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Microstructure evaluation of polymer-modified bitumen by image analysis using two-dimensional fast Fourier transform

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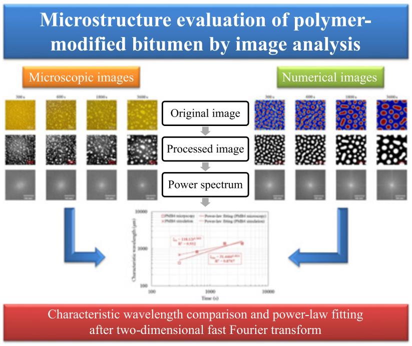
HIGHLIGHTS

- A novel method to evaluate polymer-modified bitumen microstructure is proposed.
- Two-dimensional fast Fourier transform is successfully applied to analyse images.
- The proposed method is valid for differentiating various microstructures.
- The proposed method is capable of characterising effects of temperature.
- The proposed method can characterise temporal evolution of microstructure.

GRAPHICAL ABSTRACT



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ABSTRACT

Aiming to quantitatively evaluate the microstructure of polymer-modified bitumen (PMB) for roads, this paper employs the two-dimensional fast Fourier transform (2D-FFT) to process the microscopic and numerical images of four PMBs. The related derivative parameters, including the characteristic frequency and wavelength, are computed from the 2D-FFT power spectrum. The results show that the absence/presence of a characteristic frequency (range) on the power spectrum can indicate the lack/existence of the corresponding periodical structural pattern(s) in the original PMB image. A lower characteristic frequency usually represents a coarser PMB microstructure while a higher one implies a finer PMB microstructure. The 2D-FFT method is thus valid for differentiating various PMB microstructures. The proposed method is also capable of quantitatively evaluating the effects of temperature and the temporal evolution of PMB microstructure during phase separation. As the separation continues, the decrease of characteristic frequency indicates the coarsening process of a PMB microstructure. Additionally, the numerical reproduction of the observed phase separation is evaluated with the same method. The quantitative comparison with the experimental results reveals that the simulations fairly reproduced the microscopy observation results despite some deviation. The proposed method provides a foundation for the microstructure-based modelling of PMB performance in the future.

1. Introduction

Polymer-modified bitumen (PMB) is widely used as a high-grade binder for the construction and maintenance of road surface layers in

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many countries, due to its enhanced performance comparing with the base bitumen [1–3]. As a composite material in a general sense, PMB has been intensively studied for decades in order to understand its composition-structure-property relationship [4]. Among the previous studies, PMB microstructure (in other word morphology) attracted a lot of attention from researchers all over the world [5–32]. This is because the microstructure is not only related to the PMB stability during storage and transport at high temperatures, but also affects the binder properties and thus finally the road performance at service temperatures.

Various microscopes were employed to investigate the PMB microstructure, including optical and electron microscopes. Microscopic results display the microstructure visually by images. However, the evaluation of the observed PMB microstructure relies on the analysis of the captured images. The homogeneity of a microscopic image from the storage temperature is usually qualitatively related to the PMB storage stability. But some previous studies [18–31] did try to evaluate the PMB microstructure quantitatively with the image analysis method. Most of these studies [18–20,22–24,26–28,30] discussed the percentage (area fraction) of the polymer-rich phase while some others analysed the number, size and shape of the polymer-rich droplets [20,21,25,30].

The percentage of the polymer-rich phase in a PMB is determined by the polymer content and the swelling ratio of the polymer modifier. Although it is an important factor, the percentage by itself does not reflect the specific PMB microstructure. Nevertheless, the analysis of polymer-rich droplets might be feasible for PMBs with a dispersed droplet structure, but it does have the difficulty in evaluating more complex structures, e.g. the widely reported ideal PMB microstructure with two interlocked continuous phases. Furthermore, a few derivative parameters from the average pixel information of processed images [29,31] were recently reported for quantitatively evaluating PMB microstructure and storage stability. However, the simply averaging of the image pixels may erase and hence lead to missing some of the most important PMB structural information. Thus, new quantitative parameters with higher validity are still needed for the effective evaluation of PMB microstructure. The potential applications of these parameters can be, for example, the comparison between the numerical simulation and experimental results, microstructure-based modelling of PMB performance and more.

With the aim to quantitatively evaluate the PMB microstructure, this paper uses a two-dimensional fast Fourier transform (2D-FFT) algorithm to process the PMB microscopic images and computes the related derivative parameters. After a brief description of the studied materials and employed method, the microstructures of four different PMB binders are analysed with the image analysis results. The effects of temperature are discussed. The entire PMB phase separation process observed at different time points is characterised with the computed derivative parameters. Finally, the numerical reproduction of the observed phase separation is assessed and compared with the experimental results.

2. Materials and method

2.1. Materials

Four different PMB binders are studied in this paper, i.e. PMB1, PMB2, PMB3 and PMB4. They all contain the same 5% linear styrene-butadiene-styrene (SBS) copolymer as the modifier by weight of the blend. The SBS copolymer has a weight average molecular weight of 189,000 g/mol. But the four base bitumen binders in the PMBs are from different sources despite the same penetration grade 70/100. The PMBs were prepared in laboratory by mixing the modifier and base bitumen binders at 180 °C with stirring at about 500 rpm for 3 h. The conventional PMB properties, including penetration, softening point, penetration index and 180 °C storage stability, were tested and listed in Table 1.

2.2. Method

2.2.1. Microscopy observation and numerical simulation

Both experimental and numerical PMB microstructure images are analysed and discussed in this study. The experimental images of the four PMB binders were captured by a fluorescence microscope at room temperature. The thin film method [15] was used for the microscope slide preparation by spreading a drop sample with a cover slip and fast cooling of the sample from a higher temperature. Due to the small amount and thin film of PMB for each slide, it is believed that the observations of thin film samples can represent the original PMB morphology at higher temperatures. In addition to the ordinary observation of PMB slides prepared from 180 °C, isothermal annealing conditioning was conducted for different time (0 s, 300 s, 600 s, 1800 s and 3600 s) to capture the phase separation process in the unstable PMBs. To minimise the orientation effects because of sample flowing during the spreading and conditioning, the observation was focusing on the centre areas of the slides. For more details on the experimental procedure, please refer to [17]. Although [17] focused on exploring the driving forces for the phase separation in PMB, this paper presents a new approach to PMB microstructure evaluation. To investigate the sensitivity of the new approach on evaluating the effects of temperature, the microscopy observation results of samples from 160 °C and 120 °C [33,34] are studied in this paper. They were the observation results of the same four PMBs after 1 h of isothermal annealing conditioning at the specified temperatures following the same experimental procedure.

Furthermore, numerical images from simulations are also analysed in this study. A previous paper by the authors [35] presented the used phase-field model for the phase separation in PMB and numerically reproduced the microscopy observation results of the same PMBs as in this paper during the entire phase separation process. However, the assessment of numerical reproduction on the basis of the experimental results remained an issue in spite of the visual similarity, i.e. how to compare a numerical simulation with respect to the experimental results. In this study, the experimental and numerical PMB microstructure images are processed and analysed with the same method. The computed derivative parameters of the experimental and numerical images are compared and discussed for numerical reproduction assessment.

2.2.2. Image analysis using 2D fast Fourier transform

Image analysis is the extraction of physical information from an image. In terms of microstructure characterisation, the 2D-FFT processing of microscopic images has been a widely-used method for reciprocal space image analysis of metal and polymer systems [36–42], although its use was hardly ever reported for PMBs. With this method, a microstructure is usually characterised by its 2D-FFT power spectrum. A 2D-FFT power spectrum in the reciprocal space (k space), which is

Table 1
Polymer-modified bitumen properties.

Property	Method	PMB1	PMB2	PMB3	PMB4
Penetration, 25 °C (0.1 mm)	EN 1426	57	52	56	52
Softening point, ring & ball (°C)	EN 1427	76.0	85.0	77.8	65.0
Penetration index	EN 12591	4.1	5.1	4.3	2.1
180 °C storage stability, softening point difference (°C)	EN 13399	25.5	0.0	−1.0	32.0