



Historical perspective

Surface microstructure of bitumen characterized by atomic force microscopy

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ABSTRACT

Bitumen, also called asphalt binder, plays important roles in many industrial applications. It is used as the primary binding agent in asphalt concrete, as a key component in damping systems such as rubber, and as an indispensable additive in paint and ink. Consisting of a large number of hydrocarbons of different sizes and polarities, together with heteroatoms and traces of metals, bitumen displays rich surface microstructures that affect its rheological properties. This paper reviews the current understanding of bitumen's surface microstructures characterized by Atomic Force Microscopy (AFM). Microstructures of bitumen develop to different forms depending on crude oil source, thermal history, and sample preparation method. While some bitumens display surface microstructures with fine domains, flake-like domains, and dendrite structuring, 'bee-structures' with wavy patterns several micrometers in diameter and tens of nanometers in height are commonly seen in other binders. Controversy exists regarding the chemical origin of the 'bee-structures', which has been related to the asphaltene fraction, the metal content, or the crystallizing waxes in bitumen. The rich chemistry of bitumen can result in complicated intermolecular associations such as coprecipitation of wax and metalloporphyrins in asphaltenes. Therefore, it is the molecular interactions among the different chemical components in bitumen, rather than a single chemical fraction, that are responsible for the evolution of bitumen's diverse microstructures, including the 'bee-structures'. Mechanisms such as curvature elasticity and surface wrinkling that explain the rippled structures observed in polymer crystals might be responsible for the formation of 'bee-structures' in bitumen. Despite the progress made on morphological characterization of bitumen using AFM, the fundamental question whether the microstructures observed on bitumen surfaces represent its bulk structure remains to be addressed. In addition, critical technical challenges associated with AFM characterization of bitumen surface structures are discussed, with possible solutions recommended. For future work, combining AFM with other chemical analysis tools that can generate comparable high resolution to AFM would provide an avenue to linking bitumen's chemistry to its microscopic morphological and mechanical properties and consequently benefit the efforts of developing structure-related models for bituminous materials across the different length scales.

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1. Introduction

Bitumen, also called asphalt binder, comes from the process of fractional distillation of petroleum (Considering the popularity of the words *bitumen* and *asphalt binder* in Europe and North America, respectively, these two words are used interchangeably throughout this paper.). Bitumen has been used in roof shingles for waterproofing, in cars and computers for vibration mitigation, and in paintings and inks as an additive for durability improvement. In addition, it has been widely used in road construction since ancient times (i.e., King of Babylon, 625–604 BC) [1]. At the present time, 93% of more than 2.6 million miles of paved roads in United States are surfaced with bitumen. In the paving industry, bitumen acts as a glue binding the aggregates and filler particles together to form asphalt concrete. The efforts of producing more sustainable asphalt pavements are motivated by economic and environmental considerations (i.e., the increasing cost and the depletion of the non-renewable petroleum resources). Back in 1987, the American Association of State Highway and Transportation Officials initiated a five-year research program entitled ‘The Strategic Highway Research Program (SHRP)’, with one of its units focusing on developing performance-related specifications for asphalt binder and asphalt concrete. A profound advance from this project was the development of a set of performance-based testing methods to characterize the rheology, fatigue, and thermal cracking of asphalt binders. However, due to the complex chemistry of asphalt binder and the limited available techniques for microscopic investigation [2], the fundamental question of how the variations in chemical compositions of bitumen affect the overall performance of asphalt pavements remains unanswered.

Asphalt concrete is often considered as a composite consisting of constituents with lengths ranging from nano to macro scales, as shown in Fig. 1. Continuum mechanics, together with laboratory and field tests at the pavement length scale, are often used to predict the overall performance and in-service life of asphalt pavements. However, material properties at the micro and nano scales are often needed for macroscopic investigations. For example, the rheological property of bitumen, which contributes significantly to the final performance of asphalt pavements, is proven to be related to bitumen structures at the micro and nano scales [1].

Bitumen is a complex mixture of mostly hydrocarbons (greater than 90 wt.%) with different size and polarity, together with some heteroatoms such as sulfur, nitrogen, oxygen, as well as traces of metals (e.g., iron, nickel, and vanadium). Moreover, chemical compositions of bitumen vary with their crude sources. In order to better understand bitumen's chemistry and its rheological behavior, different methods have been developed to separate the molecules in bitumen into different chemical fractions, depending on their molecular size and polarity. Among these separation methods, SARA separation (saturates, aromatics, resins, and asphaltenes, with increasing molecular weight and polarity, as shown in Fig. 2) is frequently used [4]. Asphaltenes are commonly defined as the crude-oil fraction that is soluble in aromatic solvents (e.g., toluene or benzene) but insoluble in aliphatic solvents (e.g., n-heptane, or n-pentane). Fig. 3 shows the typical molecular structures of asphaltenes [5], which are typically large polycyclic-aromatic unit sheets containing heteroatoms, dipoles, and short aliphatic side groups [6]. Asphaltenes are the heaviest component in bitumen, and they have a strong tendency to aggregate due to the attraction between polyaromatic fused rings and precipitate out from an initially stable

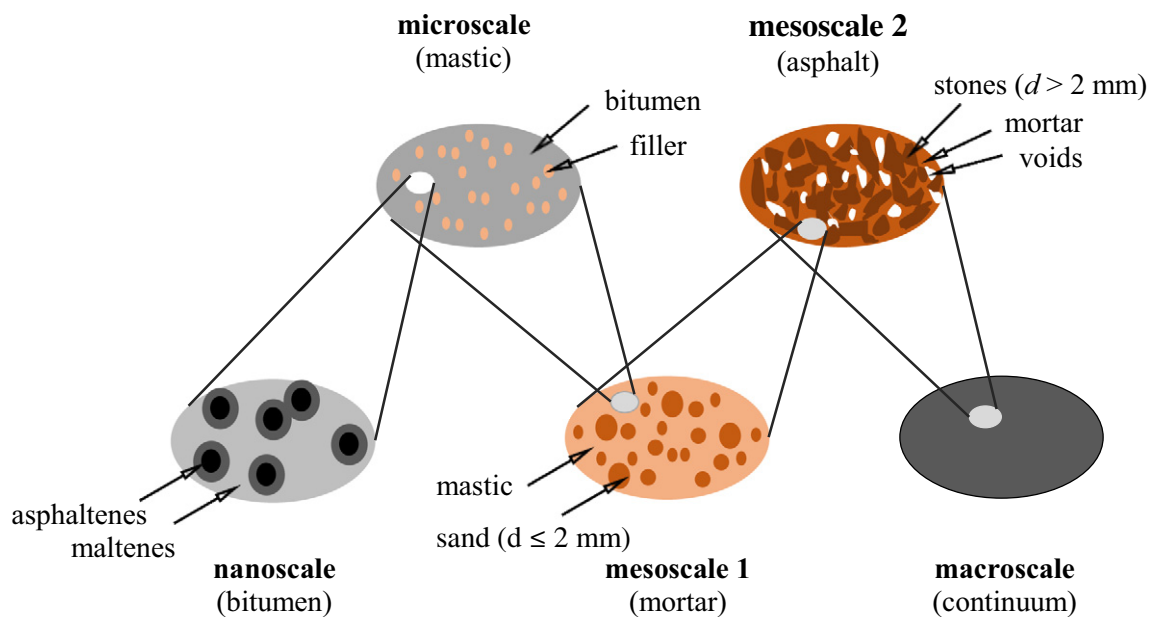


Fig. 1. Multiscale model for asphalt mixtures, including the bitumen-scale, the mastic-scale (bitumen + filler), the mortar-scale (mastic + fine aggregate with $d < 2$ mm), the asphalt-scale (mortar + aggregates with $d > 2$ mm), and the macroscale [after 3].

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