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A novel approach to evaluate fracture surfaces of aged and rejuvenator-restored asphalt using cryo-SEM and image analysis techniques

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HIGHLIGHTS

- Low-temperature cracking potential was evaluated using Cryo-SEM and image analysis techniques.
- Cryo-SEM and image analysis are identified as promising techniques in monitoring the fracture surface of aged and rejuvenator-restored binders at low temperatures.
- Rejuvenated binder samples indicated less stiffness at lower temperatures compared to the aged binder samples as the proposed Fracture Index (FI) values confirmed.
- The Fourier Transform Infrared Spectroscopy (FTIR) results were consistent with results obtained from Cryo-SEM ranking method.

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ABSTRACT

Low temperature cracking potential is a primary concern for asphalt mixtures with high RAP contents. To minimize the thermal cracking, various rejuvenators have been utilized in the past. In this study, Fourier Transform Infrared Spectroscopy (FTIR) was used to determine the level of oxidation of asphalt. To evaluate the low-temperature cracking potential of aged asphalt with and without rejuvenators, the fracture surface images were captured using Cryo-Scanning Electron Microscopy (Cryo-SEM). The Cryo-SEM images were then analyzed using the image processing technique. The rejuvenated asphalt specimens exhibited less fractured surface than the aged asphalt. Finally, the Fracture Index was computed to rank each asphalt sample in terms of low-temperature cracking potential. Based on the test results, it can be concluded that the use of the Cryo-SEM technology combined with the image processing technique is a promising method to visually observe the fracture surface of asphalt and compute the Fracture Index to estimate the low-temperature cracking susceptibility.

1. Introduction

The use of high Recycled Asphalt Pavement (RAP) mixes is increasing due to both environmental and economical reasons. However, a high RAP mix can cause asphalt mixtures to stiffen due to the hardening effect of the aged asphalt in RAP materials. During mixing, transportation and construction, the short term aging of asphalt occurs leading to an increase in a large molecular size, viscosity and stiffness [13]. The long-term aging of asphalt is affected by the steric hardening through molecular reorganization, volatilization and oxidation generated by changes in molecular structure [8]. Through the aging process, maltenes change to asphaltenes, which makes the asphalt more viscous [5,17]. Further, the aging makes the asphalt stiffer by removing the oily phase and subsequently increasing the ratio of asphaltenes to aromatics. As a result, the aged asphalt would become harder and more viscous

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but less ductile, which could negatively affect the performance of the high RAP mixes in the field especially at lower temperatures.

Low temperature cracking potential is a primary concern with high RAP mixtures. To minimize the low temperature cracking, various rejuvenators have been utilized in the past instead of bumping down a PG grade of the specified virgin asphalt for high RAP mixes. However, in some cases, the premature failures such as rutting have been observed from the high RAP mixtures with rejuvenators.

Rejuvenators may be made from lubricating oil extract and extender oils, which are great sources of maltenes. The rejuvenators mechanism of working in high RAP mixtures could be classified as these four provided steps [1]:

1) Forming a very low viscosity layer surrounding the RAP by a rejuvenator.
2) The rejuvenator begins to penetrate into the aged asphalt and makes it softer.
3) Penetration continues as the inner layer viscosity decreases whereas the outer layer viscosity increases.
4) Balance in viscosity is reached after a certain amount of time.

A number of studies have been carried out in the past to assess the effect of rejuvenators on the aged asphalt extracted from RAP mixtures. The rejuvenators increased the resistance to low temperature cracking, but decreased the resistance to rutting [20,11]. The effect of waste engine oil on the properties of aged asphalt was investigated to confirm the softening effect of engine oil. However, the engine oil decreased the compressive and tensile strength resulting in the increased rut depth [6]. Rejuvenators diffusion into the asphalt can be improved by increasing the temperature and time duration. The level of rejuvenator diffusion was quantified by measuring the penetration and chemical components of rejuvenator-coated aged asphalt [7]. An innovative idea of using encapsulated rejuvenators was presented, which will break and release rejuvenator when the stress applied on those capsules exceeds their strength [9]. Based on the study that evaluated various rejuvenators including aromatic extract, waste engine oil, waste vegetable oil, organic oil, waste vegetable grease, and distilled tall oil, the rejuvenators decreased both high and low PG temperature of the aged asphalt [22]. Incorporating proper dosage of rejuvenators was claimed to be effective in reducing the total cost of asphalt mixture production. However, long term performance of rejuvenated mixes should be investigated to ensure cost efficiency during the service life of rejuvenated high-RAP mixtures [20].

Various microscopic technologies have previously been used to examine the structure of modified asphalt. Among them, SEM has been employed in a number of studies to evaluate the physical properties of modified asphalt. However, it has been reported that monitoring microstructural properties of different types of asphalt is difficult using conventional SEM technology mainly due to the volatility of oily part of asphalt and its sensitivity to the illuminated electron beam. This problem restricted the resolving power available to probe asphalt microstructure and brought about limited magnification. To resolve this problem, Cryo-preparation equipment was introduced to make a unique observation by rapidly decreasing the sample temperature using liquid nitrogen. The combined Cryo-SEM technique has been used successfully to evaluate the surface texture characteristics of Crumb Rubber Modified (CRM) asphalt and the effect of digestion time on CRM asphalt properties. Authors reported that the Cryo-SEM analysis allowed the identification of the interaction between crumb rubber and asphalt to define the proper digestion time for the production of asphalt rubber binder [19]. A higher-resolution Field Emission Gun (FEG) Cryo-SEM has been used to examine the physical properties of polymerized asphalts. More precisely, the distribution of polymers nanoparticles throughout the bitumen matrix was followed using high-resolution Cryo-SEM technology. A "honeycomb structure" throughout the polymerized asphalt was captured as well as the nodules of polymers dispersed within the asphalt [21]. The same procedure was used to visualize and investigate polymer nodules and fracture morphologies in EVA, SB and SBS modified asphalt. Polymer dispersion of all prepared modified binders were imaged and compared at a submicrometer scale [2]. However, for years, there has not been any method aims to visualize the physical properties of asphalt and to examine how cracks would be developed throughout the asphalt at lower temperatures. This article aims to cover this gap by employing the Cryo-SEM technique which has been mainly used to monitor the structure of polymers in modified asphalt.

On the other hand, Fourier Transform Infrared Spectroscopy (FTIR) has been widely used to determine the level of oxidation due to aging. Basically, it can be deduced which chemical groups exist in the sample by examining the infrared absorption peak position, number, shape and relative strength since each functional group has a specific infrared absorption peak. To quantitatively analyze the level of asphalt aging, the obtained transmission spectrum of asphalt sample should be converted to the absorption spectrum. To evaluate the aging degree, the area under the spectrum curve related to carbonyl ($C{=}O$), sulfoxide ($S{=}O$) and saturated $C{-}H$ are then calculated. Previous studies reported that both sulfoxide and carbonyl compounds, which are produced due to aging, decreased when rejuvenators were added to the aged asphalt [3,4]. With the help of FTIR technology, any chemical changes of asphalt after aging can be evaluated; any restored properties of aged asphalt after incorporating rejuvenators can also be assessed by comparing the FTIR results for both virgin and rejuvenator-restored asphalt. In another word, the fact that how rejuvenators are capable of restoring the original chemical properties of aged asphalt can be investigated using FTIR technology. Using FTIR technology, various types of rejuvenators were investigated in terms of their effects on restoring the chemical composition of aged binders [14,3,4,16]. Karlsson used FTIR-ATR (Attenuated Total Reflectance) to assess the influence of the degree of aging on bitumen rejuvenator diffusion [12]. As a previously-proven method, FTIR is used in this study to figure out the level of consistency between its results and the results of the proposed method (Cryo-SEM).

The main objective of this research is to evaluate fracture surface properties of rejuvenator-restored aged asphalt without changing the original physiochemical characteristics. The Cryo process has been used to counteract the negative effect of asphalt oily phase. Digital image processing technique was then used to quantify cracks developed on the fractured surface due to the aging process. The results will then be compared to FTIR results to examine the consistency.

2. Materials

To prepare asphalt samples and in an effort to rejuvenate the aged asphalt, two types of commercial rejuvenators were added into the asphalt: 1) Rejuvenator "A" (petroleum oil) which is a petroleum oil with high viscosity at room temperature, 2) Rejuvenator "B" produced from refined tall oil. The properties of rejuvenators A and B are presented in Tables 1 and 2, respectively. To simulate the short-term aging condition the RTFO test (AASHTO T240-94) was initially performed on virgin PG64-22 asphalt. The short-term aged asphalt was then subjected to PAV test (AASHTO R28) to simulate a long-term aging condition. As suggested by manufacturers, the