



# Dynamic stability of ethylene-vinyl acetate copolymer/crumb rubber modified asphalt



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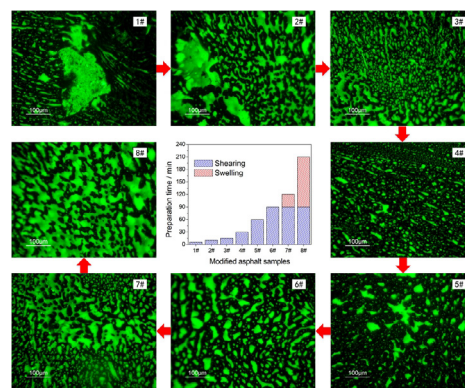
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## HIGHLIGHTS

- Dynamic stability is considered for EVA/CR modified asphalt.
- Morphology variation of modified asphalt samples in the preparation process is observed.
- AFM is used to reveal the stabilization mechanism of polymer modified asphalt.

## GRAPHICAL ABSTRACT



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## ABSTRACT

In this paper, the recovered waste packaging ethylene-vinyl acetate copolymer (EVA) and crumb rubber (CR) were used as polymer modifying agents to prepare EVA/CR modified asphalt, and the EVA/CR modified asphalt's dynamic stability were studied. After characterized and analyzed with conventional physical performance test, differential scanning calorimetry (DSC) and Fourier transform infrared spectroscopy (FTIR), it can be found that the macro and micro properties of modified asphalt in the process of modification modified asphalt evolved from an unstable state to a dynamic stable state and until to a completely final stable state after removed the shear force. The polymer particles gradually dispersed evenly in the modified asphalt and a cross-linked polymer network structure was formed during the shearing process, which were clearly showed through the fluorescence microscopy images. Finally, the interaction between the polymer modifying agents and base asphalt and its impact to the properties of the system were also analyzed combining macro, micro properties of modified asphalt and the morphology changes of modifier and asphaltene. In addition, stabilization mechanism of EVA/CR modified asphalt was revealed through the former analysis.

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## 1. Introduction

Polymer modification of base asphalt is the technique through mechanical mixing or chemical reactions of polymer and base asphalt [1], which attracted more and more researchers in the past 40 years [2–7]. Usually, the road performance of asphalt can be

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significantly improved after modification, the modification efforts not only depend on the addition of polymer, but also on the type of the base asphalt and the polymer agent [8,9]. It was showed the polymer modifier is not chemical reacted with the base asphalt, but uniformly dispersed in the base asphalt and adsorbed the asphalt's component, so it is just the coexistence in physical meaning. With good compatibility, the modifier will be dispersed into a smooth, continuous network in the modified asphalt system, or condensed into flocculent, massive blocks with bad compatibility, and then separated out and precipitated when the modified asphalt cools [10]. Compatibility is a property of the modifier fine particles uniformly distributed in the medium of asphalt without stratification, cohesion or separated from each other. It depends on the interface properties of the modifier and the base asphalt, solubility parameter and their molecular structure [1,9]. Once polymer modified asphalt that was processed by shearing, grinding, mixing or other physical method was stopped stirring in the storage and/or transport process, the polymer is easily prone to segregation or condensation. However, the compatibility directly related to the performance of modified asphalt products [7,11,12]. How to evaluate the storage stability of modified asphalt conveniently, reliably and comprehensively? The in-depth research of the compatibility and the stable mechanism of the system is needed.

In recent years, experts and scholars launched a series of studies around the field of compatibility and stability of modified asphalt. Navarro et al. [13] studied the storage stability of the rubber modified asphalt and found that the change of storage temperature directly affects the stability of modified asphalt system and leads to the distribution change of rubber particles in the system. Zhang et al. [14,15] studied the effects of ageing on the rheological properties of SBS/sulfur modified asphalt, and the results showed that the addition of sulfur leads to the formation of a reticulate structure in the system, and thereby significantly improving the thermal stability of SBS modified asphalt. Nciri et al. [16] studied the compatibility of SBS with matrix asphalt and found that the compatibility of modified asphalt system is affected by the properties of matrix asphalt and the content of modifier. Polacco et al. [17] found that a storage-stable mixture can be achieved if the polymer content remains below the total mass of 4 wt%, and the phases in the polymer concentration areas become unstable and tend to separated out without continuous stirring at a high temperature. Presti [18] thought that crumb aromatic oils can be absorbed by asphalt light components in the asphalt modification process, and then the particles swelled and softened, which further promote their compatibility. Fang et al. [19] believed that the polymer chemical structure and the chemical reaction will affect its compatibility with base asphalt, thereby affecting the performance of its modified asphalt. Wang et al. [20] argue that the poor compatibility between the polymer modifier and the asphalt is the reason for the poor stability of the polymer modified asphalt, which is due to the differences in density, molecular weight, polarity, solubility of the polymer and base asphalt. Through the fluorescence microscope microscopic observation and dynamic mechanical scanning, Liang et al. [21] studied the microstructure of the modified asphalt samples in the modification process and the results reflected the compatibilization process of the modifier and the base asphalt.

Some progress was made in the compatibility and stability exploration of modified asphalt system. However, the discussion of the stability mechanism was almost based on the thermodynamic view point and ignored the dynamic equilibrium influence on the stability of polymer modified asphalt system [4,22]. The polymer and the asphalt have high molecular weight and high viscosity. When the two materials were sheared to disperse under high temperature, the whole system is in a dynamic state, called dynamic equilibrium [23]. Actually, the stability of modified

asphalt system not only depends on the thermodynamic compatibility, but also on the disperse balance of the system which also called technological compatibility of the modified asphalt blends. Technological compatibility means that some thermodynamically compatibilization incompatible blends can be obtained by mixing them mechanically or by the compatibilization method and a sufficiently stable blend can also be obtained. This blend forms a multiphase morphology in the microscopic region, but maintains its uniformity at the macro level [24]. Based on this situation, asphalt modification was operated with waste packaging EVA and crumb rubber which as a resin and a rubber modifier in this paper, and the relationship among the asphalt modification technology, properties and the system stability balance of the EVA/CR modified asphalt were discussed and analyzed.

## 2. Materials and methods

### 2.1. Materials

Base asphalt, Xi'an 90A # asphalt, was purchased from SINOPEC Xi'an Company, and its conventional performance indicator penetration (25 °C) is of 86.1 dmm, softening point is of 51.3 °C and ductility (5 °C) is greater than 200 cm. EVA modifier particles is recycled packaging stretch film which was extruded and granulated by a SHJ-35 twin-screw extruder (Nanjing, China). Crumb rubber was crushed with waste rubber tires purchased from Xi'an Zhongxuan Rubber Factory (Shaanxi, China), and its particle size is of 30–40 mesh.

### 2.2. Preparation of modified asphalt

High speed shearing method was used to prepare modified asphalt. Firstly, 500 g asphalt in an iron container was heated until it became fluidic, and then 2 wt% EVA and 4 wt% CR were added into the asphalt and stirred for 10 min when the temperature rose to about 170 °C. Secondly, the binders were sheared using a FLUKO FM300 high shear emulsifier (Shanghai, China) for 5 min to 90 min with a shearing speed of 3000 rpm to prepare EVA/CR modified asphalt samples which listed in Table 1. Among the samples, the preparation time of the samples 1#–6# was the shearing time in the shearing process, and the preparation time of samples 7# and 8# included the shearing time 90 min and the designed swelling time in 170 °C oven.

### 2.3. Measurements

#### 2.3.1. Physical properties tests

The performance indexes of asphalt samples such as penetration, softening point and ductility (5 °C) were respectively conducted in accordance with ASTM D5, D36 and D113.

#### 2.3.2. Differential scanning calorimetry (DSC)

The thermal behavior of the modified asphalt was carried out by using a DSC 200F3 differential scanning calorimeter (Netzsch, Germany). About 10 mg asphalt sample were firstly eliminated the thermal history and then scanned in at a heating rate of 10 K/min from 40 to 160 °C under nitrogen atmosphere.

#### 2.3.3. Fourier transform infrared spectroscopy (FTIR)

Infrared spectra of asphalt samples were recorded with a FTIR-8400S spectrometer (Shimadzu, Japan) which scans from 400 to 4000  $\text{cm}^{-1}$ . A thin layer of modified asphalt samples was painted on KBr discs for testing.

#### 2.3.4. Fluorescence microscopy

Polymer distribution and its phase morphology were observed with a Nikon 80i fluorescent microscope (Japan). Hot modified asphalt drops in the glass slide, and was pressed into a thin layer with a cover glass, and then the sample was observed in fluorescent microscope with green exciting light after cooling.

#### 2.3.5. Atomic force microscopy

The morphology of modified asphalt was observed using a Pico Plus atomic force microscope (USA) that the resonance frequency was 150 kHz, force constant was 0.4 N/m and the scan rate was 1.5 Hz. Asphalt sample was heated and dropped on a 14 mm × 18 mm silicon wafer, then placed in 120 °C oven for 1 h leveling it to a diameter of about 1 cm, and finally the tested sample was made after placing it in the air more than 24 h. The tested sample should be covered to prevent contamination.

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