



## Structure characteristics of organic bentonite and the effects on rheological and aging properties of asphalt

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### ABSTRACT

In this paper, the organic bentonite (OBT) was obtained by reacted the bentonite (BT) with surfactant cetyltrimethyl ammonium bromide (CTAB). The microstructures of OBT and OBT modified asphalt were characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TG). The CTAB successfully entered the OBT interlayer, and the OBT forms an exfoliated structure in modified asphalt. Effects of OBT on the high and low temperature properties of matrix asphalt were investigated. The dynamic shear rheometer (DSR) and bending beam rheometer (BBR) analysis indicates that the high and low temperature rheological properties of asphalt were improved attribute to the addition of OBT. The short-term aging, long-term aging, and ultraviolet (UV) aging resistance properties of asphalt were also evaluated. Compared with the matrix asphalt, the OBT modified asphalt exhibited smaller changes in rutting parameter and softening point after short-term aging, and lower creep stiffness and fatigue factor after long-term aging. In the meantime, the UV aging index of OBT modified asphalt was obviously lower than the matrix asphalt. The OBT modified asphalts display better performance both at high and low temperature and better aging resistance under the OBT mixing amount of 4%–6%, especially for the high temperature performance.

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### 1. Introduction

Asphalt with good viscoelastic properties have been widely used in road pavement as the binder. Unfavorable environment conditions and vehicle loads result in the asphalt pavement damage of high temperature rutting and low temperature cracking [1]. What's more, asphalt aging is one of the most leading reasons for asphalt pavement damage. The diffusion of oxygen, heat, and ultraviolet (UV) light during the process of transportation, storage, laying down, and mixing, as well as in service life result in the aging of asphalt. It is necessary to modify and reinforce the properties of asphalt binder [2]. Blending asphalt with polymers is one of the most important methods for asphalt modification. Natural rubber and rubber products, such as styrene butadiene rubber, chloroprene rubber can reinforce high temperature rheological properties and temperature sensitivity performances of asphalt [3]. Thermoplastic resin also can enhance properties of asphalt, such as

polyethylene, and ethylene-vinyl acetate copolymer [4,5]. The most widely used of asphalt modified materials is styrene-butadiene-styrene (SBS) block copolymer. However, it cannot be overlooked that polymer-modified asphalt is expensive and has high environmental pollution [6]. Further efforts should be made for exploring new modifiers of asphalt to obtain better properties. Recently, adding a certain amount of mineral powder to asphalt could greatly improve the asphalt performance. At the same time, the addition of mineral powder in SBS, rubber modified asphalt could improve the bonding ability of asphalt, durability and anti-aging properties of asphalt. Mineral powder is cheap and easy to prepare. Moreover, the organic modified mineral powder could have better compatibility with asphalt. Powder and fiber of common bentonite, limestone powder, diatomaceous earth, cement, lime and asbestos and others are mostly used as asphalt modifiers [7–9].

In this study, cetyltrimethyl ammonium bromide (CTAB) was selected as the organic intercalation modifier of BT. The structure and surface properties of OBT were investigated. Afterwards, the OBT modified asphalts were prepared by melt blending with different contents of OBT. The effects of different proportions of OBT on the structure, physical properties, and rheological behaviors of the modified asphalt were

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investigated. What's more, physical properties, high temperature and low temperature rheological behaviors were evaluated after artificially short-term, long-term and UV aging.

## 2. Experimental methods

### 2.1. Materials

Natural sodium bentonite used in this study is composed of 95% bentonite (purchased from Xinyang, China). The CEC of the sodium bentonite was 0.9 mmol/g. The CTAB and NaOH used for this study were analytical grade. The matrix asphalt 70<sup>#</sup> was obtained from China National Offshore Oil Corporation Taizhou Petroleum Asphalt Factory.

### 2.2. Modified bentonite preparation

6.56 g of CTAB (equal to 1.5CEC) surfactant was dissolved in 250 g of deionized water. The mixture was stirred and 10 g of BT was added in the dispersed surfactant solution. Then, pH of the dispersed solution was adjusted to 11 by NaOH solution and stirred at 70 °C. The precipitates were collected by centrifugation, washed four times by deionized water. After dried at 70 °C for 12 h, the OBT powder was grinded to desired size.

### 2.3. Preparation of OBT modified asphalts

OBT modified asphalt was prepared by the melt blending method. Firstly, matrix asphalt was heated up to melting at around 120 °C. Then, four contents (2%, 4%, 6% and 8% by weight) of OBT were added to matrix asphalt. The mixtures were stirred by high-shear mixer at 140 °C ~ 155 °C (5000 rpm), than OBT modified asphalt were obtained.

### 2.4. Characterization of OBT and OBT modified asphalts

X-ray diffraction (XRD) patterns of BT, OBT and OBT modified asphalts were obtained by Rigaku D/max 2550 with Cu K $\alpha$  radiation ( $\lambda = 0.154$  nm; 40 kV and 200 mA) over the scanning range of  $2\theta = 0.5\text{--}30^\circ$  with step width of  $0.02^\circ$ . Simultaneous thermogravimetric (TG) and differential thermal analysis (DTA) curves of BT and OBT were conducted by a thermal analyzer equipment HCT-1 under atmospheric air stream, while heating from room temperature to 900 °C. The chemical characteristics of BT and OBT were measured by Fourier transform infrared spectroscopy (FTIR) with the Nicolet NEX-US 670 IR spectroscopy with the range of  $400\text{--}4000$   $\text{cm}^{-1}$ . The morphology features of OBT and the dispersion characteristic in the modified asphalts were characterized by scanning electron microscope (SEM). SEM images were recorded on the HITACHIS-3000 N electron microscope.

### 2.5. Physical properties test

The softening point, penetration (25 °C) and ductility (5 and 15 °C) of modified asphalts were measured according to the standards of ASTM D36, ASTM D5 and ASTM D113–86, respectively.

### 2.6. High and low temperature rheological characterization

The high temperature rheological characteristics of matrix asphalt and OBT modified asphalt were tested by dynamic shear rheometer (DSR) (MCR 101, Anton Paar Instruments) according to the standard of AASHTO TP5. The samples of complex shear modulus ( $G^*$ ), phase angle ( $\delta$ ) and rutting parameter ( $G^*/\sin\delta$ ) were measured in 25 mm parallel plates at the fixed frequency of 10 rad/s and variable strain.

The low temperature rheological characteristics were measured by bending beam rheometer (BBR). Creep stiffness ( $S$ ) and creep rate ( $m$ )

of the matrix asphalt and OBT modified asphalt beams ( $125 \times 6.35 \times 12.7$  mm) were measured at the loading time of 60 s at  $-15$  °C,  $-9$  °C,  $0$  °C according to the standard of AASHTO TP1.

### 2.7. Artificial simulation aging procedure

Three different artificial simulation aging procedures were used to characterize the anti-aging properties of modified asphalt, which mainly include thin-film oven test (TFOT), pressure aging vessel (PAV) and UV aging. TFOT can simulate the short term aging of asphalt during the process of transportation, storage, mixing, laying and compaction of the asphalt mixture. Asphalt was measured by DSR after heated in the thin-film oven for 5 h at 163 °C according to the standard of ASTM D1754.

PAV aging can simulate long term aging of oxidation process of asphalt taking place during the service life of the pavement according to the standard of AASHTO PP1. In this study,  $50 \pm 0.5$  g of asphalt sample which had been suffered to TFOT aging was kept in a chamber at 100 °C under the air pressure of  $2.1 \pm 0.1$  MPa for 20 h. Softening point, DSR and BBR of the aged sample were measured.

The UV aging of asphalt was simulated in a chamber with the UV lamp of 500 W.  $3.47 \pm 0.02$  g of matrix asphalt and OBT modified asphalts were poured on a pan with a diameter of  $94 \text{ mm} \pm 0.5$  mm, the thickness of asphalt film is about 1 mm. The sample was suffered with an ultraviolet intensity of about  $3.18 \text{ W/m}^2$  and analyzed by DSR test after UV irradiation for 336 h.

## 3. Results and discussion

### 3.1. XRD analysis

The XRD patterns of BT, OBT and OBT modified asphalt which characterized by XRD were shown in Fig. 1. The  $2\theta$  and the size of BT, OBT and OBT modified asphalt basal spacing ( $d_{001}$ ) were given in Table 1 by the Bragg equation:  $2d\sin\theta = n\lambda$ . The basal spacing for raw BT was 1.27 nm. The size of the OBT layer increased significantly, which expanded the basal spacing to 1.95 nm. The results show that the CTAB surfactant entered the layer of BT successfully [10]. After being mixed with asphalt, the interlayer spacing of OBT modified asphalt increased to 1.97 nm. Evidently, the interlayer spacing of OBT modified asphalt increased, which implied that the asphalt molecules intercalated into the layers of OBT during the melt blending process and OBT modified asphalt forms an intercalated or exfoliated structure [11].

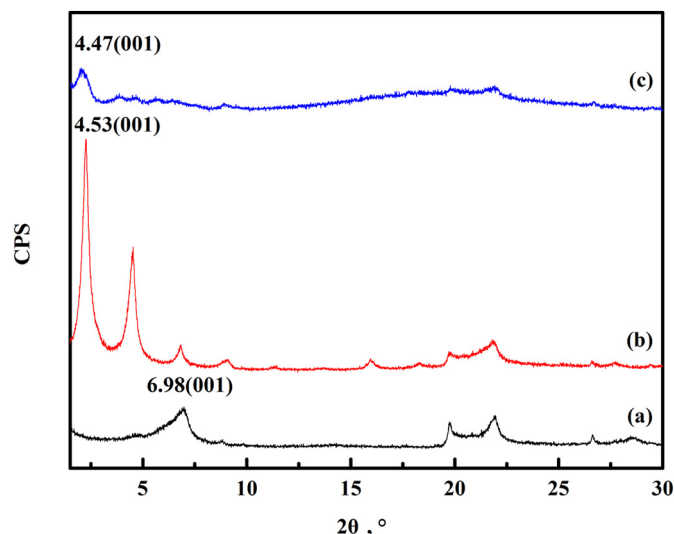


Fig. 1. XRD patterns of (a) BT, (b) OBT, (c) OBT modified asphalt.

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