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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.

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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

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