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Investigation of the aging behaviors of multi-dimensional nanomaterials modified different bitumens by Fourier transform infrared spectroscopy

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HIGHLIGHTS

- Influence of multi-dimensional nanomaterials on the aging behaviors of different bitumens is evaluated using FTIR.
- Multi-dimensional nanomaterials can effectively inhibit the carbonyl formation in bitumens during aging.
- The improvements of multi-dimensional nanomaterials on aging resistance depend on the nature of the base bitumen.

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ABSTRACT

Multi-dimensional nanomaterials consisting of nano-zinc oxide (nano-ZnO) and organic expanded vermiculite (OEVMT) were utilized to improve anti-aging performance of different types of bitumen. The chemical functional groups of different bitumens with or without multi-dimensional nanomaterials were characterized by Fourier transform infrared spectroscopy (FTIR). The influence of multi-dimensional nanomaterials on aging performances of different bitumens was evaluated using FTIR, viscosity and dynamic shear rheological tests. All binders were aged by thin film oven test (TFOT), pressure aging vessel (PAV) and ultraviolet radiation (UV) aging. Results show that there are some differences in chemical functional group among 60/80, 80/100 and 100/120 penetration grade bitumens according to FTIR analysis. Additionally, with the interfusion of multi-dimensional nanomaterials, the deterioration in physical and rheological properties and the carbonyl formation in bitumen are retarded effectively, whether during thermal-oxidative aging or during photo-oxidative aging, indicating the good aging resistance of modified bitumens. However, for the rank-ordering of improvement magnitudes in aging resistance of different bitumens, FTIR and viscosity test show different evaluation results. The improvements of multi-dimensional nanomaterials on aging resistance of the binders depend on the nature of the base bitumen.

1. Introduction

Aging is one of the main reasons causing worsening of asphalt pavement performance [1–3]. It is a quite complex process due to the multiple inducing factors, the complexity of bitumen constituents and the diversity of types of bitumen. In consideration of different inducing factors, bitumen aging mainly includes thermal-oxidative and photo-oxidative two aspects, thus the methods enhancing aging resistance of bitumen must consider the two aspects simultaneously [4–8]. In addition, since the complexity of bitumen constituents and diversity of types of bitumen, some anti-aging modifiers (e.g. antioxidant and ultraviolet absorber) have specific selectivity for bitumens from different origins, so it is of significant importance to seek suitable anti-aging modifiers for adapting to the vast majority of bitumens [9–11].

Recently, some inorganic nanomaterials such as layered silicates and inorganic nanoparticles have been introduced into bitumen for enhancing its aging resistance. Layered silicates (e.g. expanded vermiculite) added in bitumen can form intercalated/exfoliated microstructure, which can increase the average path length of the oxygen molecules infiltration and the light components volatilization in the process of thermal-oxidative aging effectively. Thus, the anti-aging performance of bitumen can be enhanced significantly [12]. In addition, the damage to bitumen molecules undergoing ultraviolet (UV) radiation can be weakened effectively due to the absorption and reflection of inorganic nanoparticles (e.g. nano-silica, nano-zinc oxide) on UV radiation [13]. However, some

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published researches demonstrated that layered silicates merely showed apparent improvement for thermal-oxidative aging resistance of bitumen, while inorganic nanoparticles only had clear enhancement for photo-oxidative aging resistance of bitumen [14–16]. Therefore, in this paper, multi-dimensional nanomaterials made up of nano-zinc oxide (nano-ZnO) of zero dimensional nanomaterials and organic expanded vermiculite (OEVMT) of two dimensional nanomaterials was applied to bitumen, which is expected to enhance both thermal-oxidative and photo-oxidative aging resistance of different types of bitumen.

Fourier transform infrared spectroscopy (FTIR) is a modern testing technique, which has been extensively used to characterize chemical functional groups of bitumen and precisely determine variable quantity of some chemical groups caused by the addition of new modifiers or aging [17–21].

The main aim of this research was to study the influence of multi-dimensional nanomaterials on thermal-oxidative and photo-oxidative aging behaviors of different types of bitumen. The FTIR test as primary measure was utilized to characterize the oxidation degree of different bitumens with or without multi-dimensional nanomaterials undergoing aging, as well as analyze the chemical functional groups of different bitumens with or without multi-dimensional nanomaterials before aging. The viscosity and dynamic shear rheological tests were also used to evaluate the physical and rheological properties of different bitumens with or without multi-dimensional nanomaterials after aging. The short-term thermal-oxidative, long-term thermal-oxidative and photo-oxidative aging of all binders were simulated by thin film oven test (TFOT), pressure aging vessel (PAV) and UV aging test, respectively.

2. Materials and methods

2.1. Materials

Three types of base bitumen (named as 70#, 90# and 110#) from different origins were adopted in this research. 70#, 90# and 110# bitumens were supplied by elfa Asphalt Co., Ltd., Panjin Northern Asphalt Co., Ltd., and Baoli Asphalt Co., Ltd., China, respectively. Besides, 70#, 90# and 110# bitumens are 60/80, 80/100 and 100/120 penetration grade bitumen, separately. The basic properties of these base bitumens are presented in Table 1.

Organic expanded vermiculite (OEVMT) was obtained by adopting expanded vermiculite (EVMT) (supplied by Shijiazhuang kinley mining Co., LTD, China) and cetyltrimethyl ammonium bromide (CTAB) (supplied by Shanghai Zhanyun Chemical Co., Ltd., China) as raw materials, and the preparation method referred to the reference [14]. The average particle size of surface modified nano-zinc oxide (nano-ZnO) (supplied by Zhoushan tomorrow nano materials Co., LTD, China) with c-(2,3-epoxypropoxy) propyltrimethoxysilane was 20 nm.

2.2. Preparation of multi-dimensional nanomaterials modified bitumens

Multi-dimensional nanomaterials modified bitumen was prepared as the following procedure: Bitumen was firstly heated to 150 ± 5 °C in an oil-bath heating container until it flowed fully. Then multi-dimensional nanomaterials containing 1%nano-ZnO + 3%OEVMT by weight of bitumen was interfused into bitumen. The mixture was sheared at 4000 r/min for 1 h using a high-speed shear apparatus at first. Subsequently, it was stirred at 2000 rpm for 1.5 h using a paddle agitator. The blank sample was obtained through the same process.

Table 1
Basic properties of three types of base bitumens.

Basic properties	70#	90#	110#
Penetration (25 °C, dmm)	71.2	85.5	105.3
Softening point (°C)	45.3	42.3	41.3
Ductility (15 °C, cm)	>150	>150	>150
Viscosity (135 °C, mPa·s)	590	456	327

2.3. Aging procedures

The simulation methods of the short-term and long-term thermal-oxidative aging of all binders adopted TFOT (ASTM D1754) and PAV (ASTM D6521), separately. The simulation method of the photo-oxidative aging used UV aging. The procedure of UV aging was as follows: The sample after TFOT aging was placed in a UV radiation draft oven for 6 days, the working temperature was 60 ± 5 °C, and the average radiation intensity on the bitumen surface was about 12 w/m^2. UV high pressure mercury lamp of 500 W (produced by Shanghai jiguang special lighting electrical appliance factory, China) as UV source was used in this research, and the main radiation wavelength is 365 nm.

2.4. Rotational viscosity test

The rotational viscosity test (135 °C) was performed by using Brookfield viscometer according to ASTM D4402.

2.5. Dynamic shear rheological test

Dynamic shear rheological test was performed by using dynamic shear rheometer according to ASTM D7175. The temperature sweep mode was adopted in the research, and the sweeping temperature ranged from 40 to 80 °C. The strain and frequency were controlled at 1% and 10 rad/s separately. The rheological parameters such as complex modulus G^* and phase angle δ were utilized to evaluate the rheological property of all samples.

2.6. Fourier transform infrared spectroscopy (FTIR) characterization

Preparation of FTIR sample: firstly, bitumen was dissolved into carbon disulfide (CS_2) and the mass ratio of bitumen to CS_2 was 0.05:1. 1 μL micro injector was used to drop 2 μL solution on KBr tablet through extracting the solution 2 times. Then infrared lamp was used to get rid of the CS_2 solvent. Finally, the sample could be tested in infrared spectrum experiment. The transmission mode of operation FTIR was used in this research. Test range: 4000–400 cm^{-1}; Number of scanning: 32; Resolution: 4 cm^{-1}.

3. Results and discussion

3.1. FTIR analysis of multi-dimensional nanomaterials modified bitumens before aging

The FTIR test results of nanomaterials and 70#, 90# and 110# bitumens before and after multi-dimensional nanomaterials modification are shown in Fig. 1(a)–(d) respectively, and the corresponding functional groups are listed detailedly in Table 2. As presented in Table 2, in contrast to 70# bitumen, 90# bitumen has two new absorption peaks at 2349 cm^{-1} and 727 cm^{-1} while lacks the absorption peak at 748 cm^{-1}. Given that the absorption peaks at 2349 cm^{-1}, 748 cm^{-1} and 727 cm^{-1} are caused by bending vibration of N—H [22], bending vibration from aromatic branched chain and synergy vibration of —CH_2—, separately. It can be determined that 90# bitumen has more the bending vibration from N—H and the synergy vibration from —CH_2—, whereas is

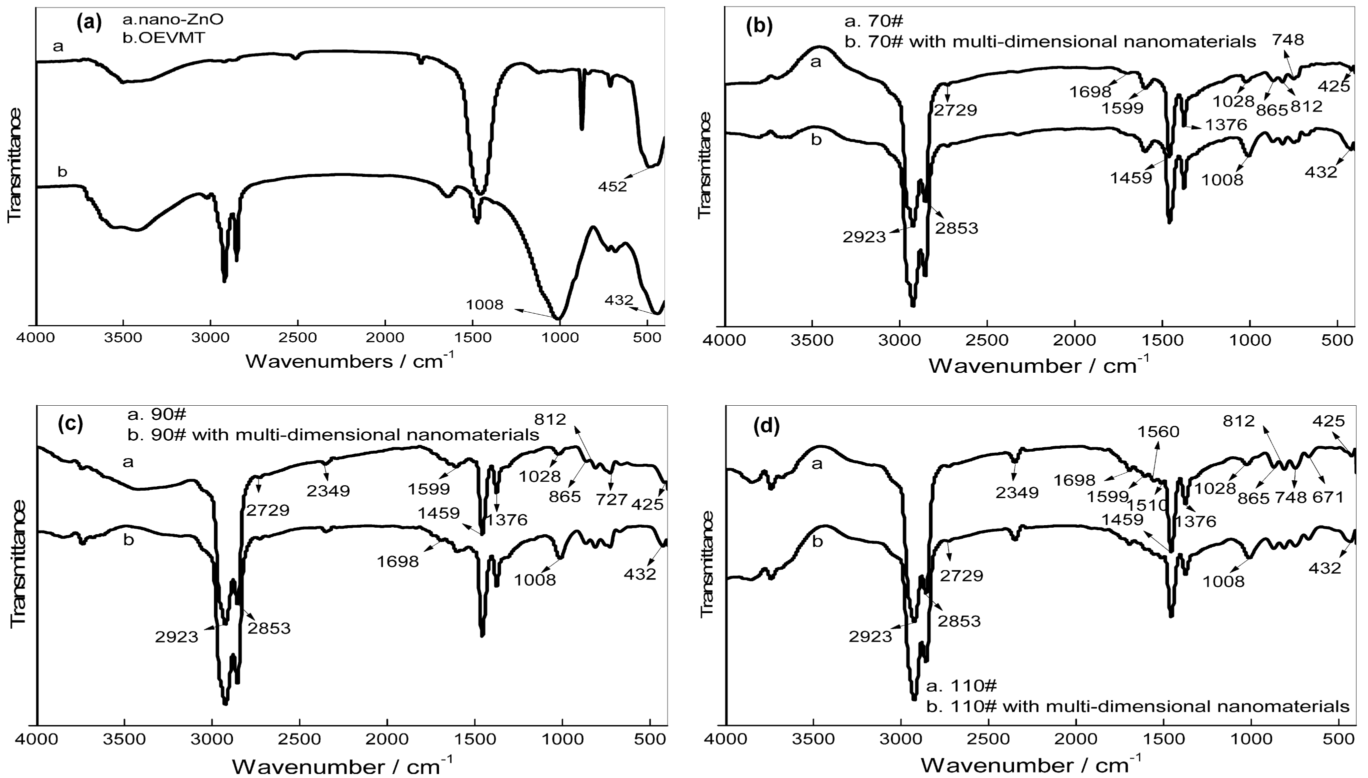

Fig. 1. FTIR of nanomaterials and three types of bitumen before and after multi-dimensional nanomaterials modification: (a) nanomaterials, (b) 70#, (c) 90# and (d) 110#.

Table 2
Functional groups of three types of bitumen before and after multi-dimensional nanomaterials modification.

Absorption wavenumber (cm^{-1})	Functional groups	70#	70# with multi-dimensional nanomaterials	90#	90# with multi-dimensional nanomaterials	110#	110# with multi-dimensional nanomaterials
2923	—CH_2— asymmetric and symmetric stretching vibration	√	√	√	√	√	√
2853		√	√	√	√	√	√
2729	—CHO stretching vibration	√	√	√	√	√	√
2349	N—H bending vibration [22]	—	—	√	√	√	√
1698	C═O stretching vibration	√	√	√	√	√	√
1599	Phenyl alkanes skeleton conjugated double bonds (C═C) stretching vibration	√	√	√	√	√	√
1560	N—H stretching and bending vibration	—	—	—	—	√	√
1510	[23]	—	—	—	—	√	√
671		—	—	—	—	√	√
1459	—CH_2— bending vibration	√	√	√	√	√	√
1376	—CH_3 umbrella vibration	√	√	√	√	√	√
1028	S═O stretching vibration	√	—	√	—	√	—
1008	Si—O—Si in OEVMT stretching vibration	—	√	—	√	—	√
865	—CH— on benzene ring out-of-plane bending vibration	√	√	√	√	√	√
812		√	√	√	√	√	√
748	Aromatic branched chain bending vibration	√	√	—	—	√	√
727	—CH_2— synergy vibration	—	—	√	√	—	—
432	Si—O—Si in OEVMT bending vibration	—	√	—	√	—	√

short of the bending vibration from aromatic branched chain in comparison with 70# bitumen. The result indicates that there are some differences in chemical functional groups between 70# and 90# bitumen. Additionally, 110# bitumen shows some new absorption peaks at 2349 cm^{-1}, 1560 cm^{-1}, 1510 cm^{-1} and 671 cm^{-1} compared with 70# bitumen. Meanwhile, 110# bitumen has these new absorption peaks at 1560 cm^{-1}, 1510 cm^{-1}, 748 cm^{-1} and 671 cm^{-1} while lacks the absorption peak at 727 cm^{-1} compared with 90# bitumen. Due to the fact that the absorption peaks at 1560 cm^{-1}, 1510 cm^{-1} and 671 cm^{-1} originate from the stretching and bending vibration of N—H and these N—H exist in different kinds of amino compound [23], so it also can be concluded that there are some differences in chemical functional groups between 110# and 70# or 90# bitumen.

Besides, for three types of bitumen, by comparison with unmodified bitumen, two new absorption peaks in multi-dimensional nanomaterials modified bitumen at 1008 cm^{-1} and 432 cm^{-1} appear, which are attributed to the stretching and bending vibration of Si—O—Si in OEVMT (as shown in Fig. 1(a)), separately. But the absorption peak at 452 cm^{-1} caused by bending vibration of nano-ZnO (as shown in Fig. 1(a)) overlaps with the absorption peak provided by bending vibration of Si—O—Si, thus

Table 3
Rotational viscosity (135 °C) of bitumens with or without multi-dimensional nanomaterials before and after different aging methods.

Bitumen samples	70#	70# with multi-dimensional nanomaterials	90#	90# with multi-dimensional nanomaterials	110#	110# with multi-dimensional nanomaterials
Unaged	630.0	644.0	486.3	523.8	408.8	477.5
TFOT aging	861.2	817.9	697.8	625.9	796.3	786.4
PAV aging	2184.2	1447.7	1174.4	978.5	2584.4	2484.9
UV aging	1177.5	1046.5	1092.2	1125.6	1134.8	1293.1

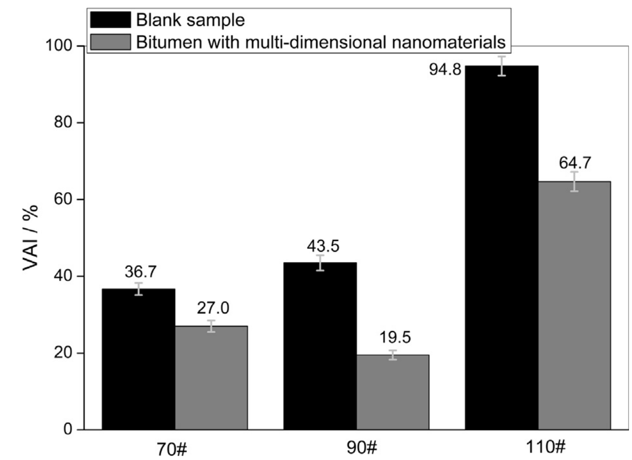

Fig. 2. VAI of bitumens with or without multi-dimensional nanomaterials after TFOT aging.

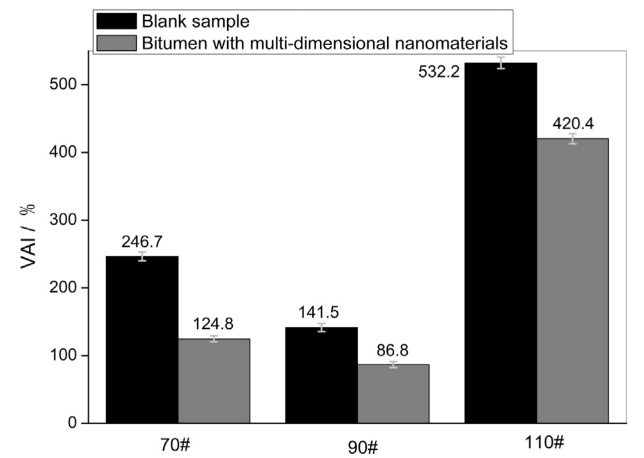

Fig. 4. VAI of bitumens with or without multi-dimensional nanomaterials after PAV aging.

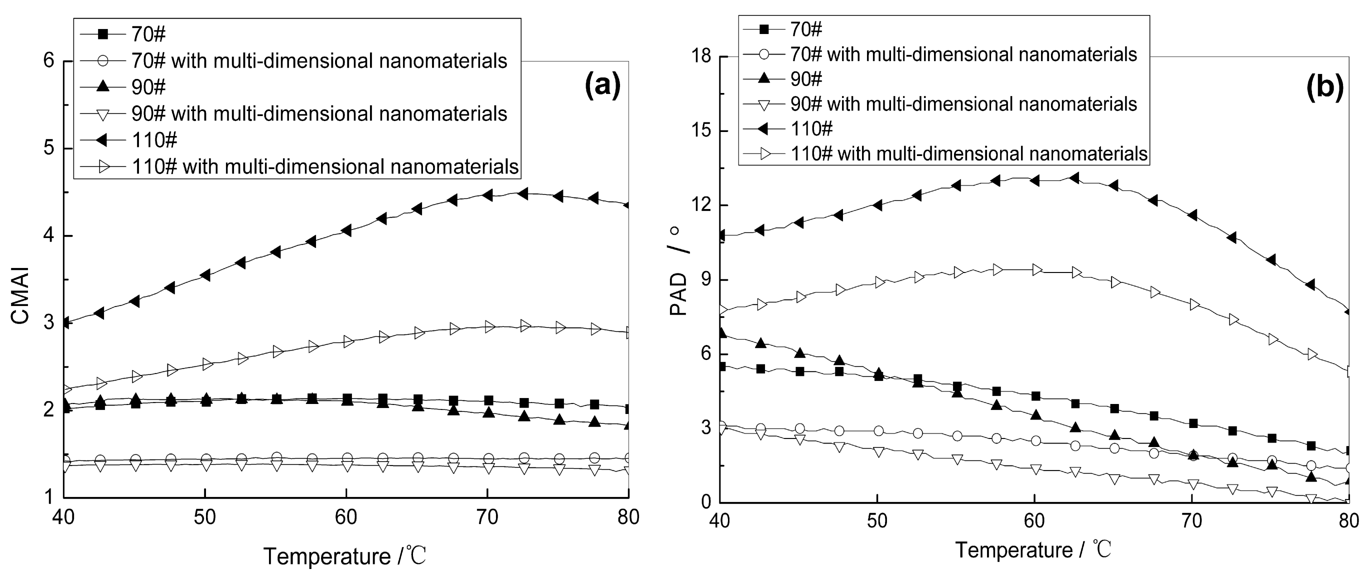

Fig. 3. CMAI and PAD of bitumens with or without multi-dimensional nanomaterials after TFOT aging.

Table 4
Carbonyl indices ($I_{C=O}$) of bitumens with or without multi-dimensional nanomaterials before and after TFOT aging.

Samples	Unaged	TFOT aging	Increment	Increment standard deviation
70#	0.037	0.073	0.036	0.003
70# with multi-dimensional nanomaterials	0.032	0.051	0.019	0.002
90#	0.065	0.100	0.035	0.003
90# with multi-dimensional nanomaterials	0.124	0.154	0.030	0.002
110#	0.224	0.321	0.097	0.004
110# with multi-dimensional nanomaterials	0.231	0.306	0.075	0.003

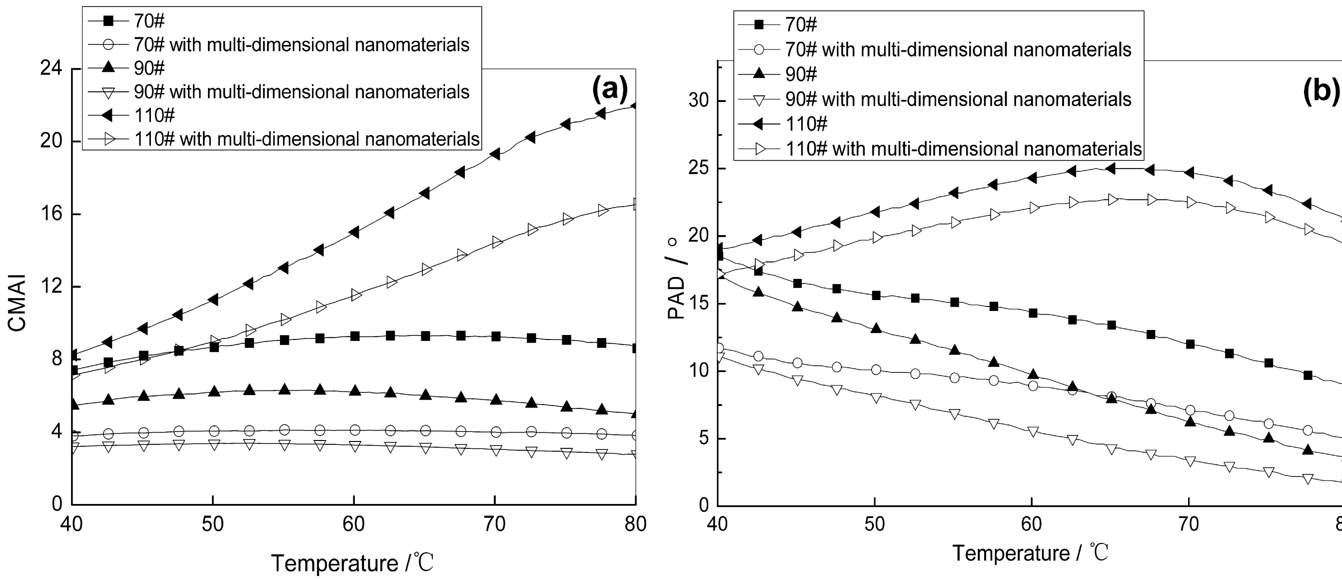

Fig. 5. CMAI and PAD of bitumens with or without multi-dimensional nanomaterials after PAV aging.

Table 5
Carbonyl indices ($I_{C=O}$) of bitumens with or without multi-dimensional nanomaterials before and after PAV aging.

Samples	Unaged	PAV aging	Increment	Increment standard deviation
70#	0.037	0.172	0.135	0.008
70# with multi-dimensional nanomaterials	0.032	0.135	0.103	0.007
90#	0.065	0.284	0.219	0.013
90# with multi-dimensional nanomaterials	0.124	0.270	0.146	0.008
110#	0.224	0.571	0.347	0.012
110# with multi-dimensional nanomaterials	0.231	0.536	0.305	0.010

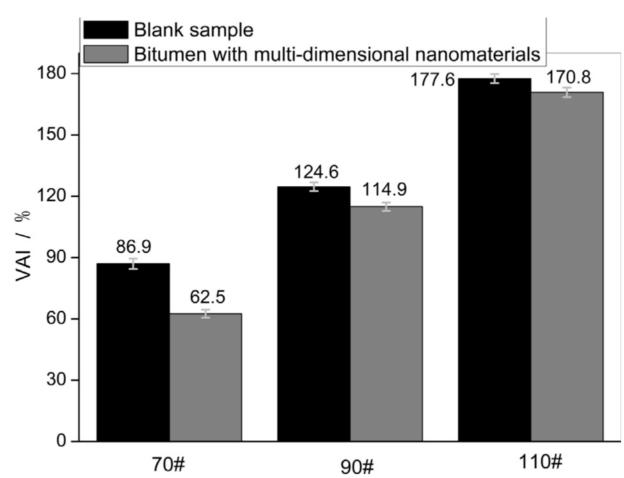

Fig. 6. VAI of bitumens with or without multi-dimensional nanomaterials after UV aging.

the two absorption peaks are difficult to distinguish. As a result, it is determined that large amount of OEVMT exists in FTIR sample, which in turn can be speculated that OEVMT has a good compatibility with three types of bitumen. However, the existence of nano-ZnO in FTIR sample can't be determined.

In summary, there are some differences in chemical functional groups among 70#, 90# and 110# bitumens. Moreover, it can be speculated that OEVMT has a good compatibility with three types of bitumen.

3.2. Analysis of multi-dimensional nanomaterials modified bitumens after TFOT aging

Generally, aging can increase viscosity of base bitumens, moreover, the more severe aging, the more obvious increase of viscosity. The viscosity data of all bitumen binders before and after different aging is listed in Table 3. Herein viscosity aging index (VAI) as an aging indicator is utilized to evaluate the influence of aging on physical property of bitumen [9]. The VAI is obtained according to the formula (1).

$$\mathrm{VAI}(\%) = (\text{Aged viscosity} - \text{Unaged viscosity}) / \text{Unaged viscosity} \times 100, \quad (1)$$

It is observed from Fig. 2 that, after TFOT aging, the VAI values of three types of bitumen decrease conspicuously with introducing multi-dimensional nanomaterials, indicating that the deterioration of physical property of three bitumens after short-term thermal oxidation is reduced effectively. Besides, the reduction values for 70#, 90# and 110# bitumens are 9.7%, 24.0% and 30.1%, respectively. It suggests that multi-dimensional nanomaterials shows the most apparent improvement for physical property of 110# bitumen after TFOT aging.

It is well known that aging can enhance complex modulus while reduce phase angle of base bitumens. In this research, complex modulus aging index (CMAI) and phase angle difference (PAD) are utilized to analyze the influence of aging on dynamic shear rheological property of all samples [24]. The CMAI and PAD are obtained according to the formula (2) and (3), respectively.

$$\mathrm{CMAI} = \text{Aged complex modulus} / \text{Unaged complex modulus}, \quad (2)$$

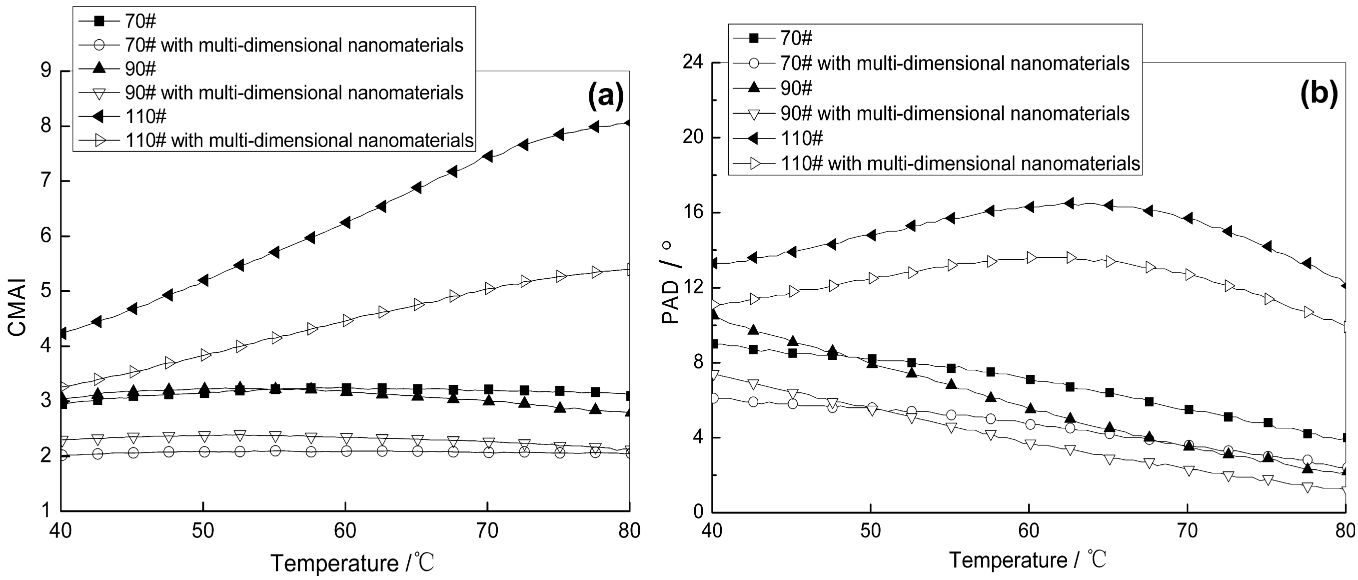

Fig. 7. CMAI and PAD of bitumens with or without multi-dimensional nanomaterials after UV aging.

Table 6
Carbonyl indices ($I_{C=O}$) of bitumens with or without multi-dimensional nanomaterials before and after UV aging.

Samples	Unaged	UV aging	Increment	Increment standard deviation
70#	0.037	0.080	0.043	0.003
70# with multi-dimensional nanomaterials	0.032	0.071	0.039	0.003
90#	0.065	0.189	0.125	0.006
90# with multi-dimensional nanomaterials	0.124	0.207	0.083	0.005
110#	0.224	0.431	0.207	0.007
110# with multi-dimensional nanomaterials	0.231	0.411	0.180	0.005

$$\text{PAD} = \text{Unaged phase angle} - \text{Aged phase angle}, \quad (3)$$

Fig. 3 shows the CMAI and PAD of bitumens with or without multi-dimensional nanomaterials after TFOT aging. It is obviously found that, after adding multi-dimensional nanomaterials, the CMAI and PAD of three types of bitumens are decreased. The result manifests that the multi-dimensional nanomaterials can slow down the influence of short-term thermal-oxidative aging on dynamic shear rheological property of these bitumens evidently.

The aging of bitumen is an oxidation process, which can cause some oxygen-containing functional groups (e.g. carbonyl) in bitumen increase. In this research, carbonyl index ($I_{C=O}$) was utilized to assess oxidation degree of bitumen undergoing aging, as presented in formula (4) [25].

$$I_{C=O} = \frac{\text{Area of carbonyl centered around } 1698\text{cm}^{-1}}{\text{Area of } CH_3 \text{ umbrella vibration centered around } 1376\text{cm}^{-1}}, \quad (4)$$

The $I_{C=O}$ of all binders after TFOT aging are listed in Table 4. As seen in Table 4, after TFOT aging, it is quite obvious that the $I_{C=O}$ of all binders increase at different degrees. However, the increased amplitude of bitumens with multi-dimensional nanomaterials is lower than that of corresponding blank samples, illustrating that the introduction of multi-dimensional nanomaterials can prevent carbonyl formation in bitumens undergoing TFOT aging. The main reason is that the formed exfoliated microstructure between bitumen and OEVMT can increase the average path length of the oxygen molecules infiltration in the process of thermal-oxidative aging [26]. Moreover, through making a comparison with three types of bitumen, it can be found that the ranking of decrement values of the $I_{C=O}$ increment of modified bitumen relative to that of unmodified bitumen is 110# > 70# > 90#, namely 0.022, 0.017 and 0.005, respectively. This result manifests that multi-dimensional nanomaterials have the best effect in inhibiting carbonyl formation for 110# bitumen, followed by 70# bitumen and finally 90# bitumen, which is different from the viscosity result.

3.3. Analysis of multi-dimensional nanomaterials modified bitumens after PAV aging

As seen in Fig. 4, after PAV aging, multi-dimensional nanomaterials modified bitumens show the lower VAI value in comparison with corresponding blank samples, as well as the lowered magnitudes for 70#, 90# and 110# bitumens are 121.9%, 54.7% and 111.8%, separately. As a result, the multi-dimensional nanomaterials slows down remarkably the worsening of physical property of bitumens undergoing long-term thermal-oxidative aging, moreover, the effect for 70# bitumen is the most evident.

Additionally, the CMAI and PAD of all binders after PAV aging are shown in Fig. 5. Compared with corresponding blank sample, after introducing multi-dimensional nanomaterials, the CMAI and PAD of bitumens is reduced obviously. It can be concluded that introduction of the multi-dimensional nanomaterials can retard the influence of long-term thermal-oxidative aging on the dynamic shear rheological property of three types of bitumens effectively.

The $I_{C=O}$ of bitumens with or without multi-dimensional nanomaterials after PAV aging are listed in Table 5. After PAV aging, the $I_{C=O}$ values of three bitumens further increase in accordance with Table 5. Nonetheless, the $I_{C=O}$ values of three types of bitumen are decreased apparently by adding multi-dimensional nanomaterials. Furthermore, the decrement ranking of three bitumens is 90# > 110# > 70#, that is 0.073, 0.042 and 0.032, respectively. The result indicates that the addition of multi-dimensional nanomaterials also can prevent effectively carbonyl formation in bitumens undergoing long-term thermal-oxidative aging. Moreover, the effect for 90# bitumen is the most conspicuous, which is also different from the result from viscosity test.

3.4. Analysis of multi-dimensional nanomaterials modified bitumens after UV aging

Fig. 6 shows the VAI values of bitumens with or without multi-dimensional nanomaterials after UV aging, it can be observed that multi-dimensional nanomaterials modified bitumens have the lower VAI values compared with the corresponding blank samples. Besides, the ranking of VAI dropped value for three bitumens after UV aging is 70# > 90# > 110# and the values are 24.4%, 9.7% and 6.8%, respectively. The result demonstrates that multi-dimensional nanomaterials can improve effectively UV aging resistance of three bitumens in physical property. Moreover, the improvement for 70# bitumen is the most prominent.

The influence of multi-dimensional nanomaterials on the dynamic shear rheological property of three types of bitumens after UV aging is shown in Fig. 7. It is observed that the bitumens with multi-dimensional nanomaterials show the lower CMAI and PAD than the bitumens without multi-dimensional nanomaterials. It means that the influence of UV aging on dynamic shear rheological property is weakened effectively with the addition of multi-dimensional nanomaterials.

Besides, the $I_{C=O}$ of all binders after UV aging are presented detailedly in Table 6. As seen in Table 6, the interfusion of multi-dimensional nanomaterials can decrease effectively the $I_{C=O}$ value of bitumens after UV aging, indicating that the carbonyl formation in bitumens is suppressed evidently. Furthermore, the decrement value ranking of three bitumens is 90# > 110# > 70#, namely 0.042, 0.027, and 0.004, separately. The result also manifests that multi-dimensional nanomaterials can inhibit evidently carbonyl formation of bitumens in the process of photo oxidation aging. Furthermore, the effect for 90# bitumen is the most apparent, which is also different from the viscosity test result.

4. Conclusions

In this paper, the influence of multi-dimensional nanomaterials on different types of bitumen before and after aging was investigated by FTIR, viscosity and dynamic shear rheological tests. The main research conclusions are as follows:

(1) Based on the FTIR analysis for all binders before aging, there are some differences in chemical functional groups among 70#, 90# and 110# bitumens. Meanwhile, it is determined that large amount of OEVMT exists in FTIR sample, which in turn can be speculated that OEVMT has a good compatibility with three types of bitumen.
(2) With the multi-dimensional nanomaterials added into bitumens, the deterioration of physical and rheological properties and the oxidation of chemical functional groups are retarded effectively, either during thermal-oxidative aging or during photo-oxidative aging.
(3) For the rank-ordering of improvement magnitudes in aging resistance of three types of bitumen, FTIR test and viscosity test show different evaluation results.

Acknowledgements

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