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Effect of inorganic ultraviolet resistance nanomaterials on the physical and rheological properties of bitumen

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HIGHLIGHTS

- The influence of LDHs, OMMT and CB on properties of bitumen was studied.
- Inorganic nanomaterials has hardening effect on bitumen.
- The microstructure and content of nanomaterials affect bitumen properties.
- No obvious effect of surface area and PH of raw nanomaterial on bitumen properties.

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ABSTRACT

The influence of the inorganic nano ultraviolet (UV) resistance materials, including organo-montmorillonite (OMMT), layered double hydroxides (LDHs) and carbon black (CB), on the physical and rheological properties of bitumen was investigated by Scanning Electronic Microscopy (SEM), Brun auer–Emmett–Teller (BET), penetration, ductility, softening point and Dynamic Shear Rheometer (DSR) tests. The effects of the factors such as micromorphology, specific surface area, pH value and content of inorganic nano UV resistance materials were explored. In this study, these inorganic nano UV resistance materials were mixed with bitumen by melt-blending, and the content was selected from 0 to 10 wt% which was consistent with the actual contents in bitumen. The results showed that these materials increased the softening point and complex modulus (G^*) of bitumen, and reduced penetration, ductility and phase angle of bitumen. OMMT showed the most obvious hardening effect followed by CB and lastly LDHs. Higher contents of the materials increased the stiffness and elastic of bitumen. Specific surface area and pH of nanomaterials had no significant correlation to performance. The hardening effect of the inorganic nano UV resistance materials modified bitumen was mainly dependent on the microstructure of the inorganic nanomaterials in bitumen and its contact area with bitumen.

1. Introduction

The aging of bitumen results in a harder and more brittle bitumen, which is one of the main reasons for asphalt pavement failures due to low temperature cracking and fatigue cracking [1–3]. It has been shown that Ultraviolet (UV) irradiation is linked to bitumen degradation during the pavement's service, i.e., UV aging [4]. Therefore, extending the service life of asphalt pavement requires improving the UV aging resistance of bitumen. The addition of UV resistance materials is one of the effective ways. Recently, some inorganic nanomaterials such as organo-montmorillonite (OMMT), layered double hydroxides (LDHs) and carbon black (CB), have attracted attention because of their obvious UV aging-resistant properties [5–8].

OMMT represents an organic cationic surfactant modified montmorillonite. OMMT is a typical layered silicate, whose layered structure can function as a shield to UV light. OMMT/bitumen nanocomposite structure (exfoliation) slows down the spreading rate of oxygen in the bitumen, which enhances the resistance ability of bitumen to thermal-oxidative aging and UV radiation aging [7–9]. LDHs have a multi-nestification layered structure, which is formed by interlayer anions and laminates with a positive charge [10]. The inorganic layer sheets functions as a shield to physically prevent the UV light. Metal atoms of layer sheets and negative ions between layer sheets can chemically absorb UV light [11–13]. CB as a typical kind of inorganic nanomaterial pigments, has a high degree of dispersion and covering power, so it can absorb the light

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energy into heat energy which is spread, or reflect harmful light to protect bitumen. Thus CB in bitumen also acts as a shield to UV light [14,15].

The addition of inorganic nanomaterials will affect the microstructure, mechanical and chemical properties of base materials [16–18]. Although OMMT, LDHs and CB have shown a significant resistance to UV aging, they also affect the penetration, ductility, softening point, viscosity of bitumen, and bring the hardening effect on bitumen [19–21]. The hardening effect decreases low temperature and fatigue performance of bitumen [7,22,23]. In recent years, much research attention was focused on the UV resistance of inorganic nano materials in bitumen [24,25], but little on their hardening effect. In this study, OMMT, LDHs and CB modified bitumen were prepared by melt blending. The impact of the inorganic nanomaterials on the physical and rheological properties of bitumen was investigated. Penetration, ductility, softening point, complex modulus were used to estimate the hardening effect. Moreover, the influences of inorganic nanomaterials with different contents, specific surface areas, pH values and microstructures on the hardening effect of bitumen were also studied.

2. Materials and test methods

2.1. Materials

The base bitumen (AH-90) was supplied by SK Corporation, Korea. Its corresponding physical properties are shown in Table 1.

The inorganic nano UV resistance materials used were OMMT, LDHs and CB. OMMT was produced by Fenhong Clay Chemical Factory, China, LDHs by RuiFa Chemical Company Limited, Jiang Su, China, and CB by Degussa Chemical Company Limited, Frankfurt, German. Their physical properties are listed in Table 2.

2.2. Modified bitumen preparation

OMMT, LDHs and CB modified bitumen were prepared by melt blending. The base bitumen was first heated to 140 °C, and then, the inorganic nanomaterial powders were slowly added to the base bitumen and sheared for 1 h at a rotation speed of 4000 rpm by a high-speed shearing mixer to ensure a good distribution of the inorganic nanomaterial powders.

Some research results showed that 5 wt% inorganic nanomaterials (CB, LDHs and OMMT) by weight could significantly improve the anti-aging performance of bitumen [8–10]. Therefore, the content of inorganic nanomaterials was chosen around 5 wt% in this study, and modified bitumen samples were prepared with LDHs, OMMT, and CB contents of 3%, 5%, 7% and 10%.

2.3. Physical performance tests

The physical performance tests: penetration test at 25 °C, ductility at 15 °C and softening point (ring&ball method) were carried out according to the standard ASTM D5, ASTM D113-86 and ASTM D36respectively.

2.4. Dynamic shear rheometer(DSR) test

Dynamic rheological properties of OMMT, LDHs and CB modified bitumen were investigated using a dynamic shear rheometer (Anton Paar Company, MCR101) under a parallel plate configuration. A temperature sweep test from 30 to 80 °C with an increment of 2 °C per minute, was performed under a strain-controlled mode at a constant frequency of 10 rad/s. The strain was maintained at 0.1% so that all testing could lie within the linear viscoelastic range. Moreover, the diameter of the plate was 25 mm, and the gap between the plates was 1 mm.

Table 1
Physical properties of base bitumen AH-90.

Penetration at 25 °C/0.1 mm	Softening point/°C	Ductility at 15 °C/cm
86	44.5	142

Table 2
Physical properties of LDHs, OMMT and CB.

Nanomaterials	Appearance	Bulk density/g/cm^3	Moisture content/%
OMMT	Yellowish powder	2.3	<3
LDHs	White powder	0.45	<0.3
CB	Black powder	0.13	<1.5

2.5. Scanning electronic microscopy (SEM) test

A SEM (JSM-5610LV, JEOL Ltd., Japan) was used for the investigation of the microstructure of the inorganic nanomaterials. SEM produces images of a sample surface by scanning it with a focused beam of electrons. The main specifications for the equipment were: Hv Mode Resolution:3 nm; LV Mode Resulution:4 nm; M agnification:18–300,000X; Accelerating voltage :0.5–30 kV; Low Vacuum Degree :1–27 Pa.

2.6. Brunauer–Emmett–Teller (BET) test

The BET (Quantachrome Instruments, Boynton Beach, Florida, USA) was used for determining the specific surface area of the inorganic nanomaterials. The frequency was 50/60 Hz and the max power 1100 VA. The available temperature range was 77–200 K. The temperature stability was ±0.01K using an ITC controller. The cooling speed was about 200 K/h. The LN2 reservoir was 3.0L. Brunauer–Emmett–Teller (BET) test is based on a theory that aims to explain the physical adsorption of gas molecules on a solid surface. BET serves as the basis for an important analysis technique for the measurement of the specific surface area of a material [26]. The BET theory relates to multi-layer adsorption, and used a non-corrosive gas (nitrogen) as an adsorbate for determining the surface area data.

2.7. pH value test

A pH meter (S400SevenExcellence, mettler toledo, Switzerland) was used for the investigation of pH value of nanomaterials. To measure pH value of nanomaterials, 1 g specimen was took and dissolved in 50 g ethanol-water. Then pH value of solution was tested by pH meter.

3. Results and discussion

3.1. Micromorphology

The SEM observations of OMMT, LDHs and CB were presented in Fig. 1. From it the apparent shape and the crystal structure of inorganic nano materials could be observed. OMMT, whose particle size was about several to dozens of microns, could be seen in Fig. 1(a). The flaky texture and aggregated structure of OMMT were observed, which was more obvious in Fig. 1(b). OMMTs are close packed structure which consist of layers of tetrahedral silicate sheets and octahedral hydroxide sheets [7]. The layered structure of OMMT helped it to block UV light.

Fig. 1(c) and (d) showed the SEM images of LDHs. They revealed that LDHs contained enormous tiny regular particles dispersed in the area (Fig. 1(c)), and the particles had different grain sizes (Fig. 1(d)). LDHs consisted of metal hydroxide lamellar structure and interlayer guest anion, and were mainly combined by electrostatic forces and little by hydrogen bond and Van der Wals forces [27,28]. Fig. 1(e) and (f) showed the SEM images of CB. They showed that CB were the spheres agglomerated by enormous tiny regular particles (Fig. 1(e)), and the particles were also different grain sizes (Fig. 1(f)). CB formed incompact structure by physisorptions, electrostatic force and Van der Wals force [29].

Comparing the 5000X SEM images of LDHs and CB, it was noted that LDHs contained enormous tiny regular particles (Fig. 1(c)), but which were more dispersed compared to CB particles (Fig. 1(e)). This phenomenon may indicate that CB was more difficult to disperse in bitumen than LDHs. Comparing the 50000X SEM images of LDHs and CB, it was noticed that CB and LDHs had a similar morphology microstructure. They all contained numerous agglomer-