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Investigating bitumen rejuvenation mechanisms using a coupled rheometry-morphology characterization approach

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HIGHLIGHTS

- Rheological properties of aged bitumen can be restored using a bio-rejuvenator.
- One can obtain good estimate of required rejuvenator content through chemo-mechanical metrics.
- An optimum rejuvenator dosage can be assigned to restore target bitumen properties.
- The "bee" length has a moderating effect on the stiffness and viscosity relationship.

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ABSTRACT

The reuse of oxidized bitumen offers a viable solution to enhancing pavements' sustainability and reducing consumption of virgin bitumen. As such use of reclaimed asphalt pavement (RAP) and recycled asphalt shingles (RAS) containing approximately 5 and 20% bitumen, respectively has been promoted by both road authorities and the pavement industry. However, due to highly oxidized nature of aforementioned bitumen, its incorporation into new asphalt pavements can negatively impact their performance and lead to premature cracking. Therefore, restoring properties of oxidized bitumen is critical to render it as a viable source. This in turn, poses an intriguing scientific inquiry pertaining the definition and means of restoration. This paper defines a chemo-mechanical metrics for measuring extent of restoration while demonstrating the use of a bio-oil produced from swine manure as a cost-effective additive for rejuvenating aged bitumen.

Bitumen used in this study includes both laboratory-aged bitumen and those extracted directly from field recovered samples. Characterization of the aged bitumen before and after rejuvenation was conducted by Rheometry, and Atomic Force Microscopy (AFM). The results of experiments showed that addition of aforementioned bio-oil referred to as bio-rejuvenator (BR) in this study significantly restored bitumen properties. In addition, correlation between size of morphological features referred to as "bees" and rheological properties suggests that "bee" length has a moderating effect on the relationship between bitumen's stiffness and viscosity.

1. Introduction

Bitumen is among the most commonly used materials for road construction in the United States, covering more than 90 percent of the nation's highways (REF). However, according to the [14], asphalt binder or so called bitumen prices have increased over 74% from $235 per ton in 2006 to $409 per ton in 2017 [14]. The increase of the price has been linked to refineries' decreasing their bitumen production, while the demand is increasing. Implementing coking technologies, refineries have managed to reduce the asphalt and road oil yield residue of crude oil from 3.4% in 2000 to 1.9% in 2016 [15,33]. In fact, aforementioned technological advances in refineries allows for extracting more high value products from crude oil leaving less residue behind [32,26]. The increase of price combined with overall global concern about depleting natural resources, led the science community and industry to focus on restoring properties of aged bitumen to allow for its

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reuse. It should be emphasized that reuse refers to consuming the reclaimed materials (aged bitumen) for the same purpose as the virgin bitumen would be used for which is in contrast to recycling in which the aged bitumen from surface course is demoted and used in lower pavement layers and base courses.

The two major sources for aged bitumen are reclaimed asphalt pavement (RAP) and recycled asphalt shingles (RAS). According to a survey conducted in 2014 by the National Asphalt Pavement Association (NAPA), the use of RAP and RAS replaced approximately 5.8 million barrels of asphalt binder and 68 million tons of virgin aggregate resulting in a cost savings of over $2.8 billion [17].

Even though both environmental and economic benefits could be obtained from increased usage of RAP in new pavement, the average RAP content used in new asphalt pavement layers is only 20.4% [17]. The concerns over plausible premature pavement cracking due to the presence of highly oxidized bitumen in RAP discourages contractors from using higher percentages of RAP. It has been documented that aged binders are stiffer, less strain-tolerant, and more susceptible to cracking than virgin material [18,24,13,29,12]. Accordingly, effective restoration of aged bitumen properties can facilitate use of RAP and RAS leading to significant environmental and economic benefits.

Most researchers have studied rejuvenation via rheology-based metrics. It should be noted that oxidative aging causes morphological changes in bitumen which may not be detectable via rheometry. Presence of micrometer-sized bee-like structures in bitumen has been documented utilizing atomic force microscopy (AFM) [37,20,34,22]. These "bee" structures were observed to be larger in aged bitumen compare to those in virgin bitumen [35,23]. It has been also reported that the number of bees decrease when subjected to aging [40,27]. Yu et al. also found that the bee structures for unmodified binder became more elongated after aging using a pressure aging vessel [38].

Several study explored effects of various rejuvenators on bitumen's mechanical properties and reported that all rejuvenators were effective in enhancing mechanical properties but the "bee" structures were not always restored to their unaged form [27,38]. Therefore, a change in mechanical properties does not necessarily correlate with morphological restoration, and there is still significant controversy about true aging and rejuvenation mechanisms. A better understanding of these mechanisms is important for the development of more effective rejuvenators.

In addition, most of commonly used rejuvenators are petroleum-based, in an attempt to create bio-based rejuvenators, several researchers examined effect of several bio-oils on promoting mechanical properties of aged bitumen [21,39,11,19,25,28,36]. However, most of these studies mainly use wood-based bio-oils, and use restoration of mechanical properties as the sole indicator for rejuvenation. This paper investigates efficacy of a bio-based rejuvenator derived from swine manure while using a combination of morphological and rheological properties as metrics for rejuvenation.

2. Materials and methods

2.1. Materials

The virgin bitumen used in this study was a Superpave PG 64–22, which is one of the most commonly used binders across the U. S. The binder was acquired through Sharpe Brothers in Greensboro, NC. The aged bitumen was provided via extraction from the field samples as well as aging bitumen in the laboratory. Field samples were from a milling project in Michigan. Laboratory aged samples were prepared using extended pressure aging at 100 °C, which has shown to correlate well with field aged samples [10]. Bio- rejuvenator BR) used in this study was derived from swine manure as described elsewhere [16].

For the sake of brevity, the laboratory aged binder is referred to as "2PAV" and the field-extracted RAP binder is referred to as "RAP."

2.2. Bitumen aging method

To prepare aged bitumen in the laboratory, a two stage aging including a short term and long term aging was use. The short term aging was done using a Rolling Thin Film Oven (RTFO) following [6]. To age bitumen, 35 g of each sample was placed into each RTFO aging bottle and heated to 163 °C for 85 min (1.3 h) while being subjected to a continuous airflow of 4 L/min. This aging procedure simulates asphalt binder that has undergone aging while being mixed with aggregates at an asphalt-mixing plant and placed in the field.

The sample was then exposed to long–term aging utilizing pressure aging vessel (PAV) following [4]. To do so, 50 g of each sample undergone short term aging was placed onto steel plate, and subjected to a pressure of 2.10 MPa at 100 °C for 40 h. Samples were then degassed using the vacuum oven at 170 °C for 30 min 0.5 h) at 15 kPa to remove air bubbles entrapped by the PAV aging. The aging time was selected based on the recommendation by [10] who showed such aging would adequately represent aging level of an asphalt pavement at the end of its service life.

2.3. Sample preparation

To prepare samples, a select dosage of bio-rejuvenator (BR) ranging from 5 to 30% was introduced to each of laboratory-aged (2PAV) and field-aged bitumen (RAP). Bitumen was heated to 135 °C for 30 mins; BR was added to bitumen and blended for 5 min at 150 rpm. Each sample was then stored in a sealed container to prevent contamination.

2.4. Chemical characterization methods

Elemental analysis of the asphalt binder was obtained using a Perkin-Elmer 2400 CHN/S analyzer. Attenuated total reflectance mode Fourier transform infrared spectroscopy (ATR-FTIR) was performed on a Cary 670 FTIR (Agilent Technologies) using a diamond ATR crystal. FTIR spectra were normalized to the area of the spectral bonds between 600 and 2000 cm^{-1}.

2.5. Thermo-mechanical methods

Viscosity measurements were conducted using the Brookfield RV-DVIII Ultra rotational viscometer, following [7]. The viscoelastic moduli results were determined using the Malvern Kinexus dynamic shear rheometer following ASTM D7552-09. Stiffness and m-value at low temperature were determined for each specimen using a Cannon Thermoelectric Bending Beam Rheometer (TE-BBR), following [8]. The Interlaken Direct Tension System (DT) was used to determine both peak load and failure strain (fracture point) low temperature, following [5].

2.6. Surface characterization methods

The Atomic Force Microscopy (AFM) was performed using a Keysight Technologies 5600LS. The cantilever was silicon with an aluminum reflex coating (TAP300, Budget Sensors), with a resonant frequency of 300 kHz and a force constant of 40 N/m. The line scan rate for the image was 0.96 lines/s in tapping mode, with an image resolution of 512 × 512. To prepare specimen, bitumen