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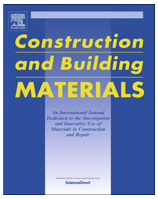

Investigation on the influences of moisture on asphalts' micro properties by using atomic force microscopy and Fourier transform infrared spectroscopy

Minghui Gong [a,b,c], Zeheng Yao [c], Zijia Xiong [a,b], Jun Yang [c], Jinxiang Hong [a,b,*]

[a] *State Key Laboratory of High Performance Civil Engineering Materials, 118 Liquan Road, Nanjing 211103, PR China*
[b] *Jiangsu Sobute New Materials Co., Ltd, 118 Liquan Road, Nanjing 211103, PR China*
[c] *School of Transportation, Southeast University, 2 Sipailou, Nanjing 210096, PR China*

HIGHLIGHTS

- Interactions between asphalt and moisture is investigated t micro scale.
- AFM and FTIR are used to investigate the mechanism of asphalt property changes during water immersion.
- Formation of microstructures in asphalts from same crude oil are closely related to long chain index.
- Polar components tend to interact with polar water molecules.

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ABSTRACT

Plenty work has been done on asphalt's moisture susceptibility at macro scales. However, limited work was done at micro scale to aid in the understanding of interactions between asphalt and moisture. This paper uses atomic force microscopy (AFM) and Fourier transform infrared spectroscopy (FTIR) to investigate the mechanism of asphalt property changes during water immersion. Six asphalt samples from same crude oils sources are prepared. Wet-conditioned samples were fabricated by immersing dry samples in water for 72 h. Samples' micro mechanical properties were obtained by AFM mechanical tests. Chemical alterations were detected by FTIR test. Results from AFM and FTIR tests are compared and analyzed. It is found that formation of asphalt microstructure is closely related to long chain index. Both microstructure and long chain index are less susceptible to water immersion when compared to matrix and other functional group indexes. AFM mechanical results show reasonable connections to FTIR results which indicates that polar components tend to interact with polar water molecules.

1. Introduction

Asphalt is a complex organic material which contains aromatic structures, aliphatic structures and oxygenated functional groups [1]. As a vital component in pavement construction, its viscoelasticity makes asphalt mixture exhibit superb pavement performance. However, asphalt pavement's performance will degrade under repeated loading coupled with various environmental effects (temperature changes, moisture and ultraviolet radiation). Moisture is considered as a main factor which leads to the deterioration of pavement structures [2]. Existence of moisture would induce the loss of adhesive bond within the aggregate and asphalt interface or the loss of cohesive bond within asphalt binder. In fact, rutting and cracking would be exacerbated with the involvement of moisture [3]. Thus, it is necessary to investigate the moisture susceptibility of asphalt materials. However, existing specification only provides boiling water stripping test and freeze–thaw indirect tensile strength test to evaluate the moisture susceptibility of asphalt-aggregate system while few specific tests are designed to obtain material (asphalt/aggregate) properties that can aid in designing mixture with better anti-moisture ability [4]. In boiling water stripping test, the moisture sensitivity is determined by the visual inspection on the striping condition of aggregate coating after water immersion. The result here only provides a qualitative evaluation of loose mixture's anti-moisture ability. Unlike boiling water striping test, freeze–thaw indirect tensile strength test uses

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* Corresponding author at: State Key Laboratory of High Performance Civil Engineering Materials, 118 Liquan Road, Nanjing 211103, PR China.

E-mail address: gongminghui@cnjsjk.cn (M. Gong).

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the ratio of mechanical properties between unconditioned and conditioned samples to determine the moisture sensitivity [5]. In order to better understand the effects of moisture on the adhesion and cohesion failures in asphalt concrete, pull-off test is proposed to quantitatively characterize the moisture susceptibility [6,7]. However, aforementioned methods merely use static loading while actual loading applied on asphalt pavement is dynamic. Thus, Hamburg wheel-tracking test is designed to simulate traffic conditions on compacted specimens immersed in water. During samples' plastic deformation, the presence of a sudden inflection point is identified as striping.

Caro summarized that methods testing asphalt mixtures' moisture susceptibility can be divided into several categories by following factors: (a) states of mixture; (b) mode of loading; (c) methodology used to induce moisture damage and (d) scale of performance measurements. Since lots of well-organized works have been carried out by considering the former three factors, researchers are making efforts to look into asphalt concrete's moisture susceptibility by multi-scale methods, e.g. atomic force microscopy (AFM), surface free energy method, nanoindentation test and freeze–thaw splitting test [8–12]. Among them, AFM is a unique way to investigate the moisture performance of asphalt binder at microscale. The interaction between AFM tip and asphalt binder can be used to simulate the bond within the aggregate and asphalt interface due to the similar atom constitution of aggregate with the silicon–nitride AFM tip [13]. The ratio of adhesive forces between dry and water-immersed samples can be used to evaluate the moisture susceptibility of asphalt at nanoscale [14]. The changes of asphalt microstructures on sample surface can reveal the mechanism of the interactions between asphalt and water. All these together make AFM a widely used method in asphalt research in recent years. Besides AFM, Fourier transform infrared spectroscopy (FTIR) was also found to be useful for providing information of asphalt molecule changes during moisture immersion. In Hung's work, FTIR result shows that water exposure induces changes in chemical composition on the surface of bitumen, which correlates well with AFM results [15]. However, only AFM morphology results were analyzed in related research. No work was done by comparing FTIR results with AFM mechanical results, which may give more information about asphalt's composition-mechanical relationships.

In our previous work, four asphalts from different crude oils were selected, and AFM was used together with FTIR to investigate the relationship between these four asphalts' chemical compositions and microstructures (bee structure namely) [1]. However, it was found that long chain index (representing the content of solid wax crystal) derived from FTIR spectrum showed no connections to the formation of bee structures. Nevertheless, this conclusion was only valid for asphalts from different crude oils and more work need to be done on asphalts from same crude oils.

Accordingly, present paper prepared six kinds of asphalts from same crude oils. AFM was adapted to characterize both the morphological and mechanical properties on the sample surface. Functional group indexes, long chain index especially, were obtained through FTIR. The connections between AFM and FTIR results were built to provide an insight into the composition-microstructure-mechanical properties relationship.

2. Objectives

The main objective of present paper is to investigate the influence of moisture on asphalts' micro properties using AFM and FTIR. Both AFM mechanical and morphological results of dry and water-immersed asphalt samples were obtained. Connections between micromechanical properties and functional groups were established through regression method. The effects of functional groups on asphalts' moisture susceptibility was also analyzed. Additionally, the relationship between FTIR long chain index and the formation of bee structures was examined to validate the findings in our previous study.

The specific contents are listed as follows:

1. Six asphalts from same crude oils with different Penetrations (Pen10, Pen30, Pen50, Pen70, Pen90 and Pen110 asphalts respectively) were prepared and then were used to fabricate the control asphalt samples. Wet-conditioned asphalt samples were prepared by immersing asphalt samples in water for 3 days (72 h) at 25 °C. Afterwards, these samples were dried at ambient temperatures in a sample box for 24 h. AFM was utilized to map the surface morphology of the control and wet-conditioned asphalt samples. Force curves were obtained and mechanical properties (modulus and adhesive force) were derived from these curves. The moisture susceptibility of different asphalts were evaluated by calculating the ratio of adhesive forces between dry and water-immersed samples.
2. FTIR test was conducted to collect the spectra of all the asphalt samples and functional group indexes were calculated. Solid wax-crystal content is characterized by long chain indexin FTIR test because of our lab limit in direct measure of wax content. The bee structure content (represented by AFM roughness) were compared to long chain index to analyze the formation mechanism of bee structures.
3. Ratio of adhesive forces and functional group indexes between wet-conditioned and dry samples were analyzed together to establish a relationship between asphalts' chemical compositions and micro moisture susceptibility. Related findings would reveal the moisture damage mechanism from microscopic view. Relationship between asphalt's chemical properties and its moisture related mechanical properties was given as well.

3. Materials and experiments

3.1. Materials

3.1.1. Asphalt binder

Pen10, Pen70 and Pen110 asphalts were obtained from China National offshore oil Corporation (CNOOC). Information about the sample preparation process is demonstrated in Fig. 1. As shown in Fig. 1, Pen10 asphalt was produced from Pen70 asphalt by compressed air oxidation method. Pen110 asphalt was obtained by adding second vacuum gas oil into Pen70 asphalt. Pen30 and Pen50 asphalts were prepared by blending Pen10 asphalt with Pen70 asphalts at different proportions. Pen90 asphalt was pre-

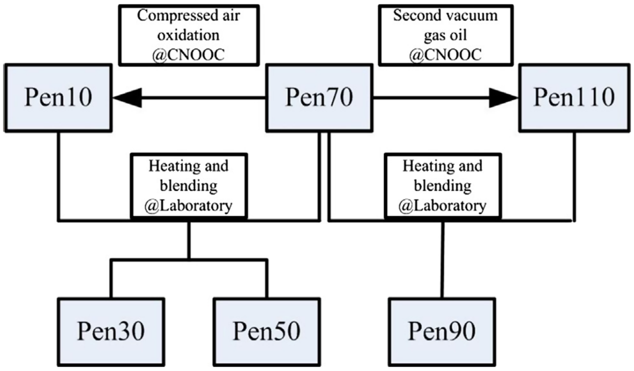

Fig. 1. Preparation process of asphalt binders.