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# Methods to prepare polymer modified bitumen samples for morphological observation

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## HIGHLIGHTS

- Various methods were evaluated to prepare morphological observation samples of polymer-modified bitumen.
- Traditional sample preparation methods are susceptible to external force, temperature, and some other manual factors.
- Improved methods can ensure surface smoothness, original morphology and laboratory reproducibility.
- Preparation parameters for improved methods are given according to microcosmic morphology.

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## ABSTRACT

Analysis of microcosmic morphology is significant to the quantitative performance evaluation of polymer-modified bitumen. Diverse methods to prepare observation samples have been used by many scholars but a standard method has not been established. In this study, various preparation methods, namely, hot bitumen dripping on microscope slides (HBDM), frozen bitumen fracture with mechanics (FBFM), and improved method, hot bitumen molding into metal wares (HBMM), were evaluated in terms of influence factors, observation effect, and microcosmic morphology parameters using styrene–butadiene–styrene block copolymer modified bitumen. Results indicate that traditional observation sample preparation methods are such susceptible to external force, temperature, and some other manual factors that obtaining samples with good reproducibility and stability is difficult. HBDM cannot ensure surface smoothness. Applying pressure on a sample by a cover slip inflates and stretches the polymer phase while heating under microscope slides changes the original morphology of the sample. Due to flexural–tensile and shearing stresses, mechanical force influences the microcosmic morphology and section smoothness of samples acquired by FBFM. Compared with traditional methods, the improved method (i.e., HBMM) can make up for the disadvantages. Finally, appropriate size and storage temperature for HBMM were given to obtain observation samples with low variability.



## 1. Introduction

Analysis of biphasic systems based on microcosmic morphological images acquired using a fluorescence microscope is an effective means to study performance and mechanism of modified bitumen [1–4]. According to the principle that polymer modifiers and bitumen show different colors under blue light, digital image processing technology can distinguish the polymer phase from the matrix bitumen background [5–8]. Sample preparation is a key process before acquiring fluorescence microscopic images. Obtaining original images with high resolution and quality is not difficult for advanced photoelectric integrated image acquisition system [9–10]. However, few scholars paid attention to the factors that influence observation sample preparation and most scholars even avoided discussion about these factors. Based on the references, diverse methods were used to prepare observation samples. Hot bitumen dripping on microscope slides (HBDM) was mentioned by Xinjun Feng and Baochang Zhang. To obtain a smooth surface, they placed cover slips on the hot bitumen dripping and applied a certain pressure [11,12]. Guangtao Gao and Chunfa Ouyang did it by heating under microscope slides [13,14]. However, Xiong Liu thought that surface smoothness would meet the requirements by placing cover slips on the hot bitumen dripping slightly and ensuring the cover slips to be horizontal as soon as possible [15]. Xianming Kong adopted frozen bitumen fracture with mechanics (FBFM) to freeze modified bitumen slices with a thickness of 3–5 cm and break them to obtain fracture sections

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directly [16]. Panagiotis fabricated a light-weight steel cylinder mold (15 mm height and 31 mm diameter) to observe the microstructure of rubber powder-modified bitumen [17]. Hot rubber powder-modified bitumen was poured into a hole punched at the center of the cylinder mold. The modified bitumen was then kept in a refrigerator for 5 h at −25 °C; additional 5 h was necessary to obtain a smooth surface after scraping.

But there were many uncertain factors which had negative effects on observation samples in the methods mentioned above. Although heat and pressure can produce a surface smooth, temperature, heat-up time, and magnitude of pressure are difficult to control. Whether these factors interfere with the microcosmic morphology structure of modified bitumen is also an inevitable question. FBFM avoids the influence of these factors, but obtaining a smooth fracture section by manual breaking is almost impossible. In addition, mechanical cutting may change the microcosmic morphology of the polymer near the fracture section. Besides, it is difficult to fix the fractured part in a microscope platform. Moreover, all these operations may change the original morphology of the samples. This study aims to establish a standard sample preparation method based on microcosmic morphology. The microcosmic morphology of polymer-modified bitumen was also quantitatively analyzed.

**Table 1**
Properties of matrix bitumen binder.

| Bitumen grade | Penetration (25 °C, 0.1 mm) | Softening point (°C) | Ductility (5 °C, cm) | Thin film oven test (163 °C, 5 h) | |
|---|---|---|---|---|---|
| | | | | Mass loss (%) | Residual ductility (15 °C, cm) |
| 70 | 74 | 47 | >100 | 0.02 | 32 |

**Table 2**
Capture parameters and image processing algorithms.

| | | | |
|---|---|---|---|
| Exposure time | 00.500.000 (Image Pro Plus) | Noise reduction | Two-dimension wiener filtering |
| Magnification | 20× | Image enhancement | Gray-scale transformation |
| Storage format | *.jp2 | Image segmentation | Otsu threshold |
| Size | 4140 × 3096 (PPI) | Binarization | im2bw (MATLAB function) |

## 2. Samples preparation methods

### 2.1. Raw materials

Matrix bitumen binder (penetration grade 70) produced by Shell Group of Companies and SBS modifier from Yueyang Petrochemical Company were used to prepare the SBS-modified bitumen for the experiment. The technical properties of the matrix bitumen binder are shown in Table 1.

### 2.2. Experimental methods

The optimal dosage of the SBS modifier is 4.5% [18–19]. Traditional preparation method of SBS-modified bitumen using high-speed shearing emulsifying machine was adopted in the experiment. Segregation test of modified bitumen requires that the difference value of upper softening point and lower softening point must be less than or equal to 2.5 °C. Hence, the shearing time was determined to be 90 min. Fig. 1 clearly shows the logical relationship between the materials, experiment, and performance. The tests were conducted according to the test specification of bitumen and bitumen mixtures used in the highways of China (JTG E20-2011) [20].

Fluorescence microscopy tests were performed with OLYMPUS BX41 microscope at room temperature. Uniform capture parameters and image processing algorithms were listed in Table 2.

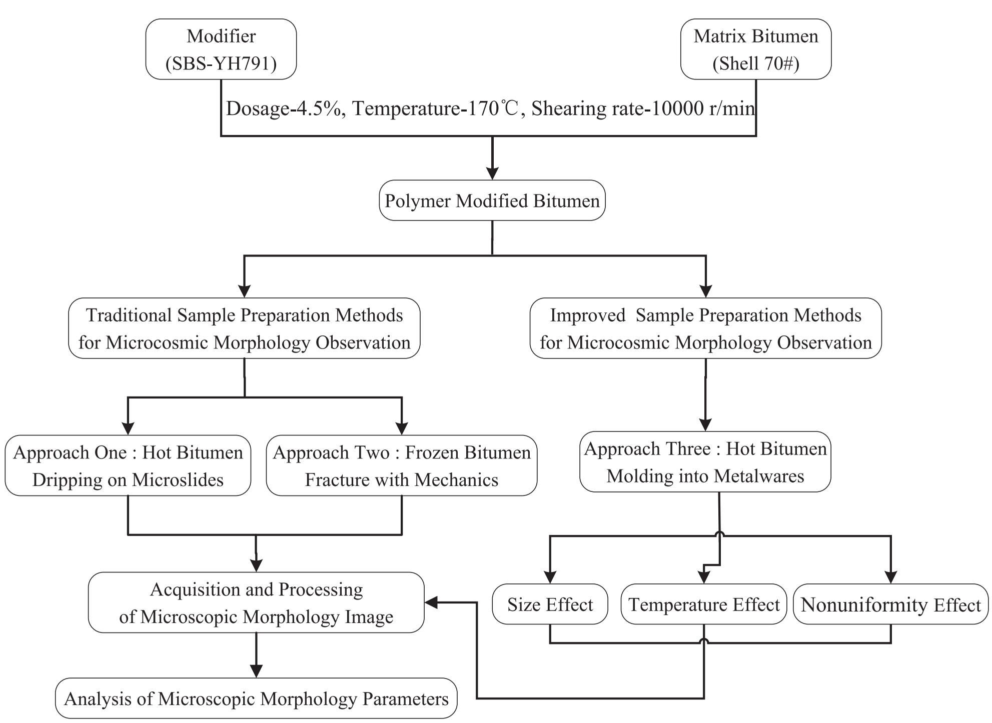


**Fig. 1.** The experimental technological process.