-1}, 2852 cm^{-1}, and 1460 cm^{-1}, which are the highest among all peaks. Long-chain alkanes are also present at 722 cm^{-1}, suggesting that the FEOs contain a substantial amount of alkane compounds. Furthermore, there are three aromatic ring absorption peaks at 1600 cm^{-1}, 865 cm^{-1}, and 810 cm^{-1}, with an appearance of aromatic compounds at 750 cm^{-1}, indicating a significant presence of aromatic substances in the FEOs. Therefore, the composition of FEOs primarily consists of alkanes and aromatics, rich in small molecular components such as saturated and aromatic fractions.

Comparing the peak values of the methylene absorption peaks at 2924 cm^{-1}, 2852 cm^{-1}, and 1460 cm^{-1} between the two types of FEOs reveals that FEO(A) has larger peak values than FEO(B), indicating that FEO(A) contains more small molecular components. In contrast, when comparing the peak values of the aromatic ring absorption peaks at 1600 cm^{-1}, 865 cm^{-1}, and 810 cm^{-1}, FEO(B) shows larger peak values than FEO(A), as does the peak for aromatic compounds at 750 cm^{-1}. This suggests that FEO(B) contains a greater quantity of aromatic components. Additionally, the peak value for the sulfoxide group at 1030 cm^{-1} is also larger in FEO(B) compared to FEO(A). In summary, as the viscosity of FEOs increases, the content of aromatic components and heteroatoms rises, while the quantity of small molecular components diminishes.

When comparing matrix asphalt with FEO regenerated asphalt, it is evident that the addition of FEO to aged asphalt does not lead to a decrease in the peak values of the carbonyl group at 1700 cm^{-1} and the sulfoxide group at 1030 cm^{-1}. This confirms that the regeneration process facilitated by FEO does not remove the carbonyl and sulfoxide groups generated during the aging of asphalt. Moreover, when comparing the regenerated asphalt with aged and matrix asphalt, there are no significant changes in the peak values for methylene and methyl groups; however, the peak values for aromatic rings at 865 cm^{-1} and 810 cm^{-1}, along with the peak for aromatic compounds at 750 cm^{-1}, have increased considerably in the regenerated asphalt. This may be due to the high content of aromatics in the FEO, which enhances the aromatic component concentration in the aged asphalt. Additionally, since aromatic rings have a dissolving effect, they help dissolve the larger molecular components that have increased due to aging. As the content of FEO in aged asphalt increases, the peak values for the aromatic rings at 865 cm^{-1} and 810 cm^{-1}, along with the peak for aromatic compounds at 750 cm^{-1}, also increase. According to the physical and rheological characteristics described earlier, the viscosity of the regenerated asphalt decreases with the increasing content of FEO, leading to a softening of the regenerated asphalt. This demonstrates that the regeneration of aged asphalt by FEO is attributed to its high aromatic content, which boosts the aromatic component concentration in the aged asphalt and improves its solubility, which had previously decreased due to increased polarity, thereby increasing the relative amount of small molecular components in the asphalt.

Comparing the peak values of regenerated asphalt from the two FEOs reveals that FEO(A) exhibits larger aromatic ring peaks at 810 cm^{-1} and aromatic peaks at 750 cm^{-1} than FEO(B). However, the peak values of the aromatic ring at 810 cm^{-1} and the aromatic compound at 750 cm^{-1} for FEO(A) are significantly smaller than those for FEO(B). This may be due to the smaller aromatic rings in FEO(A) compared to those in FEO(B), resulting in stronger solubility.

4. Conclusion

This study utilizes FEOs with varying viscosities to rejuvenate aged asphalt. Through basic physical property testing and rheological characteristic analysis, it systematically evaluates the performance of FEO rejuvenated asphalt, comparing the regeneration effects of oils with different viscosities, and delves into the regeneration mechanism in conjunction with changes in chemical properties. This research addresses the impact of the viscosity of FEO on the rejuvenation effect of asphalt, which has been overlooked in previous studies. The main conclusions of the study are as follows:

1) The addition of FEO to aged asphalt significantly enhances the performance of the rejuvenated asphalt, allowing it to reach the level of matrix asphalt, with its performance even surpassing that of matrix asphalt.

2) As the amount of FEO increases, the high-temperature deformation resistance of the rejuvenated asphalt gradually decreases, its viscosity decreases, while its medium-temperature fatigue resistance and low-temperature cracking resistance significantly improve. Conversely, as the viscosity of the FEO increases, the high-temperature deformation resistance of the rejuvenated asphalt gradually increases, its viscosity rises, while its medium-temperature fatigue resistance and low-temperature cracking resistance decrease relatively.

3) FEOs of different viscosities contain the same functional groups; however, the higher viscosity oils contain more aromatic compounds and heteroatoms, while having fewer small molecular structures. The ability of FEO to rejuvenate aged asphalt is attributed to its addition of small molecular components that are lacking in aged asphalt. The lower viscosity FEO has relatively lower polarity, which better reduces the solubility of aged asphalt compared to the higher viscosity oils, thus enhancing the solubility that decreases due to increased polarity during the aging process.

This study has not yet clarified the application of FEO rejuvenated asphalt in actual engineering projects. Future work should involve establishing experimental sections to analyze the performance of FEO rejuvenated asphalt in real-world applications.

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