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Effect of polyphosphoric acid on physical properties, chemical composition and morphology of bitumen

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HIGHLIGHTS

- The influence of PPA depends on the base bitumen.
- The PPA modification is affected by the colloidal index of base bitumen.
- The changes of morphology are in accordance with that of physical properties and chemical composition.

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ABSTRACT

The effect of polyphosphoric acid (PPA) on chemical composition, physical properties and morphology of bitumen was investigated. The morphology of the binders was characterized by atomic force microscopy. The results show that the influence of PPA on chemical composition, physical properties and morphology of bitumens depends on the base bitumen. The colloidal index shows an obvious effect on the chemical composition and physical properties as well as morphology of bitumen during PPA modification. A strong correlation is observed between morphology and chemical composition as well as physical properties for different bitumens after PPA modification.

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1. Introduction

Bitumens are important factors in pavement construction and roofing systems due to their very good binding properties [1]. Specifications on paving grade bitumens have become quite severe in order that the mixes resist climate and traffic [2]. In an attempt to change its characteristics and improve its performance, bitumen is often modified by elastomers, plastomers, thermosets, sulfur, or mineral acid. There is now much interest in the use of polyphosphoric acid (PPA) to modify bitumen since it permits to significantly harden bitumen in an easily controllable way [3–7]. After PPA modification, the high temperature rheological properties of bitumen can be improved remarkably without affecting the low temperature grade [3]. Edwards et al. studied the rheological effects of PPA in bitumen 160/220–high, medium and low temperature performance. Adding PPA especially to a non-waxy bitumen, showed considerable positive effects on the rheological behavior at higher, medium and low temperatures [8,9].

There are also some studies referred to the reaction mechanism between PPA and bitumen. Orange et al. considered that PPA acted through the neutralization of polar interactions between the stacked asphaltenes molecules, either by protonation of basic sites or by esterification. The overall effect was to increase the solvation of the asphaltenes, increasing in turn the solid fraction and hence, the viscosity [10]. Baumgardner et al. proposed various bitumen-dependent mechanism of PPA modification which also affected the lower weight components of the bitumen: co-polymerization of the saturates, alkyl aromatization of the saturates, cross-linking of neighboring bitumen segments, the formation of ionic clusters and the cyclization of alkyl-aromatics [3]. Model compounds, isoquinoline and 1-methyl-2-quinolinone, were reacted with PPA by Masson et al. It showed that both compounds formed salts with PPA [6]. Other model compounds, such as bisphenol A, butyl phenyl ether, acetophenone, benzoic acid [7], and sulfur compounds [5] were also investigated.

As mentioned above, more attention of the existed studies has been paid to the PPA modification mechanism. But there are relatively few published articles about the correlation between chemical composition and morphology of bitumen under the influence of PPA. Bitumen is a complex system of different constituents, made of hydrocarbons and hetero-atoms. After fractionation of the bitumen by specific solvents, four main chemical families (saturates, aromatics, resins, asphaltenes) are obtained [11]. The

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properties of bitumen are governed by the chemical–physical interactions of these constituents [12]. So investigating the influence of PPA on chemical composition and physical property of bitumen, especially for different base bitumens, will help to better understand the interaction of PPA with different bitumens.

The atomic force microscopy (AFM) is a very high-resolution type of scanning probe microscopy. It is capable of measuring topographic features at the nanometer-scale or even at atomic-scale resolution. The AFM has the advantage of imaging almost any type of surface, including polymers, ceramics, composites, glass, and biological samples. Loeber first used AFM for the observation of a heat-cast bitumen film, thus preserving the solid-state morphology, without the extraction of its components with solvents [13]. Ever since then, AFM has been used by more and more researchers to investigate the morphology of bitumen [3,14–16]. It allows to visualize precise details of superstructure and nanoscale structures of bitumen without special techniques of sample preparation.

In this paper, the effects of PPA on chemical composition, physical properties and morphology of different bitumens were investigated. Correlation between morphology and chemical composition as well as physical properties was examined.

2. Experimental

2.1. Materials

Three bitumens, denoted B1, B2 and B3, were used in this study. The penetration grades of B1, B2 and B3 were 92, 85 and 63 respectively. The crude source of bitumens B1 and B3 was Saudi Arabia, and the source of B2 was China. The physical properties and chemical composition of the three bitumens were listed in Table 1. PPA (105% relative contents of H_3PO_4) was supported by Changzhou Wujin Huayang Chemical Co., Ltd., China.

2.2. Preparation of PPA modified bitumen

The bitumen was heated to become a fluid in an iron container, then upon reaching about 170 °C, a certain amount of PPA was added into bitumen, and the mixture was blended at 2000 r/min rotation speed for 40 min. The pristine bitumen was also processed under the same conditions in order to compare with the PPA modified bitumen.

2.3. Physical properties test

The physical properties of bitumens, including softening point, penetration (25 °C) and ductility (15 °C), were tested according to ASTM D 36, ASTM D 5 and ASTM D 113, respectively. Brookfield viscometer (Model DV-II+, Brookfield Engineering Inc., USA) was employed to measure the viscosity of the bitumen according to ASTM D 4402.

2.4. Chemical composition

The chemical composition of bitumens was determined according to SH/T 0509-92 (China Petroleum and Chemical Standard). First, bitumen was deasphaltened to yield asphaltenes and maltenes which is the n-heptane soluble portion. Then, the maltens were further separated into three fractions, saturate (soluble in n-heptane), aromatic (soluble in toluene) and resin (soluble in toluene and ethanol absolute mixture), by column chromatography. All fractions were weighed after the complete removal of their respective solvents.

Table 1
Physical properties and chemical composition of bitumens.

Physical properties and chemical composition		B1	B2	B3
Physical properties	Penetration (25 °C, 0.1 mm)	92	85	63
	Softening point (°C)	46.3	43.4	49.0
	Ductility (15 °C, cm)	>150	>150	>150
	Viscosity (60 °C, Pa s)	151	124	245
Chemical composition	Saturates (%)	21.82	30.02	19.83
	Aromatics (%)	46.19	30.21	45.20
	Resins (%)	20.45	32.98	23.80
	Asphaltenes (%)	11.54	6.79	11.17
	Colloidal index[a]	2.00	1.72	2.23

[a] Determined according to Ref. [1].

2.5. Characterization

AFM (Model DI Nanoscope IV, American Veeco Company) was applied to investigate the micro-morphology of the binders. A hot liquid drop of bitumen at 140 °C was carefully placed on a 10 mm × 10 mm × 1 mm steel disk, which was heated for 1–2 min on a hot plate at about 140 °C, a temperature high enough to melt bitumen, but not so high that it would oxidize rapidly. The bitumen was spread out with a blade to form a round film of about 5 mm in diameter. This hot film was left on the hot plate undisturbed for an additional 1 min to allow the surface to flow to a smooth and glossy finish [14]. For AFM analysis, the film was then cooled to ambient temperature (about 5 °C), covered by a glass cap to prevent dust pick-up and annealed for a minimum of 24 h before imaging [16]. Topographic and phase images were scanned using an etched silicon probe. Cantilever was 125 μm long with curvature radius at 5–10 nm and height at 15–20 μm. The drive frequency was 260 kHz and the drive amplitude was 56 mw. Test was operated in tapping mode. All the microphotographs show a 15 μm × 15 μm region unless otherwise indicated.

3. Results and discussion

3.1. Effect of PPA on physical properties of bitumen

The physical properties of PPA modified bitumen B1 with different PPA content are shown in Fig. 1. It can be found that the softening point is enhanced quickly with the increasing of PPA content. The viscosity increases gradually when the PPA content is no more than 1 wt%. But when the PPA content exceeds 1 wt%, the viscosity increases linearly. It indicates that the high temperature property of bitumen is improved with the increase of PPA content. According to Fig. 1c and d, the ductility and penetration of bitumen B1 are decreased to some extent, especially when the PPA is increased from 0.5 wt% to 1.5 wt%. However, PPA has a little effect on the ductility with low content (0.5 wt%), and when the PPA content is more than 1.5 wt%, the decrease rates of penetration and ductility are reduced. It is generally accepted that the ductility of bitumen is largely influenced by the resins content in bitumen. According the experimental results, the resins in bitumen B1 are decreased remarkably with the increasing of PPA, and consequently, its ductility is reduced obviously.

The influences of PPA on the physical properties of different bitumens at the same addition content (1 wt%) are given in Fig. 2. Changes of softening point, viscosity increment(VI), penetration retention rate (PRR) and retained ductility (RD), which are calculated according to formulas (1)–(3), are used to evaluate the effects of PPA on the physical properties of different bitumens.

$$VI = \frac{\text{Modified viscosity value} - \text{Unmodified viscosity value}}{\text{Unmodified viscosity value}} \times 100 \quad (1)$$

$$PRR = \frac{\text{Modified penetration value}}{\text{Unmodified penetration value}} \times 100 \quad (2)$$

$$RD = \frac{\text{Modified ductility value}}{\text{Unmodified ductility value}} \times 100 \quad (3)$$

It can be seen that compared with bitumen B1 and B3, PPA shows a little influence on the physical properties of bitumen B2. Interestingly, the penetration of bitumen B2 is increased to some extent and the ductility is slightly affected by PPA. As mentioned earlier, PPA has little effect on the colloid structure of bitumen B2 due to its highest resins and the lowest asphaltenes content. Correspondingly, its physical properties are slightly influenced.

Compared with bitumens B1 and B2, there are strong interactions between PPA and bitumen B3 which shows the higher

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