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Research Article

Effects of Organic Nano Calcium Carbonate on Aging Resistance of Bio-Asphalt

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To improve the aging resistance of bioasphalt and explore the impact of organic nano calcium carbonate ($CaCO_3$) on the aging resistance of bioasphalt, the thin-film oven test (TFOT), basic property test, rheological test, four components test, and infrared spectrum test were used to study the bioasphalt with different organic nano-$CaCO_3$ contents. The results show that the addition of organic nano-$CaCO_3$ increases the residual penetration, residual ductility, and residual softening point of bioasphalt. Within the range of test dosage, the greater the dosage, the more significant the effect is. With the increase of organic nano-$CaCO_3$ content, the complex modulus aging index (*GAI*) of bioasphalt gradually decreases, and the colloidal instability index (I_C) gradually decreases. When the content is 6%, the *GAI* of organic nano-$CaCO_3$ modified bioasphalt is less than that of matrix asphalt. The nanoscale super large specific surface area of organic nano-$CaCO_3$ has an adsorption effect on the light components in bioasphalt, which slows down the volatilization loss of light components and the oxidation process of asphalt. The addition of organic nano-$CaCO_3$ helps to reduce the change rate of the carbonyl index and sulfoxide index.

1. Introduction

Bio-oil is a new energy product obtained by distillation, separation, blending, and extraction of biomass such as sawdust, straw, gutter oil, and animal excreta [1]. Bio-oil and petroleum asphalt have similar element compositions and properties and have good compatibility. The preparation of bioasphalt by adding bio-oil to petroleum asphalt can realize the utilization of waste and has gradually become a hot topic in the research field of road materials [1, 2].

At present, many studies have been carried out on the preparation and properties of bioasphalt [3–6]. Mills-Beale et al. [7] pyrolyzed and liquefied pig manure under anaerobic conditions of 380°C and 40 MPa to prepare bio-oil. Their research showed that the bio-oil would reduce the viscosity of asphalt. Fini et al. [8, 9] also used animal manure and other biomass materials to prepare bio-oil by high-pressure distillation to make bioasphalt. It was found that bio-oil only improved the low-temperature performance of asphalt. Wen et al. [10] took the bio-oil chemically processed from waste gutter oil as the research object, studied the performance of petroleum asphalt after modification, and found that the rutting resistance of asphalt decreased to a certain extent.

Mousavi et al. [11] used molecular dynamic simulation to study the modification mechanism of bio-oil on matrix petroleum asphalt from the multiscale reverse. It was found that the addition of bio-oil makes the asphaltenes in petroleum asphalt enrichment and has a certain impact on the characteristics of asphalt, including but not limited to the medium temperature fatigue and low-temperature crack resistance of asphalt. Hill et al. [9] comprehensively considered the excellent low-temperature cracking resistance of bioasphalt and the good high temperature deformation resistance of recycled asphalt mixture and studied the effect of bio-oil on the low-temperature performance of hot recycled asphalt mixture based on indirect tensile tests and acoustic emission tests. The test results showed that the low-temperature crack resistance of bioasphalt hot recycled asphalt mixture is better than that of hot mixed asphalt mixture. Pouget and Loup [12] conducted thermal aging treatments on bioasphalt mixtures and evaluated the linear viscoelasticity and low-temperature crack resistance of biologically modified asphalt mixtures. It was found that there were slight differences in modulus, tensile strength, and thermal stress between the bioasphalt mixture and petroleum asphalt mixture.

However, due to the poor thermal stability of bio-oil, the prepared bioasphalt is easy to age at high temperatures, which affects the performance and durability of bioasphalt and its mixture [13–15]. Studies have shown that the addition of carbon black, antioxidants, and UV absorbers to asphalt can improve the aging resistance of the asphalt to a certain extent [16–18]. In recent years, nanomaterials have been used to enhance the antiaging properties of asphalt and polymers. Yu et al. [19] studies have found that organic montmorillonite as a layered silicate can obviously improve the thermal oxidation resistance of asphalt. In addition, studies have shown that nano-$CaCO_3$, nano-TiO_2, nano-ZnO, and nano-SiO_2 have excellent aging resistance [20–22]. The effect of nano calcium carbonate on the antiaging properties of asphalt was studied by different simulation aging tests [23]. The results showed that nano calcium carbonate can effectively improve the antiaging properties of asphalt. At the same time, the compatibility of nano calcium titanate with asphalt can be significantly enhanced after organic treatment.

To improve the aging resistance of bioasphalt and explore the impact of organic nano calcium carbonate on the aging resistance of bioasphalt, the TFOT aging test was used to age bioasphalt with different organic nano calcium carbonate content. Basic property tests (penetration, ductility, and softening point), rheological tests, four components (saturates, aromatics, resins, and asphaltenes) test, and infrared spectrum test (Fourier transform infrared reflection, FTIR) were carried out to study the bioasphalt with different organic nano calcium carbonate content, and the effects of organic nano calcium carbonate on the aging resistance of bioasphalt were evaluated by comparing the matrix asphalt with the bioasphalt without organic nano calcium carbonate.

2. Materials and Experimental Design

2.1. Materials

2.1.1. Matrix Asphalt. The matrix asphalt used in the study is Sinopec Maoming Petrochemical Donghai 70 # Class A road asphalt. Performance indicators are shown in Table 1. All indicators meet the specification requirements.

Table 1: Performance indexes of matrix asphalt.

Test items	Test results	Test methods
Penetration (25°C)/0.1 mm	65.1	T0604-2011
Ductility (15°C)/cm	>100	T0606-2011
Softening point (°C)	52.5	T0605-2011
Density (15°C)/($g{\cdot}cm^{-3}$)	1.029	T0625-2011
Flash point (°C)	305	T0611-2011
Solubility (%)	99.7	T0607-2011
Penetration index PI	0.58	T0604-2011

2.1.2. Bio-Oil. The bio-oil (shown in Figure 1) used in the study is produced by a company in Shandong, which is mainly produced by rapid pyrolysis of waste biomass such as straw and sawdust. The main performance indexes of bio-oil are shown in Table 2.

Figure 1: : Bio-oil.

Table 2: Performance indexes of bio-oil.

Test items	Test results
pH	3.6
Moisture content (%)	9.8
Viscosity (60°C)/(Pa·s)	1.03
Density (15°C)/($g{\cdot}cm^{-3}$)	1.13

2.1.3. Organic Nano Calcium Carbonate. The nano calcium carbonate used in the study is produced by Shanghai Chenqi Chemical Technology Co., Ltd., and its main performance indexes are shown in Table 3.

Table 3: Performance indexes of nano-$CaCO_3$.

Test items	Test results
Appearance	White powder
Specific surface area ($cm^2{\cdot}g^{-1}$)	25000 ± 500
Average particle size (nm)	25
Moisture content (%)	0.25

Due to the large specific surface area of nano calcium carbonate, agglomeration is easy to occur in the asphalt. Therefore, nano calcium carbonate was organically treated to improve the dispersion of nano calcium carbonate in asphalt in this study. The specific operation steps are as follows: (1) first we measure 300 ml of deionized water and 100 g of nano calcium carbonate, then we add nano calcium carbonate into deionized water, place it in a water bath, and control the temperature at 80°C, (2) then we add 40 ml of anhydrous ethanol solution of stearic acid with a mass fraction of 5% and stir it with a mixer at the rate of 200 r/min for 2 hours, (3) then we filter the solution and dry the filter cake at a constant temperature at 80°C, (4) Finally, the organic nano calcium carbonate was obtained by grinding, which was used for subsequent experiments.

2.2. Preparation of Test Asphalts

2.2.1. Preparation of Bioasphalt. The matrix asphalt is heated and melted in an oven at 160°C. In order to evenly mix the bio-oil into the matrix asphalt, the set quality bio-oil is added to the molten matrix asphalt several times, and a high-speed shear is used to shear at the speed of 3500 r/min for 10 min. Following the shearing, it is manually stirred for 5 min to disperse the bubbles in the asphalt. The bioasphalt test sample can be obtained after completing the above steps. In this study, the content of bio-oil was set as 15% of the mass of matrix asphalt. The preparation process of bio-asphalt is shown in Figure 2.

2.2.2. Preparation of Organic Nano-$CaCO_3$ Modified Bioasphalt. The organic nano-$CaCO_3$ modified bioasphalt is also prepared by the above shear mixing method. The content of organic nano-$CaCO_3$ is added according to the mass fraction of matrix asphalt, and the test asphalt is numbered, as shown in Table 4.

2.3. Test Methods

2.3.1. Thin Film Oven Test (TFOT). The aged asphalt of different test asphalt samples prepared according to the test method of T0609-2011 in Test Specification for Asphalt and Asphalt Mixture in Highway Engineering (JTG E20-2011), namely the thin-film oven test (TFOT). In the TFOT, the aging temperature of asphalt samples is controlled at (163 ± 1)°C and the aging time is controlled at 5 hours. The asphalt samples' numbers after aging are marked as MA-A, BM-A, 2CBM-A, 4CBM-A, and 6CBM-A, respectively, and compared with the asphalt samples before aging.

2.3.2. Basic Property Tests. The asphalt samples before and after aging are tested according to the test methods of T0604-2011, T0605-2011, and T0606-2011 in the test specification E20-2011 to evaluate the influence of organic nano calcium carbonate on the basic property indexes of penetration, ductility, and softening point of bioasphalt before and after aging.

2.3.3. Rheological Tests. According to the test method T0628-2011 in the test specification E20-2011, the rheological properties of asphalt before and after aging are tested on the DHR-1 dynamic shear rheometer (DSR). The diameter of the parallel plate is 25 mm, the space between plates is 1 mm, the frequency is set as 10 rad/s, and the scanning temperature is 52~82°C with a 6°C of interval. The complex modulus index (G^*) is recorded and the complex modulus aging index is calculated according to :

$$GAI = \frac{G_A^*}{G_0^*} \tag{1}$$

where G_A^* is complex modulus after aging and G_0^* is complex modulus before aging.

2.3.4. Four Components Test. Iatroscan MK-6s TLC-FID (thin layer chromatography-flame ionization detector) was used to quantitatively analyze the changes of components of asphalt before and after aging. According to the blending theory of asphalt components, the colloidal instability index, as shown in (2), can be used to evaluate the stability of the asphalt colloidal structure. The colloidal instability index (I_c) represents the specific gravity of gel/sol in asphalt, and the lower the value is, the more stable the asphalt colloid structure is [12].

$$I_c = \frac{C_{Saturates} + C_{Asphaltenes}}{C_{Resins} + C_{Aromatics}} \tag{2}$$

2.3.5. Fourier Transform Infrared (FTIR). A Nexus 870 Fourier transform infrared spectrometer was used to analyze the chemical structure of the asphalt before and after aging. In the tests, the scanning wavenumber range was set to be 4000 cm^{-1}~500 cm^{-1}. The absorption bands of carbonyl (C=O) and sulfoxide (S=O) in the asphalt correspond to the absorption bands at 1700 cm^{-1} and 1030 cm^{-1} in the infrared spectrum. The carbonyl index (CI) and sulfoxide index (SI) were calculated according to equations (3) and (4) to characterize the relative content of carbonyl and sulfoxide groups [24].

$$CI = \frac{A_{1700cm^{-1}}}{A_{2000\sim600cm^{-1}}} \tag{3}$$

$$SI = \frac{A_{1030cm^{-1}}}{A_{2000\sim600cm^{-1}}} \tag{4}$$

where $A_{1700cm^{-1}}$ is the absorption band area with 1700 cm^{-1} wavenumber as the central carbonyl peak in the infrared spectrum, $A_{1030cm^{-1}}$ is the absorption band area with 1030 cm^{-1} wavenumber as the central sulfoxide peak in the infrared spectrum, and $A_{2000\sim600cm^{-1}}$ is the area of 2000~600 cm^{-1} wavenumber absorption band in the infrared spectrum. The change rate of carbonyl index (R_{CI}) and sulfoxide index (R_{SI}) before and after aging are calculated, respectively, as

$$R_{CI} = \frac{CI_A - CI}{CI}, \quad R_{SI} = \frac{SI_A - SI}{SI} \tag{5}$$

where CI and CI_A are carbonyl indexes before and after aging, correspondingly, SI and SI_A are sulfoxide index before and after aging.

3. Results and Discussion

3.1. Effects of Organic Nano-$CaCO_3$ on Macro Properties of Bioasphalt before and after Aging

3.1.1. Effects on Basic Properties of Bioasphalt before and after Aging. The test results of penetration, ductility, and

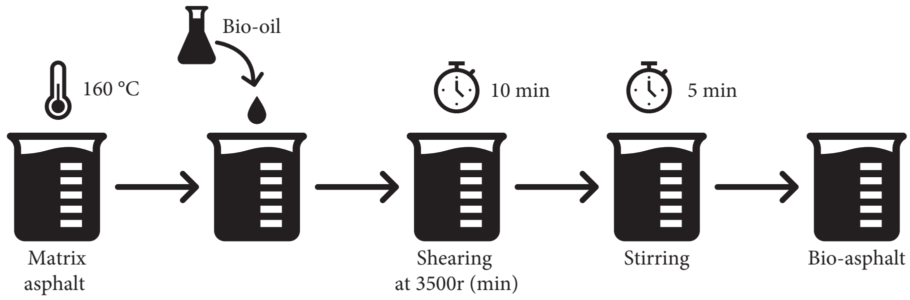


FIGURE 2: Preparation process of bioasphalt.

TABLE 4: Code abbreviation of test asphalts.

Test asphalt material composition	Test asphalt no.
Matrix asphalt	MA
15%Bio-oil + matrix asphalt	BM
2%$CaCO_3$ + 15% bio-oil + matrix asphalt	2CBM
4%$CaCO_3$ + 15% bio-oil + matrix asphalt	4CBM
6% $CaCO_3$ + 15% bio-oil + matrix asphalt	6CBM

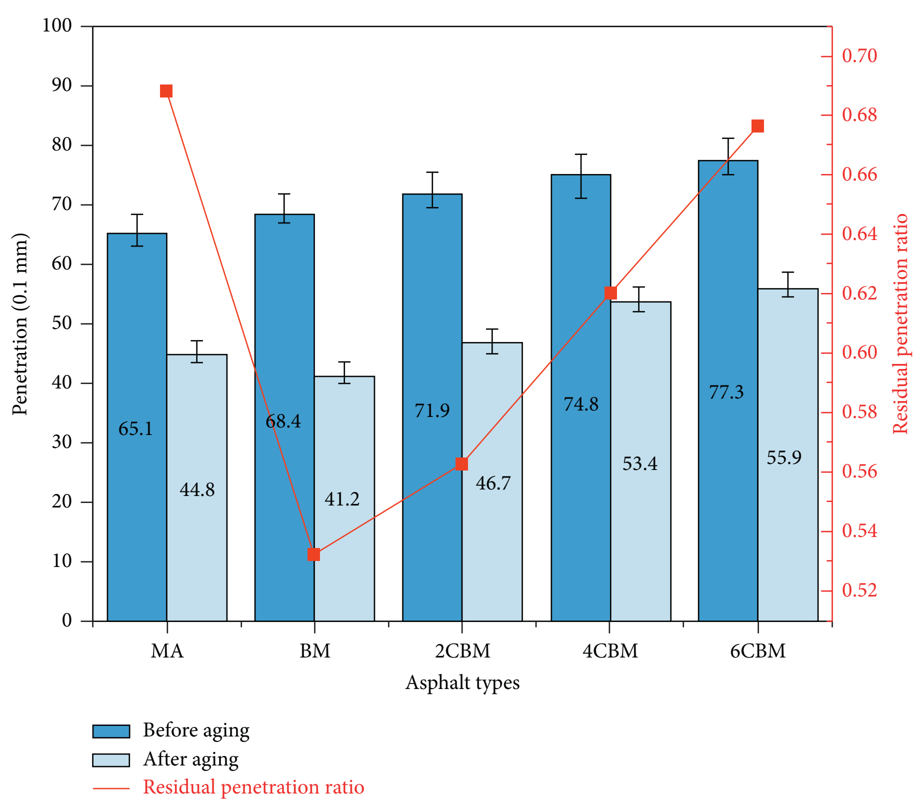


FIGURE 3: Effect of organic nano-$CaCO_3$ on the penetration of bioasphalt before and after aging.

softening point of different test asphalt samples before and after aging are shown in Figures 3–5.

Based on Figure 3, it can be seen from the change trend of the penetration of test asphalt before aging that, compared with the matrix asphalt (MA), the penetration is greatly improved after adding bio-oil. Compared with the bio-asphalt (BM), the penetration of 2CBM, 4CBM, and 6CBM gradually decreases slightly after adding 2%, 4%, and 6% organic nano calcium carbonate, respectively. The penetration changes of the asphalt before and after aging are further compared and analyzed. The residual penetration ratios (ratio of penetration after aging and that before aging) are calculated to be 0.69, 0.53, 0.56, 0.62, and 0.68, respectively. It is found that compared with the matrix asphalt, the residual penetration ratio decreases significantly after adding bio-oil and gradually increases after adding organic nano calcium carbonate, When the content of organic nano calcium carbonate is more than 4%, the residual penetration ratio meets the technical requirements of 70 # Class A road petroleum asphalt in the technical code for construction of highway asphalt pavement in China (JTG F40-2004).

By analyzing the results of the 10°C ductility test in Figure 4, it can be concluded that the ductility of bioasphalt (BM) before aging is 30.1% higher than that of matrix asphalt (MA), and the ductility decreases slightly after adding organic nano calcium carbonate (2CBM, 4CBM, and 6CBM). However, there are great differences in the change law of residual ductility of test asphalt after aging, and the residual ductility of bioasphalt (BM) after aging is 48.2% lower than that of matrix asphalt (MA). The residual ductility was improved after adding organic nano calcium carbonate. The residual ductility of bioasphalt added with 6% organic nano calcium carbonate increased by 62.8% to 36.8 cm, but it was still less than the 43.6 cm of the matrix asphalt.

It can be seen from Figure 5 that the softening point has a similar change law to the ductility at 10°C. After adding bio-oil, the softening point before aging decreases, that is, the asphalt softens, while the softening point increases significantly after aging. After adding organic nano calcium carbonate, this changing trend is alleviated.

It can be seen from the results of the penetration, 10°C ductility, and softening point test that, compared with the matrix asphalt, after adding bio-oil, the penetration and ductility become larger, and the softening point becomes smaller, while after adding organic nano calcium carbonate, the residual penetration and ductility increase, and the softening point decreases after aging. The more the content increases, the more significant the effect is, and the performance level of bioasphalt with organic nano calcium carbonate is close to that of base asphalt.

3.1.2. Effect of on Rheological Properties of Bioasphalt before and after Aging. The complex modulus (G^*) of different test asphalt samples before and after aging is obtained through rheological test, and the complex modulus aging index is calculated according to equation (1). The complex modulus curves and the calculation results of complex modulus aging index are shown in Figures 6(a) and 6(b), respectively.

In general, the smaller the complex modulus aging index (GAI), the better the aging resistance of asphalt. It can be seen from Figure 6(b) that GAI is greater than 1, indicating that the asphalts become harder after aging, which shows that the complex modulus becomes larger. Compared with the matrix asphalt, the GAI increased significantly at different test temperatures after adding bio-oil, indicating that the aging resistance decreased. After the addition of organic nano calcium carbonate, compared with the BM, the GAI gradually decreases with the increase of the content of organic nano calcium carbonate, indicating that the addition of organic nano calcium carbonate helps to improve the aging resistance of bio-asphalt. When the content is 6%, the GAI of organic nano calcium carbonate modified bio-asphalt is less than that of matrix asphalt.

3.2. Effects of Organic Nano-$CaCO_3$ on Microstructure of Bioasphalt before and after Aging

3.2.1. Effect on Components of Bioasphalt before and after Aging. The four components test results of different test asphalts are shown in Figure 7, and the calculation results of the colloidal instability index are shown in Figure 8.

When asphalt is aged, the proportion of each fraction will change with the advancement of the aging process, in which the light components, saturates and aromatics, are mainly transformed into resins and further transformed into asphaltenes. It can be seen from Figure 7 that after aging, the saturates content and aromatics content of different test asphalt samples decrease to a certain extent, and the proportion of resins and asphaltenes increases. Compared with the proportion of the matrix asphalt (MA) and the bioasphalt (BM) before aging, after adding bio-oil, the saturates content increased by 3.7%, the aromatics content increased by 1.7%, the resins decreased by 4.9% and the asphaltenes decreased by 0.5%. It means that the addition of bio-oil introduces a large amount of saturates content and aromatics content into the matrix asphalt. Comparing the components of bioasphalt before and after aging (BM and BM-A), it is found that after TFOT aging, the light components decreased significantly, the saturates decreased by 13.2%, the aromatics decreased by 6.5%, and the proportion of resins and asphaltenes increased, mainly resins increased by 15.4%. Comparing 2CBM-A, 4CBM-A, and 6CBM-A fractions, it is found that after the addition of organic nano calcium carbonate, the trend of reducing light components and increasing resins and asphaltenes is alleviated, however, the ratio of resins and asphaltenes does not reach the level of the matrix asphalt.

According to the calculation results of the colloidal instability index in Figure 8, compared with MA and BM, the original fraction proportion of the matrix asphalt is changed after adding bio-oil. Compared with that of MA, the I_c of BM increases from 0.333 to 0.393, and the colloidal stability becomes worse. However, after TFOT aging, the light fractions decrease by volatilization, while the I_c of BM-A decreases to 0.351, and the colloidal stability of bioasphalt is improved. After the addition of organic nano calcium carbonate, the I_c of 2CBM-A, 4CBM-A, and 6CBM-A gradually decreased, indicating that the addition of organic nano calcium carbonate is helpful to improve the colloidal stability of bioasphalt after short-term aging.

3.2.2. Effect on Chemical Structure of Bioasphalt before and after Aging. The infrared spectra of different asphalts are shown Figure 9. The calculation results of carbonyl index (CI) and sulfoxide index (SI), and change rates of them of different test asphalt samples before and after TFOT aging are shown in Figures 10 and 11, respectively.

It can be seen from Figure 10 that the carbonyl index of asphalt after aging is greater than that before aging. The asphalt undergoes an oxidation process and the decrease of small molecular weight compounds and the continuous

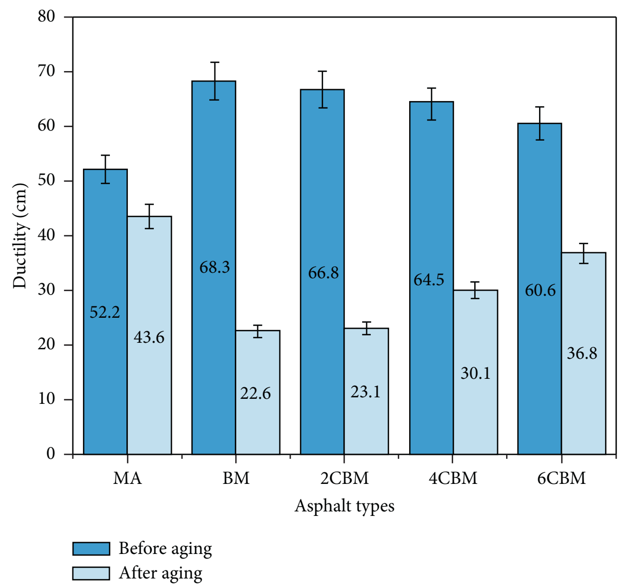


Figure 4: Effect of organic nano-$CaCO_3$ on the ductility of bioasphalt before and after aging.

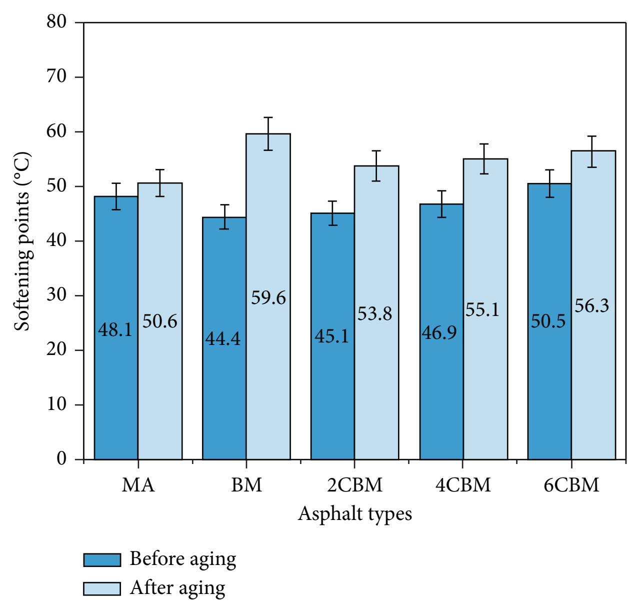


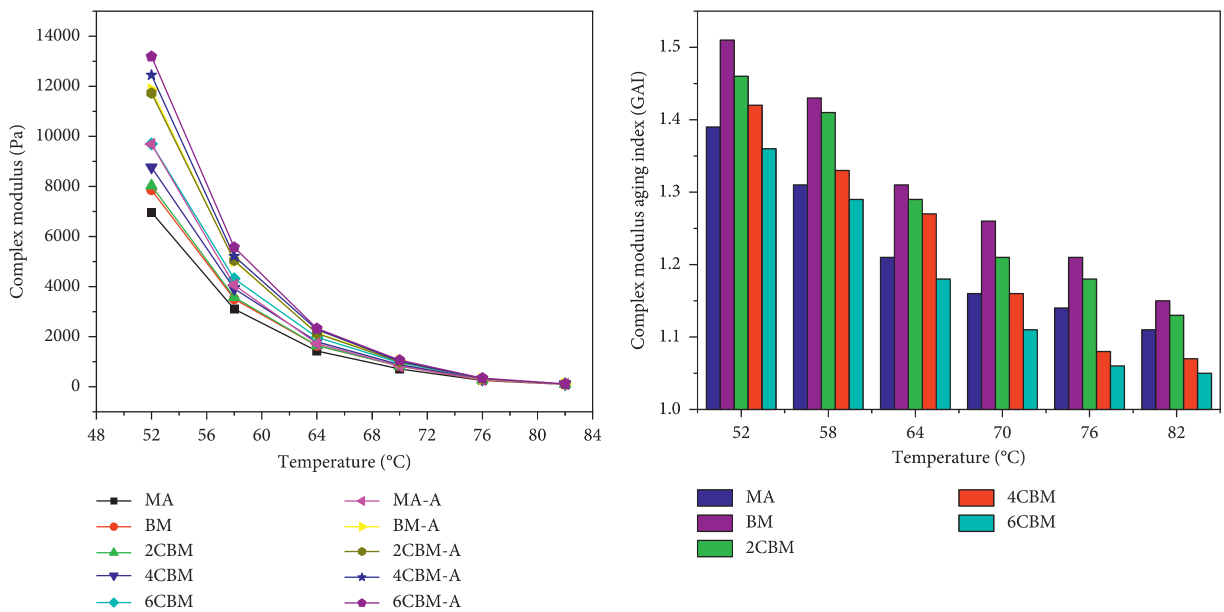
Figure 5: Effect of organic nano-$CaCO_3$ on softening point of bioasphalt before and after aging.

increase of large molecular weight compounds result in the content of carbonyl functional groups increasing after aging.

The change rate of the carbonyl index can characterize the aging resistance of asphalt. The smaller the change rate of the carbonyl index is, the stronger the aging resistance is [25]. The order of change rate of carbonyl index of the different test asphalt samples is 6CBM < MA < 4CBM < 2CBM < BM. Compared with the BM, after adding organic nano calcium carbonate, the change rate of the carbonyl index of 2CBM, 4CBM, and 6CBM gradually decreases, and the aging resistance increases with the increase of the content within the range of test contents. When the content is 6%, the change rate of the 6CBM carbonyl index is less than MA.

It can be seen from Figure 11 that the order of sulfoxide index change rate of different test asphalt samples is MA < 6CBM < 4CBM < 2CBM < BM, indicating that the addition of organic nano calcium carbonate helps to reduce the sulfoxide index change rate, and the greater the amount, the more significant the effect is. However, within the range of test amounts, the sulfoxide index change rate of organic nano calcium carbonate modified bioasphalt is greater than that of the matrix asphalt.

The organic nano calcium carbonates the effectiveness in improving the aging resistance of bioasphalt in accordance with the calculation results of the carbonyl index and sulfoxide index. Compared with the effects of the organic nano calcium carbonates on the carbonyl index and



(a) (b)

Figure 6: Effect of organic nano-$CaCO_3$ on rheological properties of bio-asphalt before and after aging. (a) Complex modulus. (b) Complex modulus aging index.

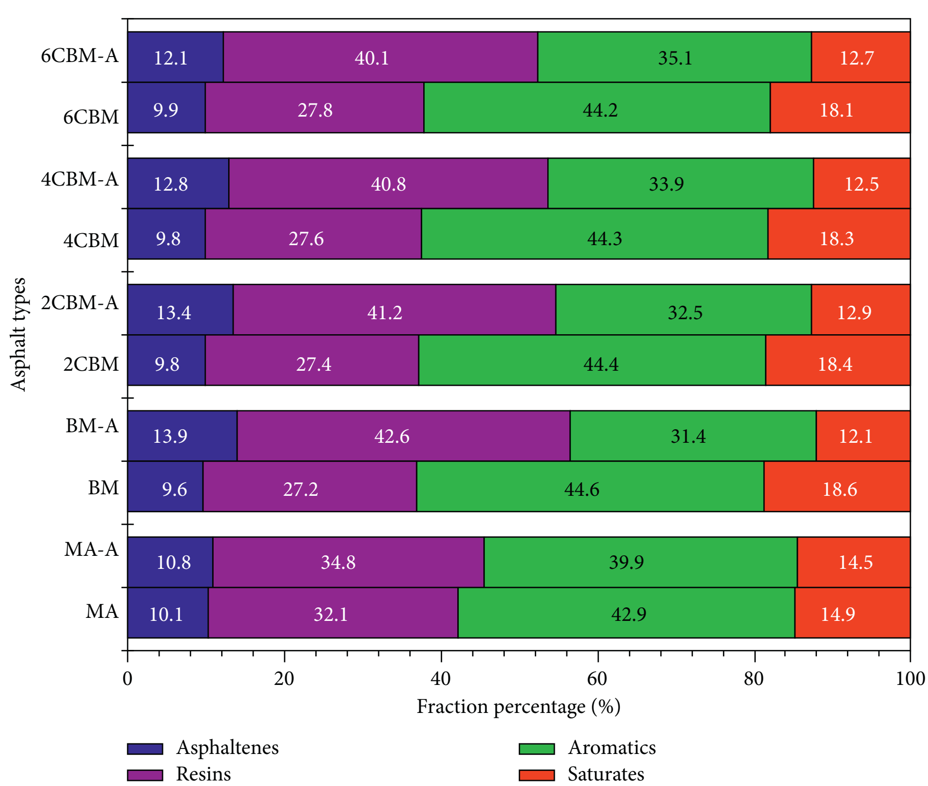


Figure 7: Effect of organic nano-$CaCO_3$ on components of bioasphalt before and after aging.

sulfoxide index, that on the carbonyl index is more significant, which may be related to the components of asphalt and the material structure of modifier [8, 18]. In the range of test contents, the effect of improvement is more obvious with the increase of content. Compared with those of the matrix asphalt, the fractions of saturates and aromatics in bioasphalt are higher. During the aging process, the saturates and aromatics are easy to volatilize and lose, and they are easy to oxidize and change into resins and asphaltenes. On one hand, the super large specific surface area of organic

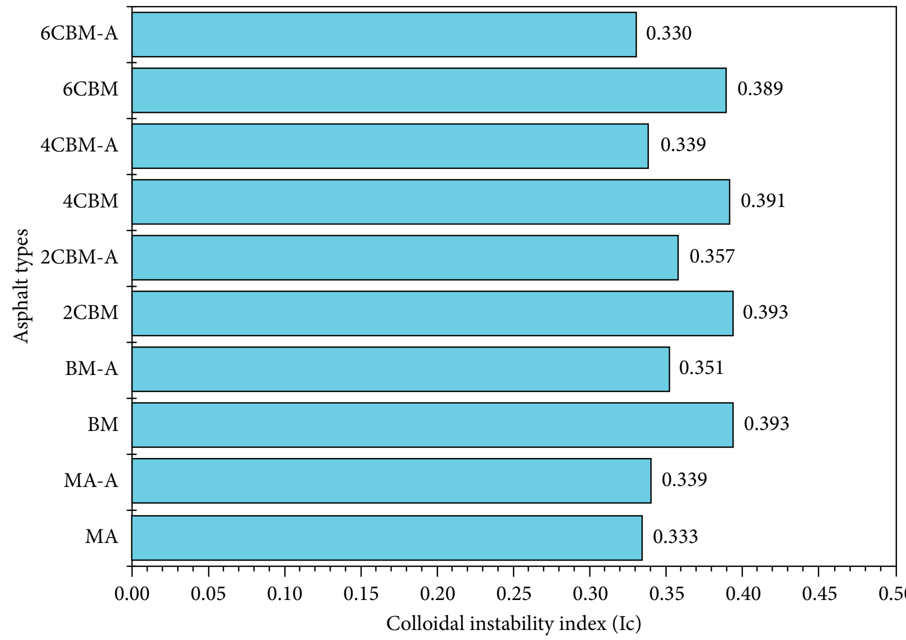


Figure 8: Effect of organic nano-$CaCO_3$ on colloidal instability index of bioasphalt before and after aging.

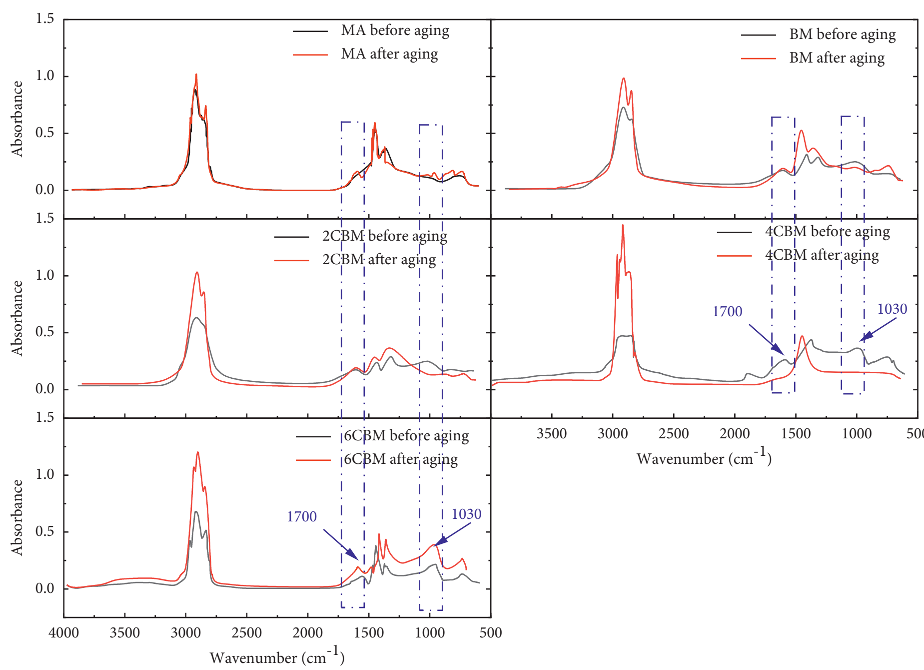


Figure 9: Infrared spectra of different asphalts.

nano calcium carbonate because of its nanoscale largely adsorbs the light components in bioasphalt and slows down the volatilization loss of light fractions. On the other hand, the multilayer structure of organic nano calcium carbonate helps to reduce the permeation rate of oxygen and slow down the oxidation process of asphalt, resulting in a reduced formation rate of carbonyl and sulfoxide oxygen-containing functional groups.

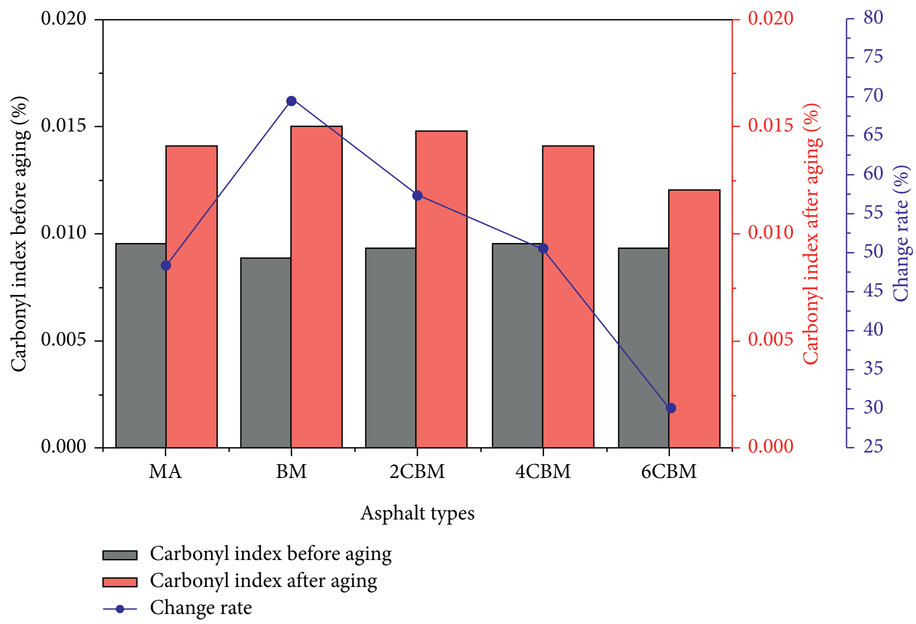


FIGURE 10: Effect of organic nano-$CaCO_3$ on carbonyl index (CI) of bioasphalt before and after aging.

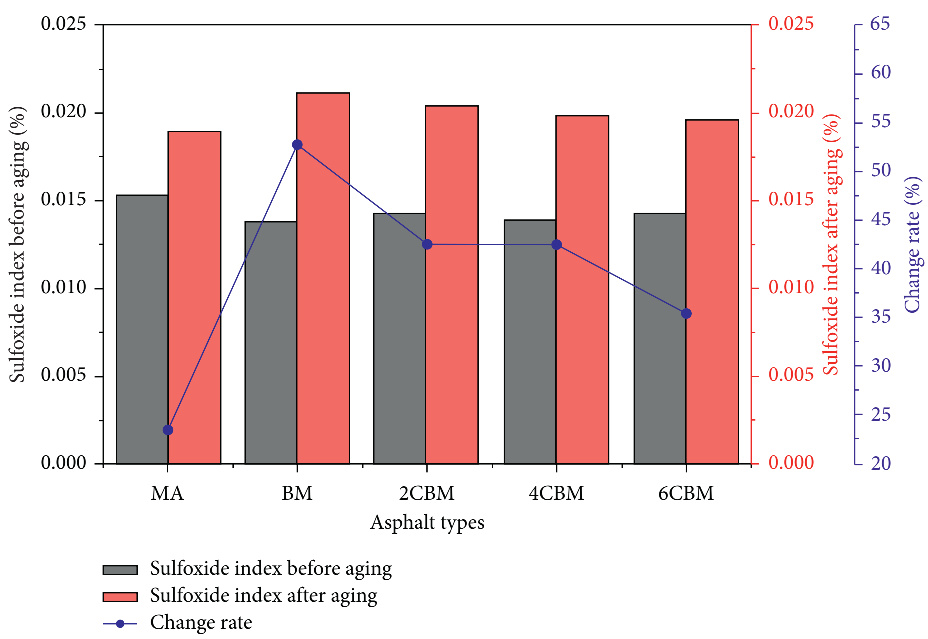


FIGURE 11: Effect of organic nano-$CaCO_3$ on sulfoxide index (SI) of bioasphalt before and after aging.

4. Conclusions

In order to explore the effects of organic nano-$CaCO_3$ on the aging resistance of bioasphalt, the TFOT aging test, basic property tests, rheological tests, four-components tests, and infrared spectrum tests were used to study the bioasphalt with different organic nano-$CaCO_3$ contents. According to the test results and analysis, the following conclusions can be drawn:

(1) Compared with the matrix asphalt, the residual penetration, residual ductility, and residual softening point of bioasphalt after aging are greatly reduced, and the addition of organic nano calcium carbonate makes the three indexes of bioasphalt increase, which is close to the performance level of the matrix asphalt.

(2) The complex modulus aging index GAI of the bioasphalt gradually decreases with the increase of the content of organic nano calcium carbonate. The addition of organic nano calcium carbonate helps to improve the aging resistance of bioasphalt. When the content is 6%, the GAI of organic nano calcium

carbonate modified bioasphalt is less than that of the matrix asphalt.

(3) Compared with MA, the I_c of the BM increased from 0.333 to 0.393. After adding organic nano calcium carbonate, the I_c of 2CBM-A, 4CBM-A, and 6CBM-A gradually decreased. Adding organic nano calcium carbonate helps to improve the colloidal stability of bioasphalt after short-term aging.

(4) Organic nano-$CaCO_3$ can improve the aging resistance of bioasphalt. Adding organic nano-$CaCO_3$ can reduce the change rate of the carbonyl index and sulfoxide index, and the greater the dosage, the more remarkable the effect is.

Data Availability

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

Conflicts of Interest

The authors declare that there are no conflicts of interest with this study.

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