

## Research Article

# Preparation and Properties of Nano-ZnO Combined with Biomass Heavy Oil Composite-Modified Asphalt

He Tao , Hongming Liu , Xiangbing Xie , Tao Sun , Ruipeng Dong , and Xiaolu Lu 

Zhengzhou University of Aeronautics, School of Civil Engineering and Architecture, Zhengzhou 450000, China

Correspondence should be addressed to Xiangbing Xie; [xiexiangbing.good@163.com](mailto:xiexiangbing.good@163.com)

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Biomass heavy oil is employed to partially substitute the petroleum-based asphalt due to its renewability, environmentally friendly, and cost-effective advantages. However, its usage weakens the high-temperature performance of petroleum asphalt. Therefore, nano-ZnO was employed as a modifier to make composite-modified asphalt to improve the high-temperature performance in this paper. It was prepared in two steps: first, bio-asphalt was prepared by adding biomass heavy oil based on a certain content of matrix asphalt and then adding nano-ZnO with the particle size of 20 nm. The modification methods of bio-asphalt and composite-modified asphalt were analyzed by Fourier infrared (FTIR) and X-ray diffraction (XRD) experiments. Three major indexes test, dynamic shear rheological test, Brookfield rotational viscosity test (RV), and thermogravimetric analysis (TG-DSC) are used to analyze the high-temperature performance of bio-asphalt and composite-modified asphalt. The results show that biomass heavy oil and nano-ZnO are used to physically and chemically modify asphalt and the modification effect of bio-asphalt is the best when the biomass heavy oil content is about 9%. The high-temperature performance of composite heavy oil-modified asphalt is improved significantly. The performance indexes of Bio-6% and 1% nano-ZnO composite-modified asphalt are better than bio-asphalt. However, the high-temperature performance of Bio-12% and 3% nano-ZnO composite-modified asphalt is the best.

## 1. Introduction

With the rapid development of the road traffic industry in China, the petroleum asphalt used in the pavement industry is also increasing gradually. As a by-product of petroleum processing, petroleum asphalt is greatly affected by its non-renewability. Thus, scholars from all over the world are constantly looking for alternatives to petroleum asphalt to alleviate this problem. Bio-asphalt gradually comes into view with its characteristics of renewable, environmental protection, economy, and so on. At present, wood chips, straw, and animal dung are mainly used to prepare biomass heavy oil [1–6] by thermal pyrolysis or thermal liquefaction technology. Cao et al. [7] took wood sawdust as raw material, prepared biomass heavy oil under the conditions of ethanol-glycol as mixed solvent and concentrated sulfuric acid as catalyst, and studied the effects of ethanol to glycol ratio and liquid-solid ratio on heavy oil yield under different conditions. Gai et al. [8] conducted

hydrothermal liquefaction of straw and microalgae and concluded that increasing the proportion of microalgae could increase the yield of bio-oil. Bio-asphalt is derived from biomass materials, and the by-products of biomass waste processed in multiple ways are added into asphalt to achieve the purpose of modification or substitution. At present, there is no exact definition of bio-asphalt at home and abroad. In recent years, scholars at home and abroad have conducted extensive research on bio-asphalt binder and mixture [1, 9–17]. Zeng et al. [18] used the waste of castor oil refining to prepare bio-asphalt and used 0, 10, 20, 30, 40, and 50% of the mass fraction of aging asphalt as the mixing amount to prepare bio-recycled asphalt, respectively, which reduced the temperature sensitivity and significantly improved the low temperature performance. From the perspective of aging resistance, He et al. [19] proposed that the content of bio-oil should not be greater than 20% when biomass heavy oil is used as modifier. Liao et al. [20] took soybean and cottonseed as raw materials and added it to matrix asphalt and

SBS-modified asphalt at conventional high speed to prepare binder. Through relevant tests, it was found that the low temperature crack resistance and fatigue resistance of the blend were improved, but the high-temperature stability was decreased. Wang [21], such as the three kinds of asphalt and asphalt mixing by hand electric drill mixer, related experimental results showed that biological blending asphalt is stiffness and fatigue performance of asphalt, the rutting resistance and high-temperature stability, low temperature crack resistance, ductility, biological binder after blending water stability of mixture meet the requirements. Fini et al. [22] analyzed the characterization of bio-asphalt through relevant tests and chemically modified the matrix asphalt after fusion. The results showed that it was more conducive to the formation of stable bio-asphalt materials. Raouf and Williams [23] also used the above materials to completely replace matrix asphalt and mix low density polyethylene (LDPE) at the same time, improving effectively the low temperature and antiaging properties. Fan and Xue [17] selected the biomass heavy oil extracted from corn straw and verified through Fourier transform infrared (FTIR) test that the biomass heavy oil could improve the low temperature rheological properties of aging asphalt by changing the internal component structure of aging asphalt. The high-temperature performance of nano-ZnO-modified asphalt prepared by Zhu [24] was significantly improved. Li et al. [25] compared the effects of spherical nano-ZnO with average particle sizes of 80 nm, 350 nm, and 2  $\mu\text{m}$  on various properties of asphalt, and the experimental results showed that the smaller the particle size, the better the modification effect.

Above all, scholars have made some achievements in the study of biologically modified asphalt. Bio-asphalt shows good performance at low temperatures but poor performance at high temperature. At present, there are relatively few studies on improving the high-temperature performance of biologically modified asphalt. Due to the promising characteristics of nano-ZnO particles, they can be used to enhance the performance of bio-asphalt. Therefore, nano-ZnO is applied as a modifier to enhance the high-temperature performance of the bio-asphalt, which can be used widely in pavement application. In this study, the composite-modified asphalt was prepared by mixing heavy oil content of 3, 6, 9, 12, and 15% (the proportion of biomass heavy oil to base asphalt), 1, 3, and 5% nano-ZnO with the bio-asphalt. The modification principle was studied by FTIR and X-ray diffraction test. The basic performance and high-temperature performance were evaluated by three major indexes, rotational viscosity (RV) test, dynamic shear rheological test, and thermogravimetric analysis. The research route is shown in Figure 1. This study can provide a theoretical reference for promoting and applying bio-asphalt in modified asphalt.

## 2. Experimental Design

### 2.1. The Raw and Processed Materials

**2.1.1. The Matrix Asphalt.** The test selects 70<sup>#</sup> asphalt as the research object (after this referred to as matrix asphalt), and the technical indexes shown in Table 1. The specification for

asphalt and asphalt mixture in highway engineering (JTG E20-2011) is used in this part.

**2.1.2. Biomass Heavy Oil.** In the experiment, waste biomass, including wheat and corn straw, is used as a raw material to undergo high-temperature cracking in a high-temperature cracking furnace. And then, the waste is obtained by distillation, oxidation, and other treatment processes. It is a solid, black and brown in color, and lighter than the traditional petroleum asphalt at room temperature. Its density is 0.899  $\text{g}\cdot\text{m}^{-3}$  and lightning is 240°C.

**2.1.3. Nanomaterials.** The nanomaterial is selected as nano-ZnO in the experiment. The particle size is 20 nm, the specific surface area is 48.16  $\text{m}^2\cdot\text{g}^{-1}$ , and the density is 0.56  $\text{g}\cdot\text{cm}^{-3}$ .

**2.2. Sample Preparation.** The preparation method of composite-modified asphalt is shown in Figure 2. First, the No. 70 matrix asphalt is put to the melting state, and then the biomass heavy oil is mixed with the matrix asphalt at the ratio of 3, 6, 9, 12, and 15% (Bio-3, 6, 9, 12, and 15) of the total matrix asphalt at 130°C and 2000 r/min [18] rotating speed through the high-speed shear instrument. The bio-asphalt is prepared by the high-speed shear for 20 min. When the temperature rose to 160°C, nano-ZnO added the mixture with 1, 3, and 5% of the total mass of bio-asphalt, respectively. High-speed shear is carried out for 50 min at a speed of 3500 r/min. Matrix asphalt (Bio-0%, 0% nano-ZnO) is treated at the same temperature and rate.

### 2.3. Test Method

**2.3.1. Fourier Infrared Analysis (FTIR).** In order to analyze the changes of chemical components of asphalt during the regeneration process of biomass heavy oil, bio-asphalt, and composite-modified asphalt, infrared spectroscopy (FTIR) tests were carried out on the above three samples. The spectral scanning range is 400–2000  $\text{cm}^{-1}$ , and the number of scanning is 300.

**2.3.2. X-Ray Diffraction Analysis (XRD).** In order to analyze the effects of biomass heavy oil and Nano-ZnO on asphalt, X-ray diffraction (XRD) tests were carried out on matrix asphalt, bio-asphalt, and composite-modified asphalt. The scanning rate is 5°/min, and the scanning range is 5°–80°.

**2.3.3. Physical Properties.** The three indexes of asphalt including the ductility, penetration, and softening point which can represent the plasticity, temperature sensitivity, heat resistance, and other properties of asphalt are the basic physical properties of asphalt. The test is conducted to determine the ductility, penetration degree, and softening point of the composite-modified asphalt according to the test rules for Highway Engineering Asphalt and Asphalt Mixture (JTG E20-2011).

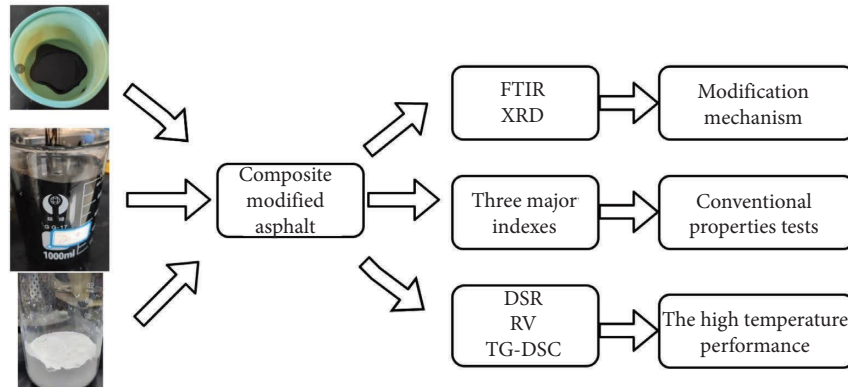


FIGURE 1: The research route.

TABLE 1: The technical index of asphalt.

Technical indicators	Technical requirements	Test results	Specifications
Penetration (25°C, 5 s, 100 g)/(0.1 mm)	60~80	62	
Penetration index PI	-1.5~+1.0	-1.27	T 0604-2011
Softening point (°C)	≥46	47.1	T 0606-2011
Ductility (10°C, 5 cm/min)/cm	≥15	36	T 0605-2011
Ductility (15°C, 5 cm/min)/cm	≥100	>100	T 0605-2011

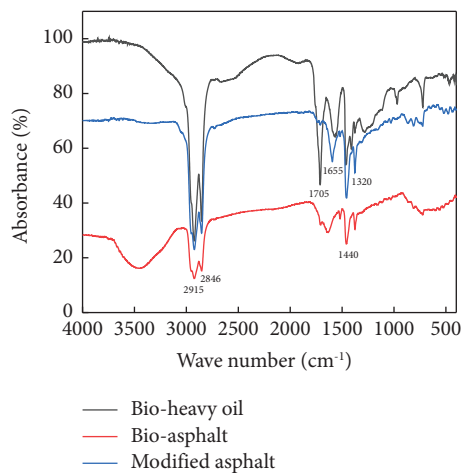


FIGURE 2: The picture of the infrared spectrum.

**2.3.4. Rheology Test.** In order to analyze the dynamic viscoelasticity of asphalt, the instrument model MCR302 & CR102 dynamic shear rheological instrument is selected for the test. The shear rheological test was carried out in the temperature control mode, where the loading frequency was 1.59 Hz, the temperature was controlled between 40 and 80°C and the type of the parallel plate is PP25, the spindle is 1 mm.

**2.3.5. Dynamic Viscosity Analysis (RV).** Viscosity is one of the basic physical properties of asphalt, which reflects the ability of asphalt to block its internal relative flow at a certain temperature and also reflects its temperature sensitivity. According to the test specification for Asphalt and Asphalt Mixture in Highway Engineering (JTG E20-2011), the

viscosity of each asphalt sample at 135°C was measured by the Brinell viscosity test, and the change rule was analyzed by comparing the viscosity changes of bio-asphalt and composite-modified asphalt.

**2.3.6. Thermogravimetric Analysis (TG-DSC).** STA409PC/4/H thermogravimetric analysis is used for testing, with a heating rate of 10°C/min and a test range of 0–800°C. The analysis draws a mass and from temperature (or time) the relationship between the analysis of mass and temperature.

### 3. Results and Discussion

**3.1. Fourier Infrared Analysis (FTIR).** As shown in Figure 2, the components of petroleum asphalt are mainly aromatic compounds, among which benzene ring structure is more. On the whole, there are two strong absorption peaks in the region of 3000–2750 cm<sup>-1</sup>, with sharp peaks, which are methylene -CH<sub>2</sub>- antisymmetric stretching vibration and symmetric stretching vibration, respectively. The stretching vibration of unsaturated bond C=C occurs at about 1660–1630 cm<sup>-1</sup>. A weak absorption peak appears at 1600 cm<sup>-1</sup>, which is reflected by the vibration of the benzene ring framework and C=O bond. There is a weak absorption peak near 1460 cm<sup>-1</sup>, and the peak shape is sharp, which is the result of C-H in-plane stretching vibration (variable angle vibration) in methyl -CH<sub>3</sub> and C-H in-plane stretching vibration (bending vibration) in -CH<sub>2</sub>-. The symmetric angular vibration of CH<sub>3</sub> occurs near 1380 cm<sup>-1</sup>. In the range of 900–650 cm<sup>-1</sup>, the original =C-H out-of-plane vibration absorption peak appears. It should be noted that Li et al. [26] concluded that the modification of asphalt by

biomass heavy oil is a physical modification, which is different from this research. Because the various biomass sources and different biological heavy oil production method leads to different functional groups. In this study, the functional groups of biomass heavy oil obviously changed when it was mixed into the asphalt. It shows that this kind of biological crude oil chemical modification is carried out on the pitch. The infrared spectrum of the composite-modified asphalt shows that nano-ZnO is mainly used to modify the bio-asphalt physically. So, physical modification and chemical modification coexist in this study.

**3.2. X-Ray Diffraction Analysis (XRD).** Compared to 70# asphalt matrix of the diffraction image (Figure 3), with the biomass heavy oil added, it peaks at  $2\theta = 9.8^\circ$  and  $2\theta = 29.1^\circ$ , this is because with the addition of biomass heavy oil, asphalt and biomass heavy oil form crystal cells after fusion. Biomass heavy oil has a certain viscosity and contains trace non-melting impurities. With the mechanical stirring of the high-speed shear machine, the heavy oil and asphalt are fully and uniformly fused biological heavy oil have a certain viscosity and contains trace not melt impurities, accompanied by high-speed shear apparatus mechanical agitation. The mixture of heavy oil and asphalt is fully uniform. When nano-ZnO was added, according to the standard diffraction pattern PDF15-0045, it was evenly distributed in the bio-asphalts, and with its addition, the composite-modified asphalt formed a stable internal structure, indicating that nano-ZnO modified the bio-asphalts by physical modification.

### 3.3. Physical Properties

**3.3.1. Ductility.** As shown in Figure 4, the ductility of biologically modified asphalt increases first and then decreases with the increase of biomass heavy oil content, and the maximum value appears at about 12%. The overall results show that bio-asphalt has an obvious improvement effect on low-temperature performance, due to the soft texture of biomass-heavy oil itself. It has a good ductility at low temperatures when mixed with matrix asphalt.

With the addition of nano-ZnO, the ductility decreases with the increase of biomass heavy oil and nano-ZnO. The effect of different nano-ZnO dosage (1%, 3%, 5%) on the ductility of bio-asphalt is consistent, showing a downward trend. Nano-ZnO inhibits the low temperature performance of biomodified asphalt to a certain extent, but the low temperature performance of the composite-modified asphalt is improved compared with matrix asphalt to a certain extent.

**3.3.2. Penetration.** As shown in Figure 5, with the increase of biomass heavy oil, the penetration degree of bio-asphalt increases significantly. The penetration degree of the three kinds of nano-ZnO composite bio-asphalts is obviously lower than that of the biomodified asphalt. The penetration

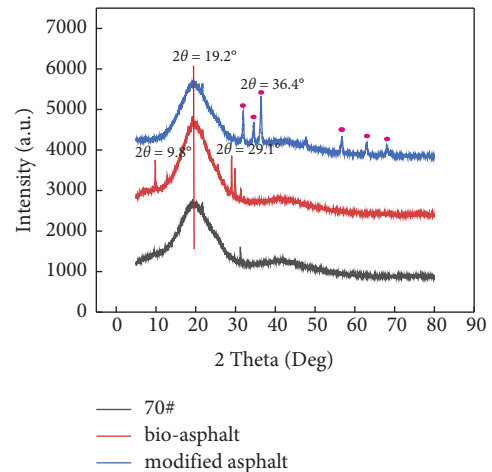


FIGURE 3: XRD patterns of three kinds of asphalt.

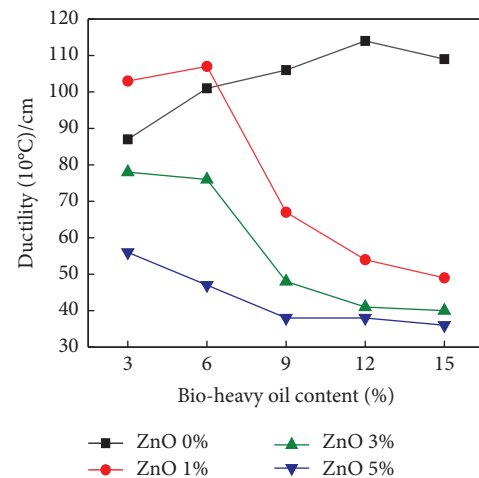


FIGURE 4: Ductility under different bio-asphalt contents.

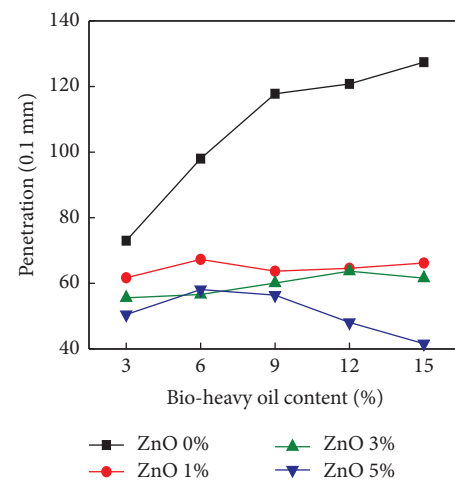


FIGURE 5: Penetration under different bio-asphalt contents.

levels of the modified asphalt are relatively good after adding 1% and 3% nano-ZnO. 5% dosage of nano-ZnO composite-modified asphalt, the level of the low level of penetration,

this is because with the increase of nanometer material, biological heavy oil, matrix asphalt, and nanometer material blending through physical and chemical reaction modification effects.

**3.3.3. Softening Point.** As shown in Figure 6, although bio-asphalt's high-temperature performance improves slightly with the increase of the mixing amount, the overall bio-asphalt has poor performance at high temperature, which is consistent with previous studies [9, 11]. After adding nano-ZnO, the three curves generally showed a trend of first increase and then decreased and improved the softening point to different degrees compared with only bio-asphalt. That is, the high-temperature performance was improved to some extent. It is worth pointing out that, with the addition of nano-ZnO with different contents (1%, 3%, and 5%), the high-temperature performance of biologically modified asphalt is significantly improved. As shown in Table 2, nano-ZnO's addition can increase the softening point of bio-asphalt by more than 20% at the highest. Even in this experiment, when the softening point of 9% bio-asphalt appears as the maximum value, it can also be increased to 6.7%. As shown in Figure 6 and Table 2, the longitudinal comparison shows that the softening point increment and increment percentage shows a trend of increasing first and then decreasing. And the softening point is the largest when the content of nano-ZnO is 3%.

In general, according to the three major indexes of asphalt, bio-asphalt has good performance at low temperature, but bio-heavy oil weakens its performance at a high temperature. When the content of nano-ZnO is 3%, composite-modified asphalt performs best and the low temperature performance can be guaranteed at the same time. However, from the test results of the ductility (10°C), a higher level of nano-ZnO content will weaken the low-temperature modification effect of asphalt by biomass heavy oil. The result of this part is consistent with references [12, 27]. Biomass heavy oil can improve the low temperature performance of asphalt, nano-ZnO can improve the high-temperature performance of asphalt.

**3.4. Dynamic Shear Rheology Test (DSR).** The complex shear modulus and rutting factor of composite-modified asphalt and matrix asphalt are shown in Figures 7 and 8.

**3.4.1. Complex Shear Modulus.** According to Figure 7, the fluidity of asphalt increases with the increase of temperature, and the values and slopes of the complex shear modulus of the bio-asphalt and composite-modified asphalt all of asphalt show a decreasing trend. This shows that 6% and 12% of bio-asphalts are more likely to come into being more considerable deformation at the same temperature. The level of bio-asphalt and complex shear modulus of the composite-modified asphalt is greater than the matrix asphalt. 9% level of bio-asphalt performance is better than any other dosage of biomass heavy oil. It is due to the physical and chemical changes in the asphalt matrix under the action of bio-asphalt and nano-ZnO.

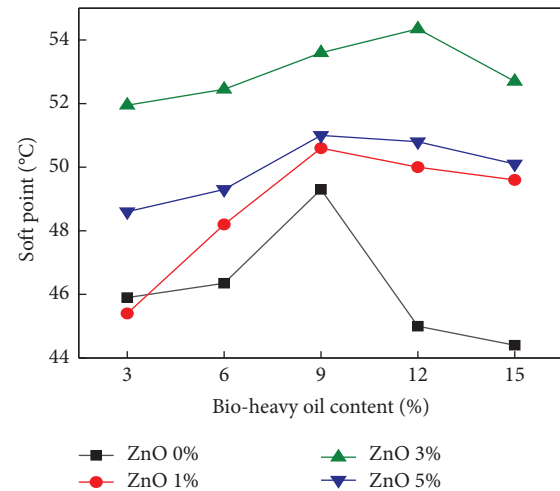


FIGURE 6: The softening point of bio-asphalt with different contents.

TABLE 2: Several significant-level softening point increment percentages (%).

Bio-asphalt nano-ZnO	6	9	12	15
1	3.9	2.6	11.1	11.7
3	10.9	6.7	20.9	18.7
5	4.7	3.4	13.3	12.8

Nano-ZnO forms a relatively stable spatial network structure [28] in the matrix asphalt during the synergistic modification of bio-asphalt, which made the composite-modified asphalt maintain a more stable state than that of bio-asphalt under high-temperature conditions. Thus, this improves the high-temperature performance of bio-asphalt.

**3.4.2. Rutting Factor.** Rutting factor ( $G^*/\sin \delta$ ) is used to measure the performance of asphalt rutting resistance. As shown in Figure 8(a), the rutting factor performs best when the bio-heavy oil content is 9%. This conclusion is consistent with that in reference [9, 17], which shows that biomass heavy oil's optimal dosage is about 10%. Figures 8(b) and 8(c) indicate that the rutting factor of bio-asphalts is improved by nano-ZnO. Bio-6%, 1% nano-ZnO and Bio-12%, and 3% nano-ZnO composite-modified asphalt have the best rutting resistance at high temperatures. This result is basically consistent with the changing trend and dosage of rutting factor in paper [27].

**3.5. Dynamic Viscosity Analysis.** The dynamic viscosity  $70^\#$ ,  $135^\circ\text{C}$  of matrix asphalt is 502 MPa-s. The dynamic viscosity of bio-asphalt is less than that of matrix asphalt expect the 9% bio-heavy oil content as shown in Figure 9. This is because the addition of biomass heavy oil reduces the consistency of asphalt and leads to a decrease in viscosity [17, 26]. The addition of nano-ZnO significantly increases the dynamic viscosity of asphalt at the same level of biomass

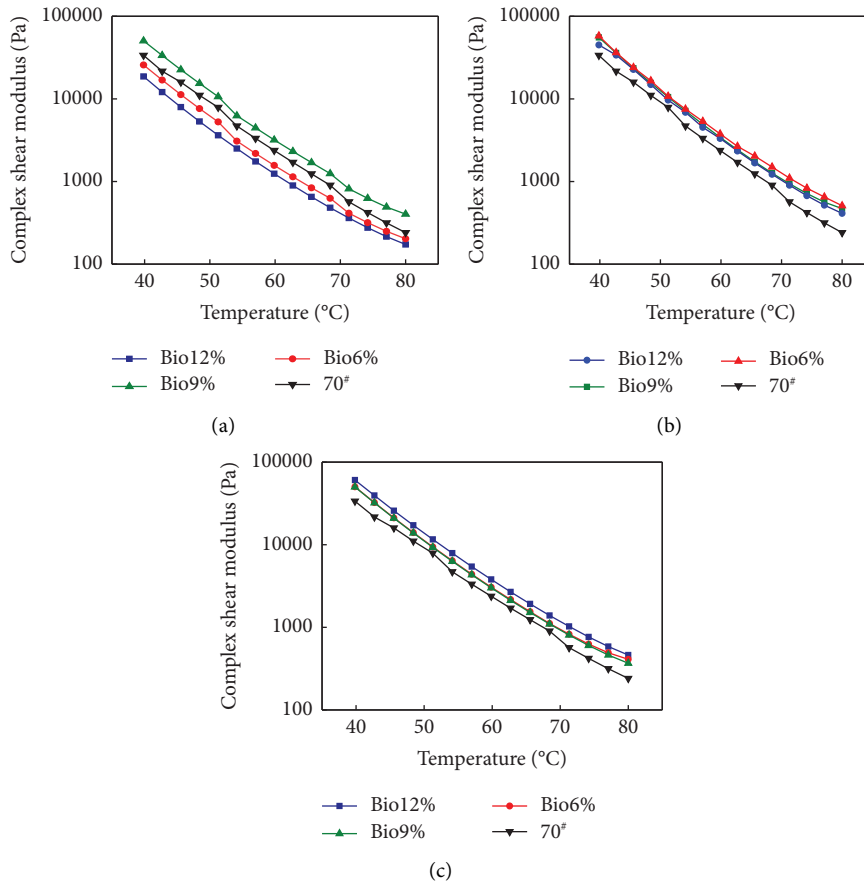


FIGURE 7: The relationship between complex shear modulus and temperature: (a) 0% nano-ZnO, (b) 1% nano-ZnO, and (c) 3% nano-ZnO.

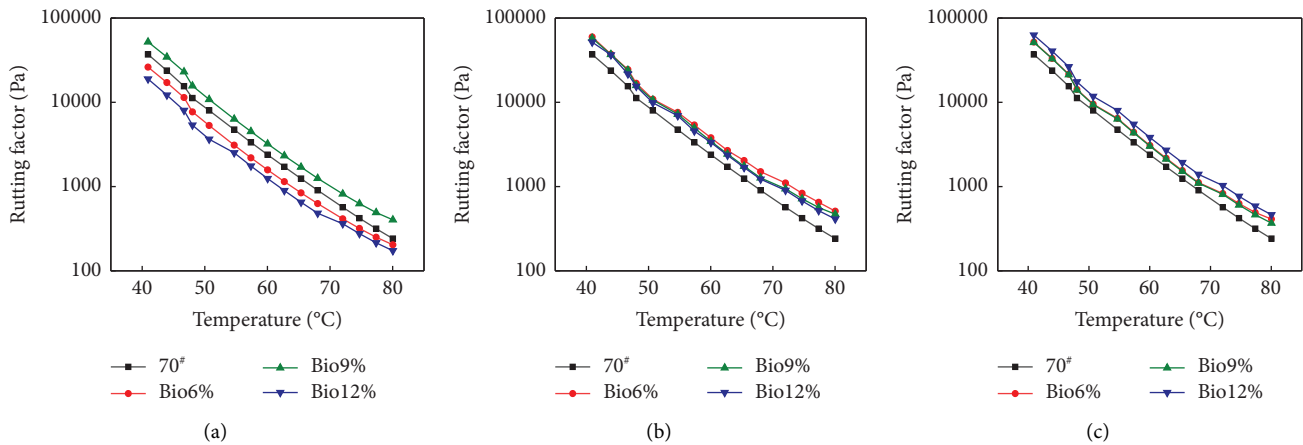


FIGURE 8: The relationship between rutting factor and temperature: (a) 0% nano-ZnO, (b) 1% nano-ZnO, and (c) 3% nano-ZnO.

heavy oil content because the spatial structure of nano-ZnO evenly distributed in the matrix asphalt at high-speed shear increases the dynamic viscosity of asphalt [28]. For nano-ZnO doped with 1%, the kinetic viscosity of pitch increased at 6% and 9%, but decreased at 12%. In contrast, the kinetic viscosity of nano-ZnO at 3% level gradually increased and the change rate also increased, indicating that the amount of nano-ZnO is insufficient to support the internal stable structure with the increase of heavy oil content. Therefore,

the experimental results show that the dynamic viscosity of 9% bio-asphalt without nano-ZnO, Bio-12, and 3% nano-ZnO composite-modified asphalt is the best. The results of this part are roughly consistent with the dosage in [26, 27].

3.6. *Thermogravimetric Analysis (TG-DSG)*. Figure 10(a) shows the TG-DTC curve of 70# asphalt. At 357°C, the weight of asphalt begins to decrease significantly. The addition of biomass heavy oil makes asphalt lose focus earlier,



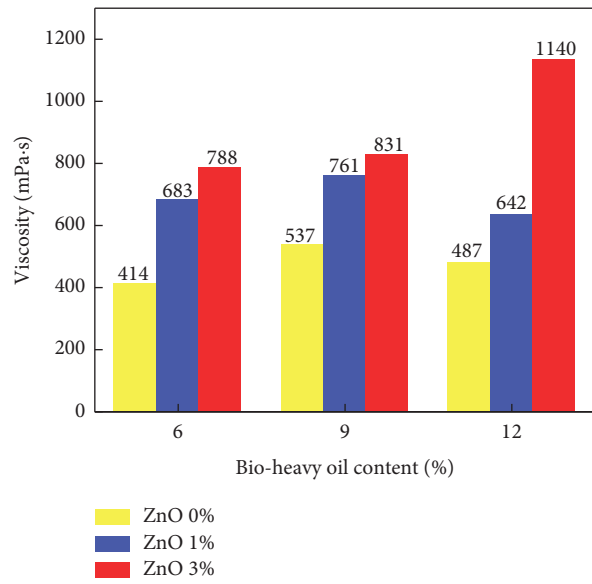


FIGURE 9: Dynamic viscosity of three kinds of asphalt.

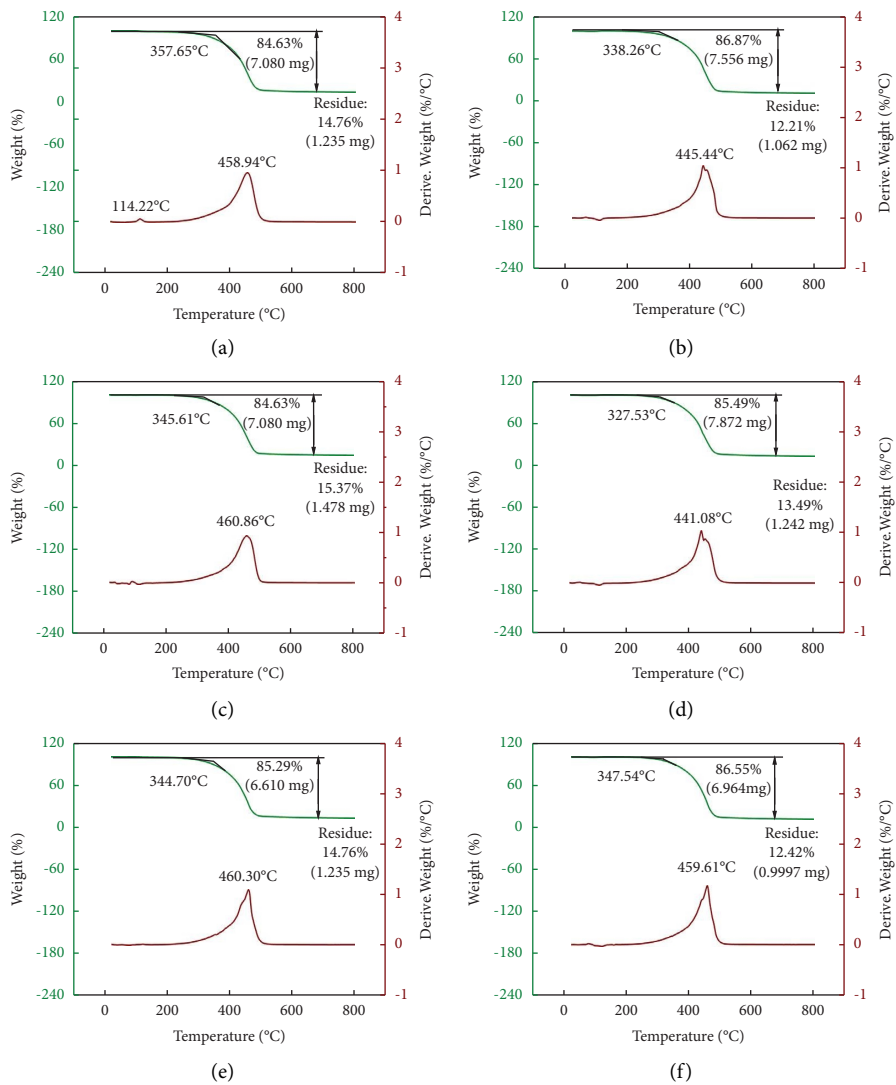


FIGURE 10: Continued.

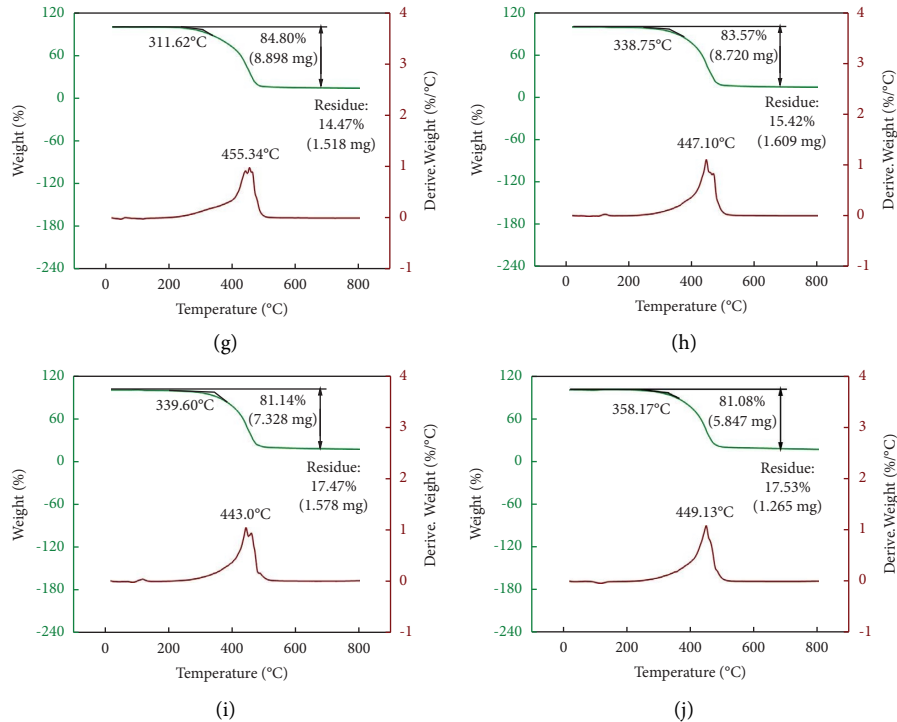


FIGURE 10: Thermogravimetric analysis of matrix asphalt, bio-asphalt, and composite-modified asphalt: (a) matrix asphalt, (b) Bio-6%, (c) Bio-9%, (d) Bio-12%, (e) Bio-6%, 1% nano-ZnO, (f) Bio-9%, 1% nano-ZnO, (g) Bio-12%, 1% nano-ZnO, (h) Bio-6%, 3% nano-ZnO, (i) Bio-9%, 3% nano-ZnO, and (j) Bio-12%, 3% nano-ZnO.

but it rebounds at 9%, and the extreme weight loss point increases by 1.92°C. It indicates that the thermal stability is improved slightly under the bio-heavy oil dosage.

Comparison of Figures 10(b)–10(j) nano-ZnO at 1% level shows that the heat loss point and extreme heat loss point of Bio-6% are increased. The heat loss point of Bio-9% increases, but the extreme heat loss point is advanced, while the extreme heat loss point of Bio-12% is the opposite. In contrast, the thermal stability of Bio-12% and 3% nano-ZnO improves. The heat loss point reaches 358.17°C, and the extreme heat loss point is 449.13°C, which is 30.64°C higher than in 10(d). The extreme heat loss point increased by 8.05°C.

Wan et al. [29] used this test method to explore the flame retardant performance of the flame retardant asphalt heat loss point and heat loss pole. This part also judges the high-temperature stability of bio-asphalt and composite-modified asphalt by them. In general, nano-ZnO can improve the performance of bio-asphalt to a certain extent, and the thermal stability of Bio-6%, 1% nano-ZnO and Bio-12, and 3% nano-ZnO composite-modified asphalt is better.

#### 4. Conclusion

- (1) FTIR and XRD experiments show that the biomass heavy oil and nano-ZnO were physically and chemically modified. The former made the asphalt softened by the chemical modification of some functional groups, and the latter formed a stable spatial network structure in the modified asphalt.

- (2) When the biomass heavy oil content is about 9%, the performance of bio-asphalt is better than any other dosage of biomass heavy oil in this experiment, mainly manifested as ductility (10°C), softening point, high-temperature rut factor, and viscosity is better than base asphalt. But there are certain defects in high-temperature performance.
- (3) After nano-ZnO combined with biomass heavy oil composite modification, the ductility (10°C) of the three composite-modified asphalt is decreased to different degrees, but all of them are better than the matrix asphalt. The composite-modified asphalt with Bio-12% and 3% nano-ZnO content has the most significant improvement in high-temperature performance under the premise that the elongation (10°C) is better than the matrix asphalt.
- (4) It suggests to prepare bio-asphalt with 9% bio-heavy oil content and to prepare composite-modified asphalt with Bio-12% and 3% nano-ZnO contents.

#### Data Availability

The data used to support the findings of this study are included within the article.

#### Conflicts of Interest

The authors declare that they have no conflicts of interest.



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