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Preparation of dodecyltrimethoxysilane surface organic LDHs and application in aging resistance of SBS modified bitumen

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Abstract

Dodecyltrimethoxysilane surface organic layered double hydroxides (LDHs) was synthesized and used to improve the aging resistance of SBS modified bitumen (SMB). According to Fourier transform infrared spectroscopy, x-ray diffraction and Contact angle meter, dodecyltrimethoxysilane has been chemically grafted onto the surface of LDHs and will not affect the crystal structure of LDHs. After dodecyltrimethoxysilane surface organic modification, the -OH on the surface of LDHs was decreased, meanwhile, some organic groups were introduced into LDHs, which contributed to reducing the surface hydrophilicity of LDHs. Then, the physical, rheological properties and chemical structure of SMB containing OLDHs and LDHs before and after aging were studied thoroughly. Compared with LDHs, the compatibility of OLDHs in SMB has been significantly enhanced. Furthermore, OLDHs modified SMB exhibited more excellent high-temperature behavior. After aging, bitumen was oxidized and SBS was degraded, the physical and rheological properties of SMB were seriously deteriorated. The addition of LDHs can suppress the destruction of aging on the performance and chemical structure of SMB, and enhance the aging resistance of SMB. The enhancement of LDHs has been notably strengthened after dodecyltrimethoxysilane surface organic modification.

1. Introduction

SBS modified bitumen (SMB) is applied as binder in the highway construction on a large scale, which is attributed to the excellent viscoelasticity [1, 2]. However, SMB is susceptible to aging under the impact of the external environment (heat, oxygen, ultraviolet). The aging will cause the properties deterioration of SMB, and eventually result in the premature failure of bituminous pavement [3–6]. Hence, it is significant to ameliorate the aging resistance of SMB. Some antiaging agents have been added into SMB, for instance antioxidant, ultraviolet absorbent, layered silicate, and inorganic nanoparticle, etc [7–9]. These antiaging agents can all improve the aging resistance of SMB in some extent, but have limitations, such as, the function of antioxidants is single and only improved the thermal oxygen aging resistance of SMB, the improvement effectiveness of ultraviolet absorbents on the aging resistance is connected with the kind of SMB, the compatibility and dispersibility of layered silicates and inorganic nanoparticles are poor in SMB [10–12]. The existing problems more or less restrain the modification effect or application of these antiaging agents in SMB.
Layered double hydroxides (LDHs) are a kind of ionic layered clay compounds, which are consist of the laminate of metallic oxide and interlamellar anions, the metal laminates can physically reflect ultraviolet light and obstruct the infiltration of oxygen and heat. Meanwhile, interlamellar anions can also chemically assimilate ultraviolet light in some degree \([13–15]\). Hence, LDHs will be a preferable option to enhance the aging resistance of SMB. Recently, some researchers have already applied LDHs in the modification of SMB, Xu et al.\(^\text{[16]}\) found that LDHs can inhibit the chemical structure change and alleviate the performance deterioration of SMB during the ultraviolet aging. Zhao et al.\(^\text{[17]}\) showed that LDHs can suppress the damage of ultraviolet on rheological property of SMB. However, it is noteworthy that LDHs can not fully bring into play the enhancement for the aging resistance of SMB, because of the immense –OH on the surface of LDHs, which resulted in the inhomogeneous dispersion of LDHs in SMB.

To make full use of the improvement of LDHs on the aging resistance of SMB, in the research, dodecyltrimethoxysilane has been applied to surface organically modify LDHs, and the structures and surface property of organic LDHs were analyzed by Fourier transform infrared spectroscopy (FTIR), x-ray diffraction (XRD) and Contact angle meter (CAM). The impact of organic LDHs on the compatibility, physical and rheological properties, aging resistance and chemical structure of SMB were investigated.

### 2. Materials and experimental methods

#### 2.1. Materials

SMB was provided from Xiamen Sunlit Co. Ltd, China, the physical property parameters of SMB are listed in table 1. LDHs (\([\text{Mg}_{2+3}\text{Al}_{3+}^{2+}\text{OH}_{1+4}\text{(CO}_3^{2−}\text{)}_{\text{x/2}}\text{H}_2\text{O}\]) was offered from Nantong Advance Chemicals Co., Ltd, China. Dodecyltrimethoxysilane was supplied from Sinopharm Chemical Reagent Co., Ltd, China.

#### 2.2. Preparation of surface organic modified LDHs

Firstly, LDHs powder and dodecyltrimethoxysilane (3.0% by weight of LDH) were successively dissolved and dispersed in ethanol-water mixture (90%, volumetric fraction), and then adding acetic acid into the mixture to maintain the PH at 4. In order to ensure the sufficient reaction between LDHs and dodecyltrimethoxysilane, the solution was under the nitrogen protection and rapid mixing for 3 h. Then, LDHs modified by dodecyltrimethoxysilane (OLDHs) was repeated washing by vacuum filtration. Finally, OLDHs was dried in a vacuum drying chamber and ground into powders (a diameter of 0.075 mm). To maintain the consistency of the experiment, the particle size of unmodified LDHs was also 0.075 mm.

#### 2.3. Preparation of modified bitumen

SMB was heated to flow regime at 170 °C, and different addition amounts (1%~4% weight of bitumen) of OLDHs and LDHs were slowly added into SMB. Then, the mixture continued to be heated at 180 °C and was stirred at the high speed stirring for 0.5 h. For ease of later description, SMB contained LDHs and SMB contained OLDHs were successively aliased as LSMB and OLSMB, respectively.

#### 2.4. Aging procedures

Rolling thin film oven test was conducted to imitate the thermo-oxidative aging of SMB in the preparation and paving process, which was based on ASTM D 2872. Then, the binder residues were transferred to an ultraviolet aging oven (the UV intensity of 2000 μW cm\(^{-2}\)) at 60 °C for 7 days, which was used to imitate the ultraviolet aging of SMB in the service period.

#### 2.5. Fourier transform infrared spectroscopy test

FTIR spectrum of LDHs and dodecyltrimethoxysilane organic LDHs (KBr disk method), SMB samples before and after aging (liquid film method) were measured by FTIR spectrometer (PerkinElmer Corp., USA). The scanning rate and corresponding test resolution were 64 min\(^{-1}\) and 4 cm\(^{-1}\).

### Table 1. Physical properties of SBS modified bitumen.

| Items                    | Measured values |
|--------------------------|-----------------|
| Penetration (25 °C, 0.1mm) | 57.2            |
| Ductility (5 °C, cm)     | 36.5            |
| Softening point (°C)     | 78.2            |
| Viscosity (135 °C, Pa.s) | 1.65            |

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2.6. X-ray diffraction test
XRD (Palyntical Corp., NL) was applied to analyze the crystal structure of LDHs and dodecyltrimethoxysilane organic LDHs. The scanning rate of 2θ diffractive angle was 2° min⁻¹, the scanning scope was from 0.5° to 60°.

2.7. Static contact angle test
The hydrophilic static contact angles of LDHs and dodecyltrimethoxysilane organic LDHs were tested by CAM (Lauda Corp., GER). The volume of water droplet and the dropping speed were 2 μl and 1 μl s⁻¹, respectively.

2.8. Storage stability test
The storage stability of SMB was estimated by the segregation test, which was based on ASTM D 5976. Firstly, the fluid SMB was injected into an aluminum pipe. Then, the pipe with SMB samples were placed at 163 ± 5 °C for 48 h. After that, the aluminum pipe was transferred to the refrigerator (−2 °C for 3 h) for cooling, and the pipe was averagely divided into three parts. Finally, the softening points of the top and bottom part were tested. The absolute difference value of softening points (ΔS) was used to appraise the storage stability of SMB.

2.9. Physical and rheological properties test
The physical properties (penetration, softening point, ductility) of all SMB specimens before and after aging were acquired according to ASTM D 5, D 36 and D 113. The viscosity was obtained from Brookfield rotational viscometer (Brookfield Engineering Corp., USA) on basis of ASTM D 4402.

The performance of SMB will change before and after aging. To estimate the aging extent of different samples, penetration retention rate (PRR), softening point increment (SPI), ductility retention rate (DRR) and viscosity aging index (VAI) were adopted in this paper. The larger the PRR and DRR, the smaller the SPI and VAI, the better the aging resistance of SMB. These aging indexes can be computed by the following formulas (1)~(4):

\[
PRR = \frac{\text{Penetration after aging}}{\text{Penetration before aging}} \times 100
\]

\[
SPI = \frac{\text{Softening point after aging} - \text{Softening point before aging}}{\text{Softening point before aging}} \times 100
\]

\[
DRR = \frac{\text{Ductility after aging} - \text{Ductility before aging}}{\text{Ductility before aging}} \times 100
\]

\[
VAI = \frac{\text{Viscosity after aging} - \text{Viscosity before aging}}{\text{Viscosity before aging}} \times 100
\]

The rheological properties (complex modulus (G*) and phase angle (δ), ranging from 30 °C to 80 °C) of all SMB samples were achieved by dynamic shear rheometer (Anntton Paar Corp., AT). The test sweep frequency and heating rate were 10 rad s⁻¹ and 2 °C min⁻¹.

3. Results and discussion

3.1. FTIR analysis
The FTIR spectrums of LDHs and dodecyltrimethoxysilane modified LDHs are depicted in figure 1. In the FTIR spectrum of LDHs, an obvious absorption peak around 3688 cm⁻¹ can be observed, which ascribed the stretching vibrations of –OH on the surface of LDHs. However, the absorption peak of –OH was vanished in the FTIR spectrum of OLDHs, it manifested that the –OH amount on the surface of OLDHs declined, which was due to the reaction between the –OH and the hydrolyzed Si–OH of dodecyltrimethoxysilane. The decline of –OH was conducive to inhibit the intergranular agglomeration of LDHs and ameliorate the dispersiveness of LDHs in SMB. Furthermore, in the FTIR spectrum of OLDHs, there were some new absorption peaks, such as the peaks located at 2970 cm⁻¹ and 2875 cm⁻¹ (the stretching vibrations of CH₂ and CH₃). The newly grafting organic groups on the surface of LDHs after dodecyltrimethoxysilane modification were helpful to heighten the compatibility of LDHs in SMB.

3.2. XRD analysis
Because of the multistage laminate structure of LDHs, the interlamellar anions of LDHs can be replaced, which results in the crystal structure changes of LDHs, and ultimately will affect the improvement of LDHs for the aging resistance of bitumen [18–20]. Although it could confirm that dodecyltrimethoxysilane has been chemically grafted on the surface of LDHs through the analysis of FTIR, it could not confirm that dodecyltrimethoxysilane was intercalated into the interlamination of LDHs, namely whether dodecyltrimethoxysilane organic modification has an impact on the crystal structure and the aging resistance of LDHs or not. If dodecyltrimethoxysilane has been intercalated into the interlamination of LDHs, which will cause the interlamellar spacing variation of LDHs. On the contrary, the interlamellar spacing of LDHs will not alter. In order to validate this, it can calculate and contrast the interlamellar spacing of LDHs before and after dodecyltrimethoxysilane modification via the Bragg equation (formula (5)).
Where \( n, \lambda, \theta \) and \( d \) reflects the diffraction level, incident wavelength, diffraction half-angle and the interlamellar spacing, respectively. The larger the \( d \), the wider the interlamellar spacing of LDHs.

The XRD patterns of LDHs and dodecyltrimethoxysilane organic LDHs are plotted in figure 2. It can be observed that the main diffraction peaks (such as 003, 006, 009) of OLDHs were sharp and strong, which was essentially consistent with that of LDHs. The result showed that OLDHs and LDHs all had regular crystal and interlamellar structure. The obtained interlamellar spacing of OLDHs and LDHs by the Bragg equation is listed in table 2. The interlamellar spacing of LDHs was 0.76 nm, and the interlamellar spacing variation of LDHs after dodecyltrimethoxysilane modification was extremely tiny (0.77 nm), which indicated that dodecyltrimethoxysilane has not been replaced into the interlamellar spacing of LDHs, and will not affect the

\[
n\lambda = 2d \sin \theta
\]
crystal structure of LDHs. Combined with the result of FTIR and XRD, it can show that dodecyltrimethoxysilane was only chemically grafted onto the surface of LDHs and will not affect the aging resistance of LDHs. The reaction scheme between dodecyltrimethoxysilane and LDHs is showed in figure 3. Firstly, the hydrolysis of dodecyltrimethoxysilane generated Si-OH. Then, the Si-OH reacted with the surface hydroxyl radical of LDHs. At last, dodecyltrimethoxysilane was grafted onto the surface of LDHs, which resulted in the surface hydroxyl radical reducing of LDHs and the newly introduced organic groups into LDHs.

3.3. Hydrophilia analysis

The hydrophilic static contact angles of LDHs and OLDHs are described in figure 4. The hydrophilic static contact angle of LDHs was 19.1° (as in figure 4(a)), which showed that LDHs possessed better hydrophilia. LDHs possessing better hydrophilia was due to the surface –OH (polar groups) of LDHs, which was just the reason that caused the inhomogeneous dispersion of LDHs in SMB. Compared with LDHs, the contact angle of OLDHs raised to 73.9°, which was because that dodecyltrimethoxysilane surface organic modification reduced the -OH amount and introduced organic groups (as the results of FTIR and XRD). The results of hydrophilic static contact angles showed that dodecyltrimethoxysilane surface organic modification was beneficial to reduce the hydrophilia of LDHs.

3.4. Storage stability

The storage stability of all bitumen samples is demonstrated in figure 5. It can be found that the ΔS of SMB was 2.4 °C, which was due to the segregation of SBS in bitumen during the storage. The reason was that SBS absorbed the light components of bitumen, resulting in the swelling of SBS and floating upward in bitumen (the softening point of the top part increase during the storage stability test). Compared with SMB, the ΔS of LSMB decreased, and the decrease tendency became more remarkable with the LDHs content increasing. Unfortunately, it did not mean that LSMB possessed better storage stability. Conversely, it was due to the intergranular agglomeration of LDHs, which caused the segregation sinking of LDHs in SMB (the softening point of the bottom part increase.

| Samples | \(d_{105} (°)\) | \(d (\text{nm})\) |
|---------|-----------------|-----------------|
| LDHs    | 11.60           | 0.76            |
| OLDHs   | 11.47           | 0.77            |

Figure 3. The reaction scheme between dodecyltrimethoxysilane and LDHs.
contrary with that of SBS), and eventually leading to the $\Delta S$ of LSMB significant decrease. Noteworthily, the $\Delta S$ of OLSMB increased to a certain extent compared with LSMB, but it was less than that of SMB, the result showed that dodecyltrimethoxysilane surface organic modification could improve the storage stability of LDHs in SMB, which was attribute to the inhibiting of dodecyltrimethoxysilane surface organic modification on the agglomeration between LDHs particles and the improvement on the compatibility of LDHs in SMB.

3.5. Physical properties

The physical properties of all SMB samples are exhibited in figure 6. Compare with SMB, the addition of LDHs and OLDHs can increase the softening point and viscosity of binder samples, reduce the penetration and ductility. Because of the inorganic layered structure, the introduction of LDHs or OLDHs in SMB will impede the movement of SBS molecules and bitumen molecules in some extent, which leaded to the physical properties changes of SMB. Furthermore, the influence of OLDHs was more obvious than that of LDHs, which was attributed to the better disperstiveness and stability of OLDHs in SMB.

After aging, the physical properties of SMB seriously deteriorated, the penetration and ductility remarkably decreased, simultaneously, softening point and viscosity increased. The physical property aging indexes (PPR,
SPI, DRR and VAI of all samples are showed in figure 6 (embedded figure). PPR and DRR of SMB was 53.0% and 6.6%, the addition of LDHs and OLDHs inhibited the penetration and ductility reduction of SMB, the PPR and DRR of LSMB increased to 66.4% and 14.7%, and OLSMB showed more remarkable (the PPR of 84.2% and the DRR of 36.8%). Correspondingly, the SPI and VAI values ranked in the order of SMB > LSMB > OLSMB. The result of physical property indexes showed that LDHs and OLDHs can relieve the impact of aging on SMB, and improve the aging resistance of SMB, particularly LDHs after dodecyltrimethoxysilane surface organic modification.

3.6. Rheological property
The complex moduli ($G^*$) of SMB, LSMB and OLSMB before and after aging are displayed in figure 7. It was observed that the $G^*$ of LSMB and OLSMB were higher than that of SMB, and this was because that LDHs and OLDHs acted as the elastic component in SMB. Compared with LSMB, OLSMB performed more obviously, which was credited to the better dispersiveness and stability of OLDHs in SMB. After ultraviolet aging, SMB became stiff, the $G^*$ of all bitumen samples increased to a varying degree, SMB was the most significant, followed by LSMB, OLSMB was the least, the size order was the opposite of that before aging. The result showed that OLDHs and LDHs can relieve the hardening and embrittlement of SMB during the aging, and improve the aging resistance of SMB, particularly OLDHs, which was ascribed the better compatibility of LDHs in SMB after dodecyltrimethoxysilane surface organic modification.

The phase angles ($\delta$) of SMB, LSMB and OLSMB before and after aging are presented in figure 8. Due to the temperature-sensitive of bitumen, the $\delta$ of SMB increased in general with the temperature increase. However, it was noteworthy that a platform can be observed in all the SMB samples, the $\delta$ of SMB samples had little change with the temperature variation during the area. The emergence of the platform was contributed by the network
structure of SBS, the network structure of SBS showed better elastic property during this temperature region, which resulted in the minor change of $\delta$ of SMB with the temperature increasing. When exceeding the platform region, the $\delta$ of SMB significantly increased with the temperature increasing, this was because that the network structure of SBS was gradually disassociated, which caused the viscous component increase of SMB [10, 21, 22]. The temperature ranges for $\delta$ platform of all binder samples are illustrated in table 3, the temperature range of SMB was $39.2 \degree C \sim 49.1 \degree C$ before aging, the temperature ranges of LSMB and OLSMB were enlarged obviously, which were $42.9 \degree C \sim 52.0 \degree C$ and $50.1 \degree C \sim 58.2 \degree C$, respectively. The result manifested that LDHs and OLDHs contributed to improving the disassociation temperature of the network structure of SBS, which was conducive to inhibit the network structure disappearance of SBS at high temperatures and enhance the thermostability of SBS in bitumen.

Figure 7. The complex modulus of all SMB samples before and after aging.

Figure 8. The phase angle of all SMB samples before and after aging.
After aging, the δ of all SMB samples decreased to varying degrees, and the size order of δ was OLSMB > LSMB > SMB, it indicated that OLDHs and LDHs could relieve the impact of the aging on the δ of SMB, especially OLDHs. Furthermore, it could be observed that the δ platform of SMB and LSMB was vanished after aging, which was due to the aging degradation of SBS. Compared to SMB and LSMB, an obvious δ platform (from 44.9 °C to 50.8 °C) can still be found in OLSMB, it manifested that OLDHs not only exhibited excellent performance in ameliorating the aging resistance of bitumen, but also could ameliorate the aging degradation resistance of SBS.

3.7. Chemical structure analysis

The aging of SMB is a complex chemical reaction, on the one hand, the chemical structure of bitumen molecule would change during aging, meanwhile, SBS molecule would degrade [23–25]. To investigate the chemical structural variation of SMB before and after aging, the infrared spectra of SMB, LSMB and OLSMB before and after aging are graphed in Figure 9. In comparison with SMB, an absorption peak (around at 3450 cm$^{-1}$) can be found in LSMB and OLSMB, which was the stretching vibration of crystal water in the interlamination of LDHs, besides, some absorption peaks in the fingerprint region were observed (such as 546 cm$^{-1}$, 451 cm$^{-1}$, etc), which was the lattice vibration of metal hydroxides in the metal laminate of LDHs. During the aging, the chemical structure of bitumen molecule and SBS molecule will change under the influence of oxygen, heat and ultraviolet, which resulted in the content variation of some characteristic groups [26–28], for instance, the peaks at 1700 cm$^{-1}$ (the stretching vibration of C=O), 1030 cm$^{-1}$ (the symmetrical stretching vibration of S=O) and 966 cm$^{-1}$ (the bending vibration of the carbon-hydrogen bond in C=C). Hence, it can be quantitatively analyzed the content of these characteristic groups (C=O, S=O and C=C) before and after aging, which was utilized to estimate the aging resistance of SMB. The corresponding calculation formulas of characteristic groups are showed in the equations (6)–(8), respectively.

$$I_{C=O} = \frac{\text{Area of carbonyl band centered around 1700 cm}^{-1}}{\sum \text{area of spectral bands between 2000 cm}^{-1} \text{ and 600 cm}^{-1}} \times 100$$

$$I_{S=O} = \frac{\text{Area of carbonyl band centered around 1030 cm}^{-1}}{\sum \text{area of spectral bands between 2000 cm}^{-1} \text{ and 600 cm}^{-1}} \times 100$$

$$I_{C=C} = \frac{\text{Area of carbonyl band centered around 966 cm}^{-1}}{\sum \text{area of spectral bands between 2000 cm}^{-1} \text{ and 600 cm}^{-1}} \times 100$$

The characteristic groups content of all SMB samples are portrayed in Table 4. The contents of C=O and S=O were significantly increased after aging than that before aging, but the content of C=C decreased. The aging
of bitumen was considered as the oxidization process, thus resulting in the content of C=O and S=O increasing after aging. Correspondingly, the aging of SBS mainly occurred molecular degradation, which leaded to the content of C=C decreasing after aging. Compared with SMB (υc,o = 19.14, υs,o = 2.43, υc,c = 0.44), the characteristic groups content variation of LSMB (υc,o = 16.23, υs,o = 1.96, υc,c = 0.34) and OLSMB (υc,o = 10.83, υs,o = 1.05, υc,c = 0.25) decreased significantly, especially OLSMB. The results showed that LDHs can prevent the oxidization of bitumen and the molecular degradation of SBS, and improve the aging resistance of SMB, the improvement of LDHs has been further enhanced after dodecyltrimethoxysilane surface organic modification.

### 4. Conclusions

Dodecyltrimethoxysilane surface organic modified LDHs was synthesized and utilized to enhance the aging resistance of SMB. The chemical structure and hydrophilia of LDHs and dodecyltrimethoxysilane organic LDHs were analyzed by FTIR, XRD and CAM, and the effect of OLDHs on physical, rheological properties and chemical structure of SMB before and after aging were thoroughly investigated. Based on the experimental results, the conclusions can be drawn as follows:

1. Dodecyltrimethoxysilane has been chemically grafted to the surface of LDHs, and will not affect the crystal structure of LDHs. The hydrophilia of LDHs was obviously diminished after dodecyltrimethoxysilane organic modification.

2. Compared with LDHs, OLDHs exhibited better compatibility and stability in SMB. The addition of LDHs and OLDHs can ameliorate the high-temperature stability of SBS in bitumen, especially OLDHs, which was helpful for the high-temperature properties of SMB.

3. After aging, SMB became rigid and brittle, the physical and rheological properties of SMB seriously deteriorated. LDHs can alleviate the destruction of aging on the performance of SMB, and the inhibitory of LDHs was further enhanced after dodecyltrimethoxysilane surface organic modification.

4. The aging caused the oxidization of bitumen molecular and the degradation of SBS molecular, OLDHs could restrain the chemical structure variation of SMB during aging. OLDHs showed better performance in improving the aging resistance of SMB.

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### Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.
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