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COMPREHENSIVE PERFORMANCES OF HYBRID-MODIFIED ASPHALT MIXTURES WITH NANO-ZnO AND STYRENE-BUTADIENE-STYRENE (SBS) MODIFIERS

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Abstract. An effort was made to improve the separation issue of styrene-butadiene-styrene modifiers in styrene-butadiene-styrene-modified asphalt binders and to further enhance their performance by adding nano-ZnO to the styrene-butadiene-styrene-modified asphalt binder to prepare compound

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modified binder. First, the optimum nano-ZnO dosage was determined based on conventional tests, i.e., penetration, ring and ball softening point, and ductility at a fixed styrene-butadiene-styrene dosage; then, dynamic shear rheometer, bending beam rheometer, and fluorescence microscopy tests were conducted to evaluate the properties of the nano-ZnO/styrene-butadiene-styrene hybrid-modified asphalt binders. Finally, the rutting test, trabecular bending test, submerged Marshall test, and the freeze-thaw splitting test was conducted to evaluate the properties of the asphalt mixtures produced with the hybrid-modified binders. The main results showed that: the optimum nano-ZnO dosage, which was 4% by weight to the asphalt binder, and an improvement of the separation issue of the styrene-butadiene-styrene, and the flexural tensile strength and the maximum bending tensile strain of the hybrid-modified asphalt mixtures increased by 13.4% and 16.4%, respectively. In addition, the residual stability and the tensile strength ratio also increased by 4.3% and 4.8%, respectively, compared to the styrene-butadiene-styrene-modified asphalt mixtures. In conclusion, nano-ZnO improves the low-temperature and water stability performances of the styrene-butadiene-styrene-modified asphalt mixture.

Keywords: hybrid-modified asphalt, nano-ZnO, performance properties, styrene–butadiene–styrene (SBS), separation, surface treatment.

Introduction

In recent years, the styrene–butadiene–styrene (SBS) modified asphalt binder has been widely used in pavement construction due to its excellent performance (Elseifi et al., 2003). However, the SBS-modified asphalt sometimes has poor thermal storage stability, and segregation easily occurs, i.e., SBS modifier separation from the base asphalt binder (Xiao et al., 2005). The segregation is mainly caused by the physical and chemical properties of the SBS and asphalt being quite different from each other. The basic chemical reaction between the SBS modifier and the base asphalt binder is unobvious. The physical coexistence and integration between the SBS and base binder phases mainly improve the performance of modified asphalt binders. The system of the two phases with poor compatibility is usually unstable in the properties (Xiong et al., 2005). Improving the compatibility helps increase the properties of the SBS-modified asphalt binders.

Nanomaterials exhibit a high surface at the nanometer level (Albrka, 2018; Cheraghian et al., 2021; Enieb & Diab, 2017; Sun et al., 2016; Yao & You, 2017). Studies have shown that using nanomaterials as modifiers in asphalt binders improves many modified asphalt binder properties, including their temperature stability and antistripping property (Chen et al., 2017; De Melo & Trichês, 2016; Long et al., 2020; Sabaraya, 2018; Ye et al., 2020; Zhan et al., 2020). Nano-ZnO has attracted more research attention due to its low price and effectiveness in improving some properties. Xiao et al. (2010) and Zhang et al. (2016) studied the microstructure, conventional properties, and modification mechanism of nano-ZnO/SBS hybrid-modified asphalt binders. Xiao et al. (2010) and Zhang et al. (2016) concluded that nano-ZnO improves the SBS modifier uniformity in base asphalt binders by reducing its separation. Arabani et al. (2013) concluded that nano-ZnO is beneficial for improving the sensitivity of asphalt binder to temperature. At the same time, it improves the rutting resistance of its modified asphalt mixtures. Zhang (2016) evaluated the performance of asphalt mixtures by adding 5% nano-ZnO into the SBS-modified asphalt binder. Su et al. (2020) investigated the influence of nano-ZnO/SBS on the physical properties and structure of asphalt through the molecular dynamics method. In addition, Zhang et al. (2018) studied asphalt binders and mixtures modified by three different spherical-sized ZnO nano-particles, i.e., 2 μm, 80 nm, and 350 nm. The previous studies found that the evaluation of hybrid-modified asphalt binders with nano-ZnO and SBS is mainly separated from that of asphalt mixtures. Moreover, the effect of nano-ZnO on improving the dispersion of the SBS modifier in base asphalt is rarely studied.

This study aims to investigate the effect of nano-ZnO on improving the SBS modifier dispersion in base asphalt and systematically study the performance of nano-ZnO/SBS hybrid-modified asphalt. To this end, the optimum nano-ZnO dosage was determined based on the penetration, ring and ball softening point, and ductility tests. The rheological properties of the hybrid-modified asphalt binder with the selected optimum dosage are studied. Furthermore, the uniformity of the modifier in the asphalt is analysed through a fluorescence microscopy test. Finally, the rutting, bending beam, submerged Marshall, and freeze-thaw splitting tests are conducted to evaluate the improvement effects of asphalt mixtures with hybrid-modified asphalt binders at optimum nano-ZnO dosage.

1. Materials and experimental design

1.1. Materials

The base asphalt binder selected for the experiment was SK with a Pen grade of Pen #70 obtained from South Korea. Table 1 presents the technical properties of the base asphalt binder. The SBS modifier was a linear-type modifier (YH-791) from Yueyang Petrochemical Company. The blocking ratio S/B was 30/70. Nano-ZnO was provided by Nanjing

Hongde Nano Material Co., Ltd. Table 2 lists the technical properties of nano-ZnO.

Coarse basalt aggregates from Qujing, Yunnan and fine limestone aggregates from Kunming, Yunnan, were used. Limestone was used as the mineral filler. All indices met China-specific requirements (Table 3). A dense-graded asphalt mixture of AC-13C was used. Figure 1 depicts the gradation.

Table 1. Properties of base asphalt binder

			Rolling Thin-Film Oven Test (RTFOT)		
Penetration 25 °C, 100 g, 5 s 0.1 mm	Softening point, °C	Ductility*, cm	Quality change, %	Residual penetration ratio, %	Residual ductility*, cm
73.5	47.2	45.6	0.58	68.5	8.9

Note: *10 °C.

Table 2. Nano-ZnO properties

Testing item	Molecular weight, Da	Purity, %	Specific surface area, $m^2 \cdot g^{-1}$	Average particle size, nm	Density, $g \cdot cm^{-3}$	Volatile matter at 105 °C, %
Detection value	81.39	no less than 99.0	60	30	5.6	0.18

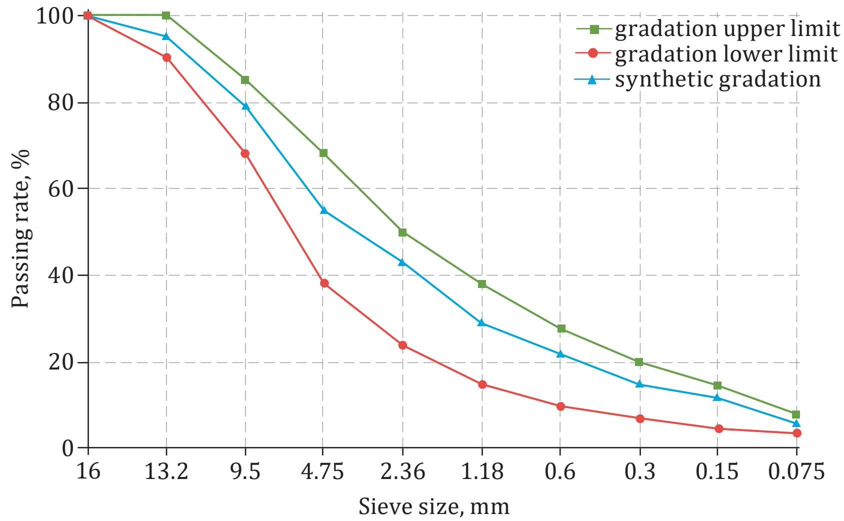


Figure 1. AC-13 gradation

Table 3. Aggregate properties

Testing item	Coarse aggregates								Fine aggregates
	Crushing value, %	Los Angeles abrasion loss, %	Apparent relative density		Water absorption rate, %		Needle flake content, %	Adhesion level, grade	Sand equivalent, %
			10–16 mm	5–10 mm	10–16 mm	5–10 mm			
Detection value	16.10	18.40	2.917	2.910	0.580	0.750	5.40	4	68

1.2. Surface treatment of nano-ZnO

Nanomaterials have a large specific surface area and a strong surface activity; thus, the agglomeration phenomenon is most likely to occur when the nano-particles are directly mixed with asphalt binder. This phenomenon makes it difficult to uniformly and thoroughly mix nano-ZnO within the base asphalt binder. The surface pretreatment of nano-ZnO is necessary to address the problem. The steps for the surface pretreatment are as follows:

1. nano-ZnO is placed in a 180 °C oven for 12 h to achieve complete drying;
2. an evenly alcohol aqueous solution with anhydrous ethanol and deionised water (weight ratio 10:1) is prepared; the silane coupling agent (KH-560 type) with a quality equal to 2% of nano-ZnO is added and stirred until uniformity;
3. a certain amount of nano-ZnO is added and stirred for 1 h under a high speed of 3000 rpm; the solution is then filtered at an ambient temperature and placed in a 105 °C oven for 4 h to achieve drying;
4. surface-treated nano-ZnO is obtained after grinding.

The lipophilicity test was performed to evaluate the surface treatment effect of nano-ZnO. The test process is as follows:

1. the original nano-ZnO and the surface-treated one with the same quality are poured into two beakers with 50 ml water; a thin layer is evenly spread on the water surface in each beaker;
2. methanol reagents are added to these two beakers until nano-ZnO is entirely wetted by methanol.

Lipophilicity was calculated as follows (Equation (1)):

$$Lipophilicity = \frac{V}{V+50}100\,(\%), \tag{1}$$

where V represents the added methanol content, ml.

The calculations showed that the lipophilicity of nano-ZnO before and after treatment was 26.5% and 57.9%, respectively, indicating a significant improvement after surface treatment.

1.3. Preparation of the nano-ZnO/SBS hybrid-modified asphalt binder samples

A high-speed shearing mixer was used to prepare the nano-ZnO/SBS hybrid-modified asphalt binder. The SBS modifier, which was 4% by weight to the base asphalt binder, and the designed amount of nano-ZnO were sheared together with the base asphalt binder at a speed of 6000 rpm for 30 min at 170 °C. The prepared nano-ZnO/SBS hybrid-modified asphalt binders were cured at 120 °C for 2 h.

The preparation process of the SBS-modified asphalt was the same as that of nano-hybrid-modified asphalt. A 4% SBS modifier was added to the base asphalt, which was sheared at 6000 rpm for 30 min and then placed in the oven at 120 °C for 2 h.

2. Determination of the optimum nano-ZnO dosage

The SBS modifier, which was 4% by weight of the base asphalt binder, was used herein because this amount is commonly used for SBS-modified asphalt binders in China (Xiong et al., 2005). Conventional tests on the modified asphalt binders with 4% SBS and nano-ZnO with seven different dosages, i.e., 0%–6%, were performed to determine the optimal nano-ZnO dosage in hybrid modification. Figure 2 presents the test results.

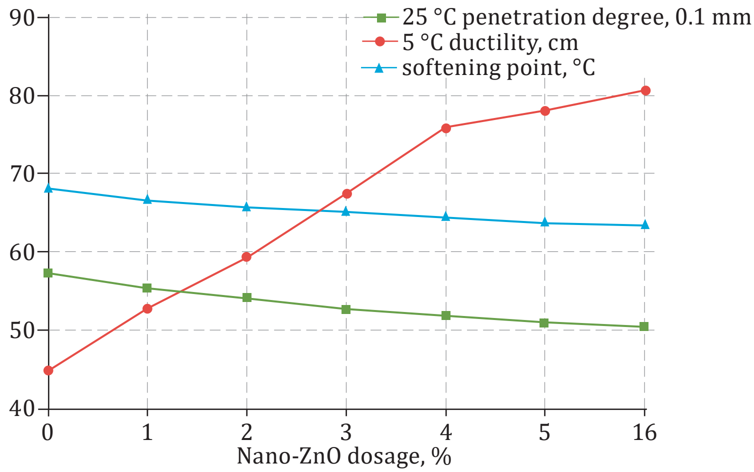


Figure 2. Three key indices of the nano-ZnO/SBS hybrid-modified asphalt binders

Figure 2 illustrates that the penetration gradually decreased with the increase of the amount of nano-ZnO mainly because nano-ZnO had a large surface area, which absorbs some macromolecule components within the base asphalt binder. Meanwhile, the ductilities gradually increased with the amount of nano-ZnO, indicating that nano-ZnO effectively improves the low-temperature property of the SBS-modified asphalt. However, the increase rate gradually decreased when the content exceeded 4%. Moreover, the softening point showed a decreasing trend when a maximum decrease of 4.7 °C was observed for 6% nano-ZnO content.

The optimum nano-ZnO dosage was selected to be 4% owing to the minor decrease in the penetration and softening point and the considerable increase in ductility for this dosage. The SBS-modified asphalt with 4% SBS and the nano-ZnO/SBS hybrid-modified asphalt with 4% SBS and 4% nano-ZnO were utilised for the subsequent tests.

3. Properties of the nano-ZnO/SBS hybrid-modified asphalt binder

3.1. Rheological properties determined by the dynamic shear rheometer tests

The high-temperature properties of the SBS-modified and nano-ZnO/SBS hybrid-modified asphalt binders were tested using a dynamic shear rheometer (DSR) with a 10 rad/s shear rate. Figure 3 presents the test results.

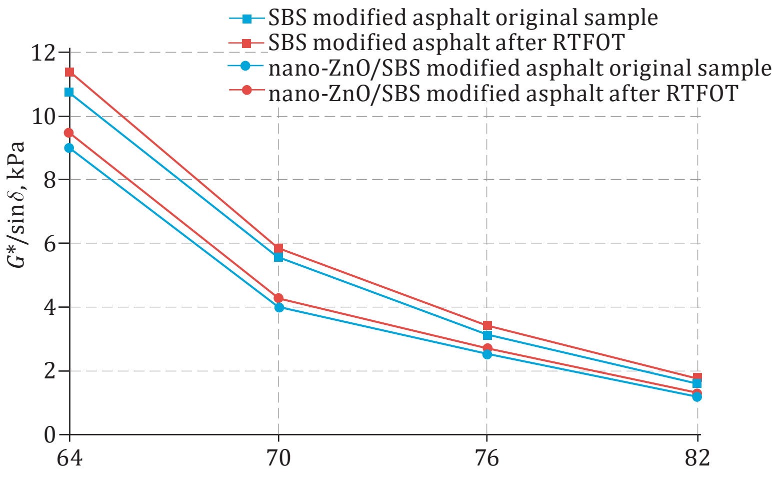


Figure 3. Rutting factor of the modified asphalt binder

As shown in Figure 3, the values of G*/sinδ, which denotes the rutting resistance index, were generally lower for the hybrid-modified asphalt binders than the SBS-modified binders, regardless of the temperatures tested, i.e., from 64 °C to 82 °C. The G*/sinδ values of the Rolling Thin-Film Oven Test aged samples (*JTG E20–2011 Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering*) also showed the same trend as the original binders. The PG grade of both the SBS single-modified binders was PG 76, similar to the hybrid-modified binders. Adding 4% nano-ZnO material in the SBS-modified asphalt binder indicated minor changes in the rutting resistance index and high-temperature grade.

3.2. Bending beam rheometer tests

A bending beam rheometer (BBR) was used to test the low-temperature property of the two types of modified asphalt binders. The test temperatures were −12 °C, −18 °C, and −24 °C. Figure 4 presents the bending creep stiffness modulus S test results and the creep curve slope *m*.

Figure 4 shows that at the same test temperature, the creep stiffness modulus *S* of the nano-ZnO/SBS hybrid-modified asphalt binder was lower, while the slope *m* of the creep curve was high compared to those of the SBS-modified binders. This phenomenon indicated that the low-temperature stress relaxation was better than the SBS-modified asphalt binder. The requirements of *S* no more than 300 MPa and *m* no less than

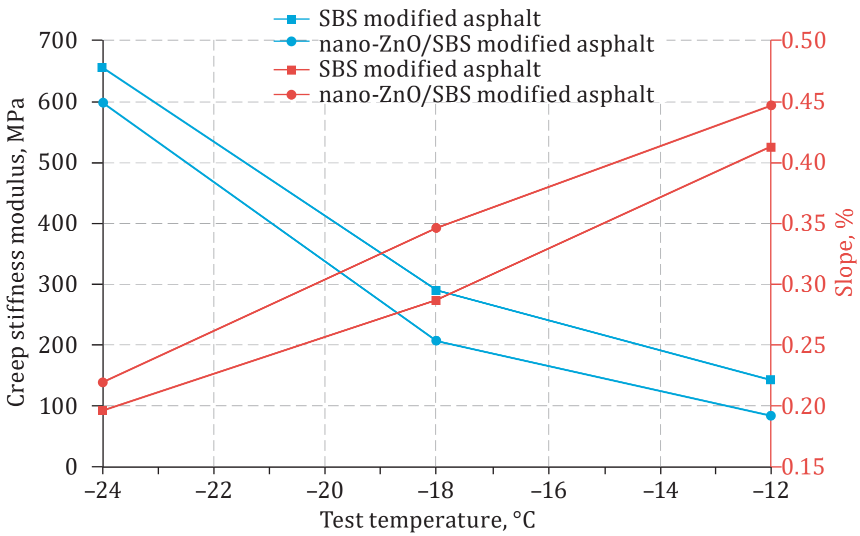


Figure 4. Test results of bending beam rheometer test

0.3 were met when the test temperature was −12 °C for the SBS-modified asphalt binders and −18 °C for the nano-ZnO/SBS hybrid-modified asphalt binders. That is, the low-temperature grades of these two asphalt binders were different after the nano-ZnO addition. The nano-ZnO/SBS hybrid-modified asphalt increased the low-temperature grade from −22 °C to −28 °C, consistent with the test results (Table 3), showing that the 5 °C ductilities were improved by increasing the nano-ZnO content.

3.3. Fluorescence tests

The SBS-modified asphalt and nano-ZnO/SBS hybrid-modified asphalt samples were placed in an aluminium tube with a 25 mm diameter and 140 mm length to study the effect of nano-ZnO on the SBS-modified asphalt segregation. The samples were placed at 163 °C for 12 h, 24 h, and 48 h and cooled in a refrigerator for at least 4 h. The top and bottom samples were then taken for the DSR test. Table 4 presents the test results.

The results showed that with the increase of the storage time, the $G^*/\sin\delta$ top–bottom ratio of the nano-ZnO/SBS hybrid-modified asphalt and the SBS-modified asphalt gradually decreased. Moreover, the $G^*/\sin\delta$ top–bottom ratio of the nano-ZnO/SBS hybrid-modified asphalt was consistently lower than that of the SBS-modified asphalt, indicating that nano-ZnO improves the storage stability of the latter.

Under the same preparation process and test conditions, a fluorescence microscopy analysis was performed on both SBS-modified and nano-ZnO/SBS hybrid-modified asphalt binder samples. Nano-ZnO did not produce fluoresce after being irradiated by laser. The yellow-green light in Figure 5 illustrates the distribution of the SBS modifiers in asphalt. Figure 5 depicts microscopic fluorescence images.

Table 4. Results of the rutting factor ($G^*/\sin\delta$) **

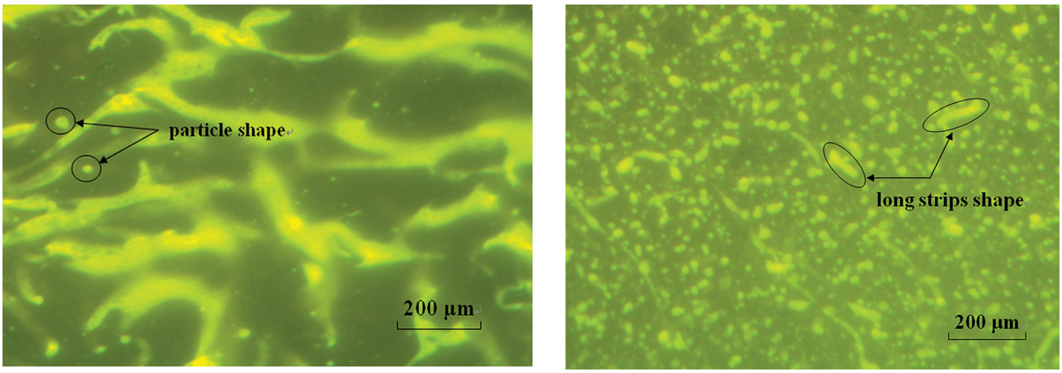
Storage time, h	SBS-modified asphalt			Nano-ZnO/SBS hybrid-modified asphalt		
	Top sample, kPa	Bottom sample, kPa	Top–bottom ratio	Top sample, kPa	Bottom sample, kPa	Top–bottom ratio
12	3.969	3.341	1.19	3.549	2.730	1.13
24	4.738	4.139	1.14	4.121	3.781	1.09
48	5.945	5.577	1.07	5.169	4.970	1.04

Note: **76 °C.

Figure 5a demonstrates that the SBS modifiers were unevenly and poorly dispersed in the base asphalt binder with long strips and small particles. Some of these modifiers showed the accumulation phenomenon. SBS was much more evenly dispersed in the form of tiny particles. The long strips were considerably reduced, thinner, and shorter in Figure 5b. This phenomenon indicated that the separation effect was improved after adding and mixing nano-ZnO. The improved effect was due to the large specific surface area and the many unpaired electrons of nano-ZnO, which physically combined well with polymer-modified asphalt. Meanwhile, nano-ZnO showed strong surface activity. The presence of free electrons on its surface (Kang et al., 2010) made cross-linking with polymer groups easy.

4. Properties of the nano-ZnO/SBS hybrid-modified asphalt mixtures

A dense-graded asphalt mixture of AC-13C was selected. The Marshall test shows that the optimum asphalt dosages of the SBS-modified asphalt were 4.6% and 4.7%, respectively. The high-temperature stability, low-temperature crack resistance, and water stability tests were conducted to evaluate the SBS-modified asphalt and nano-ZnO/SBS hybrid-modified asphalt mixtures.



a) SBS-modified asphalt **b)** nano-ZnO/SBS hybrid-modified asphalt

Figure 5. Fluorescence micrographs of the two types of modified asphalt binders

4.1. High-temperature performance

The rutting test by *JTG E20–2011* was conducted to investigate the high-temperature property of the two types of asphalt mixture. The rutting test temperature was 60 °C. The pressure was 0.7 MPa. The dynamic stability index was used to evaluate the high-temperature rutting resistance of the asphalt mixture. Equation (2) presents the calculation formula. Figure 6 shows the test results.

$$DS = \frac{(t_2 - t_1)N}{d_2 - d_1} C_1 C_2, \tag{2}$$

where DS stands for the dynamic stability of the asphalt mixture, times·mm^{-1}; d_1– the deformation corresponding to a time t_1, mm; d_2 – the deformation corresponding to a time t_2, mm; C_1 – the type coefficient of the testing machine (test value 1.0); C_2 – the specimen coefficient (test value 1.0); N – the round-trip rolling speed of the test wheel, which is usually 42 times per minute.

As depicted in Figure 6, the dynamic stability of the SBS-modified asphalt mixture was 4013 times·mm^{-1}, while that of the nano-ZnO/SBS hybrid-modified asphalt mixture was 3795 times·mm^{-1}. In other words, the dynamic stability of the nano-ZnO/SBS hybrid-modified asphalt mixture was reduced by 5.4% compared to that of the SBS-modified asphalt mixture. This decreasing trend of mixtures was consistent with that shown by the DSR results for the asphalt binders. Although the dynamic stability of the mixtures decreased after the nano-ZnO addition, the high-temperature properties of the nano-ZnO/SBS hybrid-modified asphalt still met the technical requirements of the specification, i.e., DS was no less than 2400 times·mm^{-1}.

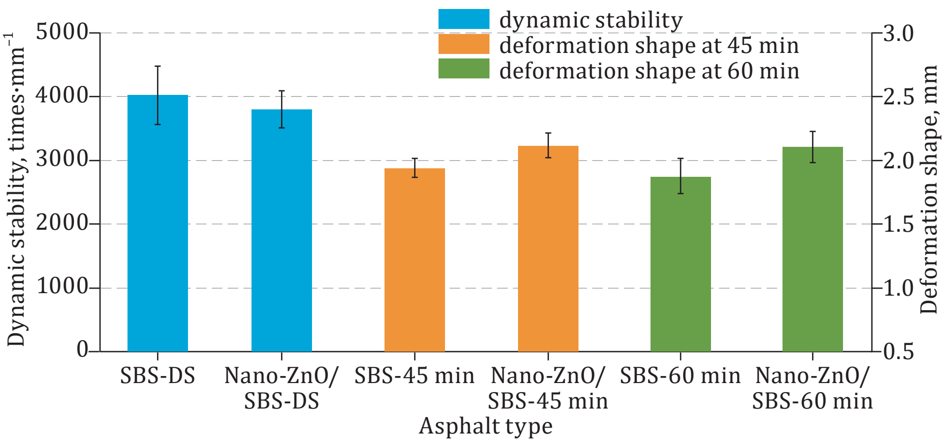


Figure 6. Results of the rutting test

4.2. Low-temperature crack resistance

The trabecular bending test (*JTG E20–2011*) was performed to evaluate the deformation performance of the asphalt mixture. The test temperature was −10 °C. The loading rate was 50 mm/min. The indicators of the flexural tensile strength and the maximum bending strain were used to evaluate the low-temperature crack resistance. The calculation formulas below are Equations (3)–(5). Figure 7 displays the test results.

$$\sigma_0 = \frac{3LF_0}{2bh^2}10^{-6}, \tag{3}$$

$$\varepsilon(t) = \frac{6hd(t)}{L^2}, \tag{4}$$

$$S(t) = \frac{\sigma_0}{\varepsilon(t)}, \tag{5}$$

where σ_0 – the creep bending tensile stress of the specimens, MPa; $\varepsilon(t)$ – the bending tensile strain at the bottom of the beam; $S(t)$ – the bending creep stiffness modulus of the specimens, MPa; t_1 and t_2– the starting and ending times, respectively, of the straight line segment in the creep stability period, s; ε_1 and ε_2 – correspond to the creep strain at a time t_1 and t_2, respectively; F_0 – the load is borne by the specimen during the test loading, N; b – the cross-section width m; h – the interview piece height, m; L – the specimen span, m; $d(t)$ – the mid-span deflection varying with time t during loading, m.

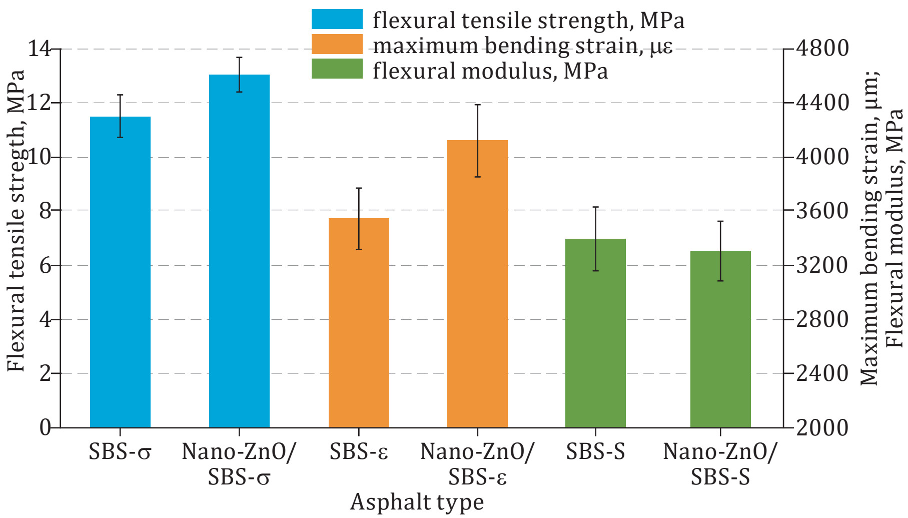


Figure 7. Results of the trabecular bending test

Figure 7 shows that the flexural tensile strength and the maximum bending strain of the asphalt mixture increased by 13.4% and 16.4%, respectively, after adding nano-ZnO. In other words, the low-temperature properties of the SBS-modified asphalt, like crack and deformation resistance, of the asphalt mixture were significantly improved by the nano-ZnO addition. The main reason for this result is that the cohesion within the SBS-modified asphalt was improved as the temperature sensitivity of the nano-ZnO/SBS hybrid-modified asphalt decreased. Moreover, the adhesion between the asphalt binder and the aggregates also increased, further reflecting the advantages of the low-temperature performance of the nano-ZnO/SBS hybrid-modified asphalt mixture.

4.3. Moisture susceptibility

The moisture susceptibility performance of the two asphalt mixtures was tested through the submerged Marshall and freeze-thaw splitting tests (*JTG E20–2011*). The submerged Marshall stability test before and after immersion was performed to evaluate the spalling resistance of the asphalt mixture damaged by water. The water temperature was 60 °C, and the loading rate was 50 mm/min. Then residual stability indicators were used to evaluate the water stability. The calculation formula is presented as Equation (6):

$$MS_0 = \frac{MS_1}{MS}100, \tag{6}$$

where MS_0 – the residual stability of the specimens after immersion, %; MS_1 – the stability of specimens after water immersion for 48 h, kN; MS – the stability of the specimen in water for 0.5 h, kN.

For the freeze-thaw splitting test, the freeze-thaw cycle on the specimens was conducted at −18 °C for 16 h. The tensile strength ratios of the freeze-thaw and non-freeze-thaw specimens were then measured and calculated. The tensile strength ratio (TSR) indicators were used to evaluate the water stability. Equations (7)–(9) present the calculation formula. Figure 8 exhibits the test results.

$$R_{T1} = 0.006287\frac{P_{T_1}}{h_1}, \tag{7}$$

$$R_{T2} = 0.006287\frac{P_{T_2}}{h_2}, \tag{8}$$

$$TSR = \frac{\overline{R}_{T_2}}{\overline{R}_{T_1}}100, \tag{9}$$

where the TSR stands for the tensile strength ratio of the freeze-thaw splitting test, %; $\overline{R}_{T_1}$ – the average value of splitting tensile strength of the first group of specimens without freeze-thaw cycles, MPa; $\overline{R}_{T_2}$ – the average value of splitting tensile strength of the second group of specimens after the freeze-thaw cycles, MPa; R_{T_1} – the splitting tensile strength of the first group of individual specimens without freeze-thaw cycles, MPa; R_{T_2} denotes the splitting tensile strength of the second group of individual specimens subjected to freeze-thaw cycles, MPa; P_{T_1} – the test load values of the first group of individual specimens, N; P_{T_2} – the test load values of the second group of individual specimens, N; h_1 – the height of each specimen in the first group, mm; h_2 – the height of each specimen in the second group, mm.

Compared to the SBS-modified asphalt mixture, the residual stability and the TSR of the nano-ZnO/SBS hybrid-modified asphalt mixture increased by 4.3% and 4.8%, respectively (Figure 8). These increased trends indicated that the nano-ZnO addition improved the water damage resistance of the SBS-modified asphalt mixture to a certain extent because the proportion of the structural asphalt binder in the mixture was increased when nano-ZnO was added, which consequently improved the water stability. The large surface area of nano-ZnO made the nano-ZnO/SBS hybrid-modified asphalt form a strong surface effect, which improved the proportion of structural asphalt and the adhesion between asphalt and mineral aggregate.

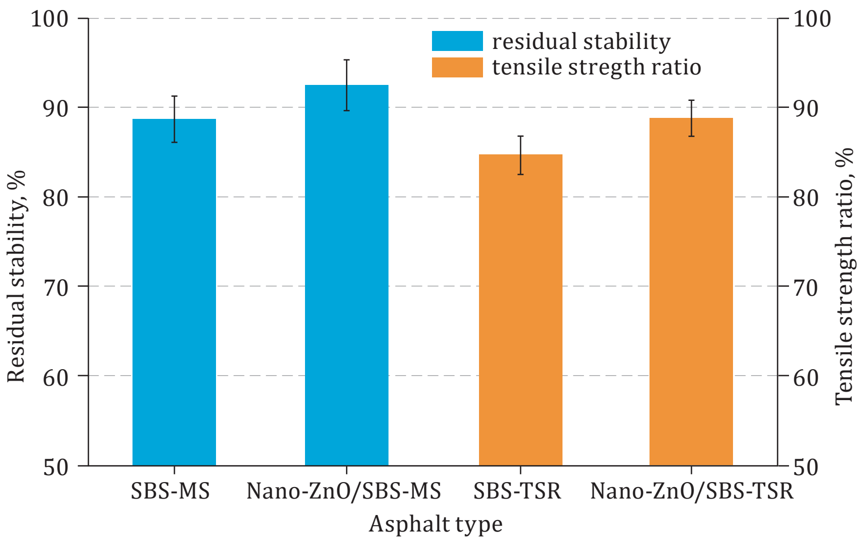


Figure 8. Results of the submerged Marshall and freeze-thaw splitting tests

Conclusions

This study determined the optimum nano-ZnO dosage of hybrid-modified asphalt binders. The dynamic shear rheometer, bending beam rheometer, and fluorescence microscopy tests were performed to evaluate the properties of the nano-ZnO/SBS hybrid-modified asphalt binders. The rutting, trabecular bending, submerged Marshall, and freeze-thaw splitting tests were conducted on the asphalt mixtures. The following conclusions are drawn from the research results.

1. With the increasing nano-ZnO content, the penetration and softening point of the nano-ZnO/SBS hybrid-modified asphalt binder gradually decreased while the ductility gradually increased. The optimal nano-ZnO dosage selected in the conventional asphalt binder test was 4%.
2. The rutting factor of the nano-ZnO/SBS hybrid-modified asphalt was slightly lower than that of the SBS-modified asphalt. Nano-ZnO slightly affected the high-temperature performance of the SBS-modified asphalt binder. The high-temperature grades of the two binders were the same, but the low-temperature grade of the SBS-modified asphalt binder changed from −22 °C to −28 °C when nano-ZnO was added.
3. The particle size of the nano-ZnO and SBS inside the base asphalt binder was small and evenly distributed. Nano-ZnO improves the dispersion effect of SBS inside the base asphalt binder.
4. Despite the slight decrease in the high-temperature performance of the SBS-modified asphalt mixture after incorporating 4% nano-ZnO, it still met the specifications. The flexural tensile strength and the maximum bending strain of the nano-ZnO/SBS hybrid-modified asphalt mixture were 1.13 and 1.16 times those of the SBS-modified asphalt mixture. Moreover, the residual stability and the tensile strength ratio improved by 4.3% and 4.8%, respectively, compared to the SBS-modified asphalt mixtures. That is, nano-ZnO improved the low-temperature and water stability performances of the SBS-modified asphalt mixture.

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