



Full Length Article

Comparison of ESEM and physical properties of virgin and laboratory aged asphalt binders

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ABSTRACT

The physical and microstructural properties of four straight run asphalt binders were examined and compared in combination with short term aging (RTFOT) and long-term (PAV) laboratory aging. RTFOT aging was conducted at temperatures of 123, 143 and 163 °C. The physical testing parameters included penetration, softening point and Dynamic Shear Rheology (DSR) complex shear modulus and phase angle at 10 °C. The binders selected came from four different sources and had the same penetration grading (70/100). They all showed an increase in stiffness with aging, including with the increase in RTFOT temperatures and especially with PAV aging. The microstructural evolution of the binder was examined by Environmental Scanning Electron Microscopy (ESEM) on aged binders at 123 and 163 °C. The physical changes with aging corresponded to an evolution in the binders' 'fibril' microstructure under ESEM, as a result of electron beam exposure, with the microstructure getting denser with PAV aging. This densification (fibril area) of the microstructure was quantified with image analysis for the virgin and RTFOT aged samples, and the fibril formation time was also measured. The asphalt binders showed varied ESEM 'fingerprints' and aged in different ways. The ESEM 'formation time' and 'fibril area' of the binders generally showed good correlation with the physical properties, although this was not the case for all of the binders due to their unique aging characteristics.

1. Introduction

Despite the widespread use of asphalt binders, there remains a lot to be understood about its nature [1]. Although asphalt binder (bitumen) only represents around 5% of the mass of typical hot mix asphalt mixtures, it plays a key role in determining the behaviour and the performance of the mixture and has a significant impact on the performance of the pavement structure [2]. Furthermore, it is the binder that undergoes physical and chemical aging during the pavement service life [3], making its further understanding of vital importance.

A promising but not yet well understood technique for observing the nature of asphalt binder is Environmental Scanning Electron Microscopy (ESEM) [4]. ESEM is designed to study wet, and oily materials as it allows observations of such materials in their natural state

without de-oiling procedures required for the vacuum environment of a conventional Scanning Electron Microscope (SEM). The image obtained by the ESEM is a result of the interaction of the electron beam with the sample at an atomic level. The secondary electrons that are used in this study are emitted by the sample due to the interaction with the primary electron beam and they represent the sample topography. The volume of interaction between the electron beam and the sample is more at the surface resulting in more electrons escaping from the peaks than from the valleys resulting in peaks being brighter and valleys dark [5]. When an asphalt binder sample is placed in the ESEM, the subjection of the sample to an electron beam (in secondary electron mode), causes it to form 'fibril' microstructures that eventually stabilize. The parameters retrieved from this could be i) the density of the fibril + microstructure [6], ii) the size and shape of the fibrils [7] and iii) the amount of time

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the structure takes to form them and stabilize [8].

There have been a few studies on asphalt binder with this technique that have confirmed the formation of the fibril microstructure [7]. Initially, researchers assumed that the structures developed as a result of electron exposure was related to the heaviest molecules in the binder (i.e. asphaltenes). It has since been proposed that they correspond to a part of the maltenes (intermediate to light molecular weight) fraction and possibly a part of the asphaltenes [9]. The microstructure has been found to evolve with both binder aging and tensile forces [4]. Furthermore, mixes of virgin and aged binders have been shown to produce hybrid fibril microstructures in ESEM, sharing the properties of both parent binders [10]. The microstructure has also been found to form at observation temperatures as low as -80°C [11].

There are still questions regarding the reasons why the fibril microstructure forms and what it represents. A similar phenomenon has been observed where a non-conductive organic material is bombarded with electrons [12]. In its conventional use, an SEM would be used on a conductive material or one with a conductive coating, where the electrons (energy) sent to the material would mostly pass through the sample [13]. Asphalt binders are too viscous to be treated with the coating successfully [14]. When the binder is exposed to the electron beam, inelastic collisions between the materials occur and the energy stays inside the sample [15]. From the understanding of this phenomenon in other organic, viscous and volatile materials observed with the electron beam, it is known that one of the effects of this is a significant amount of local heating being generated [16]. Another is the addition to the dispersion of certain molecules like aliphatics, which would be less rigid than the aromatic parts of the binder [17]. A study looking at asphalt binder in this context found a slightly higher aliphatic signal in the area where the irradiation occurred, but a stronger aromatics presence on the fibrils themselves [11].

Despite some very interesting results, the relation of the ESEM findings to asphalt binder physical performance and aging needs to be further understood. This study is part of a larger inter-laboratory study of the RILEM Technical Committee 252 CMB. The virgin and aged binders were subject to both physical testing (Penetration, Softening Point (R&B), Rheology (DSR)) and chemical testing using FTIR performed by ten laboratories in five countries [18]. The objective of the current paper is to analyse these same four straight run asphalt binders that have the same penetration grade, and the evolution of their microstructures after short term aging (RTFOT) and subsequent long-term (PAV) laboratory aging. The ESEM analysis was performed by the University of Waterloo and the binder samples were aged by Empa. This study will allow the authors to: i) validate previous findings on the evolution of aged binder under ESEM, ii) understand the differences of straight run binders from different sources under ESEM and iii) attempt to find a correlation between the physical properties of the binder (penetration, softening point, rheology) and the findings of the ESEM analysis. Overall, this would allow for the validation of the ESEM findings and a better understanding of how they can be interpreted, ultimately giving us further insight into the nature of asphalt binder.

2. Materials and methods

2.1. Materials

2.1.1. Asphalt binders

Four 70/100 penetration specified asphalt binders (EN 12591) from different crude sources were used in this study and identified as B501, B502, B503 and B504. Table 1 provides the properties of the four binder samples according to European Standards (EN 1426, 1427) and the US Performance Grade Specifications (AASHTO M 320-10). The penetration and softening point of the binders was very similar, with B502 being somewhat stiffer (67 1/10 mm) and B504 being somewhat less stiff (81 1/10 mm). The performance grading of the binders was 64–22, except for B501, which graded slightly higher at 70–22.

Table 1

Properties of asphalt binder samples.

Sample	Penetration at 25 °C [1/10 mm]	Softening Point [°C]	PG
B501	77	46.4	70–22
B502	67	47.6	64–22
B503	79	46.3	64–22
B504	81	45.5	64–22

2.2. Methods

2.2.1. Laboratory binder aging

The entire set of binders was short- and long-term aged. Short-term aging was performed according to the RTFOT method (EN 12607-1) with a duration of 75 min. In addition to the standard RTFOT temperature of 163°C , two additional temperatures, 143°C and 123°C , were used to isolate and evaluate the effect of temperature on the aging process. The choice of reduced temperatures was based on warm mix asphalt technologies, which were looked at in a parallel study. Long-term aging was conducted using the PAV device (EN 14769). It was carried out after RTFOT at a temperature of 100°C and an air pressure of 2.1 MPa for 20 h. The RTFOT and PAV have been standard laboratory asphalt aging methods for decades [19].

2.2.2. Penetration, ring and ball

To relate to the different aging effects with standard tests, conventional asphalt binder testing, which is well correlated with bitumen aging [18], was performed. The softening point test according to EN 1427 which represents the consistency at high temperatures. The penetration was tested according to EN 1426 and represents the consistency at intermediate temperatures. Values of penetration and softening point were determined for all asphalt binder samples. Although penetration and softening point are empirical methods they still indicate performance of regular unmodified asphalt binder and are used for product classification of asphalt binders according to European Standards.

2.2.3. Dynamic Shear Rheology

The Dynamic Shear Rheometer (DSR) was used to measure the rheological behaviour of the binder samples under oscillatory sinusoidal loading. The linear viscoelastic (LVE) parameters [20,21] such as complex shear modulus $|G^*|$ and phase angle δ , can be obtained over a wide range of temperatures and frequencies, based on a standard testing procedure. In the present study, DSR testing was carried out with a parallel-plate geometry with an 8 mm diameter and 2 mm gap for temperatures between -10°C and $+40^{\circ}\text{C}$ and a 25 mm diameter and 1 mm gap from $+30^{\circ}\text{C}$ to $+80^{\circ}\text{C}$. The tests were carried out with temperature gradients of 10°C and a frequency sweep from 0.1 Hz to 10 Hz at each temperature. Not all the laboratories were able to perform the tests at the lowest temperatures, so the sample sizes varied. The ESEM testing was conducted at the University of Waterloo on the binders aged at Empa.

2.2.4. Environmental Scanning Electron Microscopy

According to a protocol developed previously [8], the binders were softened by placing them inside covered containers and heating them in an oven for approximately 1 h at 110°C . Approximately 0.1 g was subsequently poured from the containers into 8 mm diameter sample mold using a spatula and the sample was flattened by holding the mold on a hotplate at 150°C for approximately 10 s for the virgin binder and up to 30 s for the stiffer aged binders. These temperatures and heating times were kept to a minimum so as to not age the asphalt binder, while still having it malleable enough to transfer to the mold. The samples were stored at a low temperature of 7°C for 24 h in order to keep aging and changes to the microstructure to a minimum, before observations.

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