Multiscale Characterization of Field Aged SMA and PA Mixtures

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ABSTRACT

The degradation of bituminous materials due to ageing has a significant effect on asphalt pavement performance. In this study, one porous asphalt (PA) and one stone mastic asphalt (SMA) pavement, which are commonly used in the national highway and provincial roads in the Netherlands, were constructed at TU Delft in 2014 and have been exposed to the actual climatic condition since then. To study the change in the pavement's mechanical properties, asphalt cores were collected from both test sections annually. Cyclic indirect tensile tests were performed to determine changes in the mechanical behavior of asphalt cores due to ageing. To investigate the ageing behavior across the pavement depth, the bitumen was extracted and recovered from 13 mm slices along the depths of cores. The chemical composition and rheological properties of the field-recovered bitumen, as well as that of original bitumen aged in standard short- and long-term ageing protocols, were investigated by means of the Fourier Transform Infrared spectrometer and Dynamic Shear Rheometer. The results show that the effect of mineral aggregate packing, and hence of air-void distribution and connectivity, on the ageing sensitivity of the pavements with time was significant, as the changes of the stiffness of the PA were greater than that of SMA mixtures. In addition, the results of field-recovered bitumen show that there is an ageing gradient in PA layer, whereas, the ageing of SMA mainly happens at the surface. Moreover, the results from the field-recovered and laboratory-aged bitumen demonstrate a weak relation between field ageing and standard laboratory ageing protocols.

Keywords
Field ageing, Porous mixture, SMA mixture, Stiffness, Chemistry, Rheology

INTRODUCTION

With 139,000 km of public roads over an area of 42,000 km^2, the Netherlands has one of the most dense road networks in the world. Among them, more than 90% of Dutch highway network is covered with porous asphalt concrete, which has been used as surface wearing courses since 1987 (Van der Zwan et al., 1990). This type of mixtures forms a permeable composition that consists of a relatively high voids content. As a result, porous asphalt provides a significant reduction of noise (Ding & Wang, 2017), reduction of splash and spray in the wet weather conditions (Hsu et al., 2011), and improvement in drainage and, skid and rutting resistance (Chen et al., 2015).

Ravelling is the most common distress identified in porous asphalt concrete. It is defined as the loss of stones from the road surface. Failure occurs either at the interface between the asphalt binder and the aggregates (adhesive damage) or within the asphalt binder (cohesive damage). Due to the loss of the stones, the roughness of the pavement increases, leading to unsafe driving conditions and high levels of noise pollution. The development of ravelling on the road surface shortens the service life of a pavement (Huurman et al., 2010).

Ageing of bituminous materials is believed to be a major contributor to ravelling of asphalt pavements (Airey & Choi, 2002). Bitumen is an organic substance, so its mechanical and chemical properties evolve with time. Studies have demonstrated that ductility and penetration of bitumen reduce, while its softening point increase as a result of ageing (Saoula et al., 2013). Ultimately, the viscosity of the bitumen increases and the material becomes stiffer. This may cause the mixture to become excessively hard and brittle, and consequently more susceptible to cracking at low temperature (Zhang et al., 2019). In addition, ageing may also cause the mixture to become less durable in terms of wear resistance, moisture susceptibility and fatigue life (Das et al., 2015).

The bitumen ageing normally occurs in two stages. Initially, short-term ageing (STA) occurs during asphalt production and pavement construction, and followed by long-term ageing (LTA) of the asphalt mixture during pavement service life (Qian et al., 2020). STA can be well simulated and calibrated in the laboratory; the most commonly used test is the Rolling Thin Film Oven (RTFO) (Lu & Isacsson, 2002). However, the LTA of bitumen will need long periods of ageing if performed at service temperature and pressure conditions. In the lab, the pressure ageing vessels (PAV) test is thought to mimic the age hardening of bitumen during the first five to ten years of pavement service life (Shalaby, 2004). In reality, though, asphalt pavements would serve in the field for years under a variety of environmental factors, such as temperature, oxygen, Ultraviolet (UV) radiation, and rain/snow (moisture) (Xu et al., 2018).

OBJECTIVES

This work focuses on multiscale characterization of field aged PA and SMA mixtures. The main objectives of this study are to: (i) investigate the effect of field ageing on the PA and SMA mixtures; (ii) determine the changes in the chemical composition and mechanical properties of field-recovered bitumen from different depths; (iii) evaluate the performance of compare the chemical properties and stiffness of field-recovered bitumen against standard laboratory-aged bitumen.

MATERIALS AND METHODS

Materials and mixture design

PA and SMA mixtures are commonly used in the national highway and provincial roads in the Netherlands, respectively. In this study, one PA and one SMA mixture were designed using the aggregate gradations shown in **Figure 1**. The same type of PEN 70/100 bitumen, one of the most commonly used in the Netherlands, was used for both mixtures. The bitumen content was 5.0% and 6.4% based on the total weight of mix for the PA and the SMA mixtures, respectively. A factory limestone filler with 26.4% calcium hydroxide, i.e., Wigro 60K filler, was used for both mixtures. Wigro 60K is the commonly used filler in the Netherlands due to its contribution to improving the moisture sensitivity of pavements (Hagos et al., 2009). For both mixtures, crushed sandstone, i.e., Norwegian sandstone was used, with a nominal maximum size of 16 mm. The target air void content was 16% and 5% for the PA and SMA mixtures, respectively. The specifications of the studied materials, i.e., bitumen, filler, and aggregate, can be found in the previous study (Jing et al., 2021).

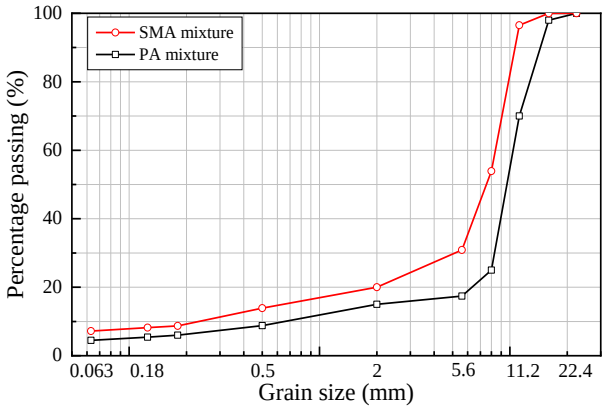

Figure 1 Gradations of PA and SMA mixtures.

Tests on asphalt pavement cores

In this study, samples with a diameter of 100 mm and a thickness of 50 mm were cored from the PA and SMA test sections on an annual basis since 2014. The details of the construction of the test sections can be found in previous publication (Jing et al., 2019). The dynamic modulus (stiffness) of asphalt mixtures is generally used for pavement design, for example in the Mechanistic-Empirical Pavement Design Guide

(MEPDG), to determine pavement thickness and predict pavement performance. Considering the thickness and diameter of the field cores, the dynamic modulus was determined by means of the Cyclic Indirect Tensile Test (IT-CY) according to NEN-EN 12697-26. For each mixture, three replicates were tested using the Universal Testing Machine (UTM) at five frequencies i.e., 0.5, 1, 2, 5, and 10 Hz and four testing temperatures i.e., 0, 10, 20, and 30 °C. The conditioning time before testing was set to 4 hours to equilibrate the temperature of the sample. The effect of time/frequency and temperature on the mechanical behavior of the viscoelastic asphaltic materials was described by master curves using the time-temperature superposition (TTS) principle. The results of the IT-CY test offer a better understanding of the evolution of mixture stiffness in time.

Tests on bitumen samples

To compare the effect of ageing at different depths, the PA and SMA cores of 2018 (the most recent available recovered bitumen samples from the field) were cut into three 13 mm slices from top to bottom, and the remaining part was discarded. The slices are marked with T (Top), M (Middle), and B (Bottom), respectively (**Figure 2**). Bitumen was extracted from the slices by using the fully automatic bitumen extraction apparatus according to the EN 12697-1 European standard. Dichloromethane was used as a solvent during bitumen extraction. Vacuum evaporation using a rotary evaporator was conducted to separate the solvent from bitumen. After the recovery of bitumen, FTIR tests were performed to check if the solvent was fully evaporated. If the solvent was still present in the sample, this would appear in the FTIR results as two special peaks (at around 1250 cm^{-1} and 750 cm^{-1}) in the spectrum (Jing et al., 2019).

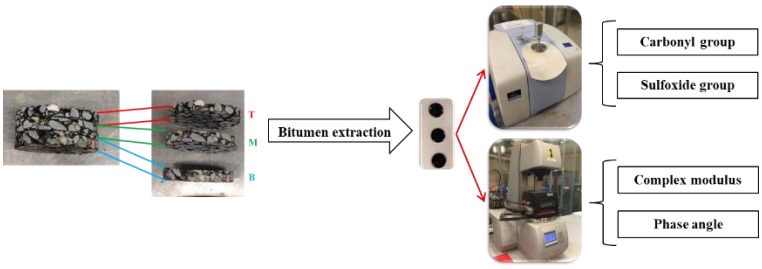

Figure 2 Tests performed on field-recovered bitumen.

Fourier Transform Infrared (FTIR) spectroscopy is generally adopted as a tool to monitor the changes in the chemical composition of bitumen after ageing and to quantify oxidation. In this study, a Perkin Elmer Spectrum 100 FTIR spectrometer was used in the attenuated total reflectance (ATR) mode to identify the chemical functional groups of the bitumen. The sample was scanned 20 times with a fixed instrument resolution of 4 cm^{-1}. The wavenumbers range was set to vary from 600 to 4000 cm^{-1}. At least three repetition tests were done for each ageing condition.

The rheological properties of field-recovered bitumen were characterized by means of Dynamic Shear Rheometer (DSR) according to NEN-EN 14770. The bitumen samples were tested using the parallel-plates configuration. Initially, the linear viscoelastic (LVE) strain range of bitumen samples was determined using amplitude sweep tests. The frequency sweep tests were performed at five different temperatures (0, 10, 20, 30, and 40 °C). During the tests, the frequency varied in a logarithmic manner from 50 Hz to 0.01 Hz. At least three repetition tests were done for each ageing condition.

For comparison, the virgin PEN 70/100 bitumen was subjected to standard laboratory ageing. To be specific, STA was simulated by using the RTFO test at 163 °C for 75 minutes, in accordance with the EN 12607-1 test standard. LTA was simulated by using PAV at 100 °C and 2.1 MPa (20 atm) for 20 hours, according to the EN 14769 ageing standard. Then the chemical and rheological properties of bitumen at fresh, STA and LTA conditions were also evaluated using FTIR and DSR.

RESULTS AND DISCUSSION

Mixture stiffness

According to the TTS principle, the master curve of the dynamic modulus was generated at a reference temperature of 20 °C. **Figure 3** shows the evolution of stiffness for the PA and SMA mixtures from 2014 to 2021.

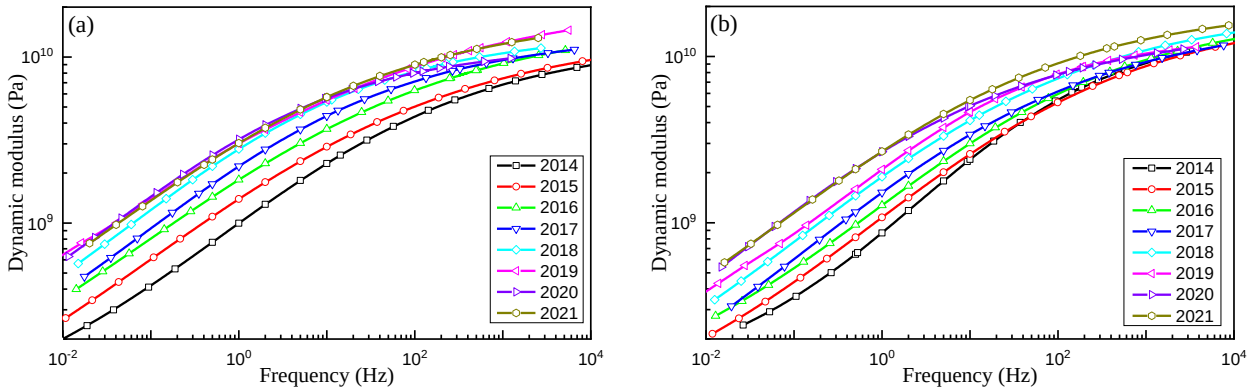

Figure 3 Master curves of asphalt mixture at reference temperature 20 °C, (a) PA mixture, (b) SMA mixture.

Overall, the dynamic modulus of both PA and SMA mixtures increases with time. The rate of modulus change is higher for the PA than for the SMA mixture before 2018. This is mainly because of the high void content of PA, which leads to inherently high sensitivity to oxidative ageing. According to the CT scan results of core samples, the PA mixture had 16% and the SMA mixture had 5% of air voids, as shown in previous study (Jing et al., 2019). It has been found that, the high air void content of PA results in high interconnectivity of the voids network, in which more than 90% of the total air voids are interconnected, while less than 10% of the total air voids have been reported to be interconnected in SMA mixtures (Varveri et al., 2014). However, after 2018, the rate of modulus change is less for the PA than for the SMA mixture. This is mainly because of the high initial rate of oxidation for the PA mixtures in the first years, which slowed down after 2018 (as approaching towards the ultimate ageing state). The PA stiffness has not increased significantly after 2018 (4 years' field ageing), while the SMA stiffness has no increased significantly after 2020 (6 years' field ageing).

Changes in the chemical functionality of bitumen

The FTIR tests were performed on fresh, laboratory-aged and field-recovered bitumen samples to determine their chemical changes due to ageing. The carbonyls group (C=O) at 1753-1660 cm^{-1} wavenumbers and the sulfoxides group (S=O) band at 1047-995 cm^{-1} wavenumbers, in the FTIR spectrum, were utilized to quantify bitumen oxidation. The carbonyl and sulfoxide indices were calculated using Equation (1) and (2) to quantify the changes in the area under the two peaks using the vertical limit bands as shown in **Figure 4** (Van den Bergh, et al., 2011).

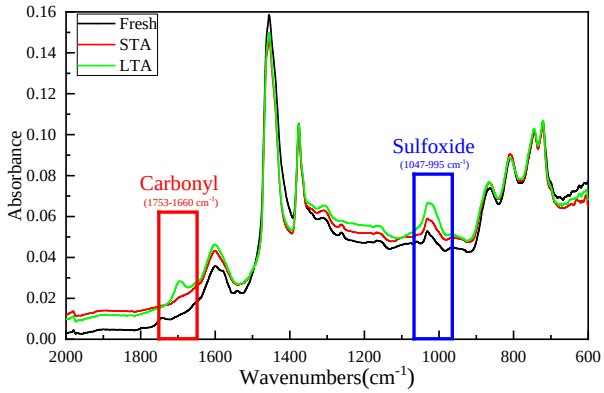

Figure 4 FTIR spectra of laboratory-aged bitumen.

$$\text{Carbonyl index} = \frac{A_{1700}}{\sum A} \quad (1)$$

$$\text{Sulfoxide index} = \frac{A_{1030}}{\sum A} \quad (2)$$

$$\text{where } \sum A = A_{(2953,2923,2862)} + A_{1700} + A_{1600} + A_{1460} + A_{1376} + A_{1030} + A_{864} + A_{814} + A_{743} + A_{724}$$

Table 1 Vertical Limit Bands with the Corresponding Functional Groups (Van den Bergh, et al., 2011)

Area	Vertical Band Limit (cm^{-1})	Functional Groups
A_{724}	734-710	Long chains
A_{743}	783-734	Out of plane adjacent
A_{814}	838-783	Out of plane adjacent
A_{864}	912-838	Out of plane singlet
A_{1030}	1047-995	Oxygenated functions - sulfoxide
A_{1376}	1390-1350	Branched aliphatic structures
A_{1460}	1525-1395	Aliphatic structures
A_{1600}	1670-1535	Aromatic structures
A_{1700}	1753-1660	Oxygenated functions - carbonyl
A_{2862}	2880-2820	Stretching symmetric
A_{2953}	2990-2880	Stretching aromatic

In **Figure 5**, it is interesting to note that no carbonyls are formed after STA, while the sulfoxides show an increasing trend. This can be explained by the fact that sulfur is more reactive than carbon in bitumen and verifies the higher rate production of sulfoxides than ketones reported in earlier studies (Lesueur, 2009). On the contrary, the formation of carbonyls starts after LTA probably because by that time most of sulfur has been consumed. This phenomenon was discussed in the previous study (Jing et al., 2018).

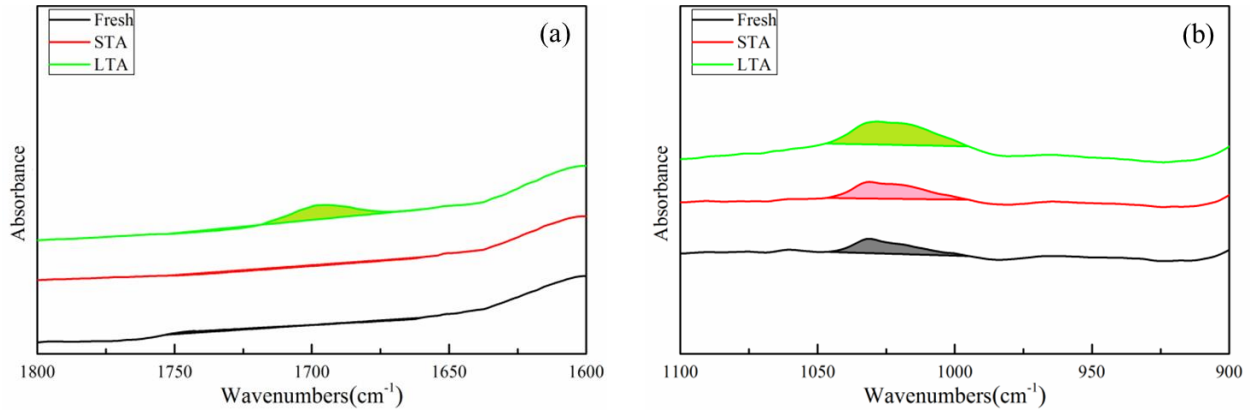

Figure 5 The areas of specific ageing functional group, (a) carbonyl, (b) sulfoxide.

The quantitative indices for carbonyls and sulfoxides of the laboratory-aged bitumen and recovered bitumen of PA are shown in **Figure 6** (a) and (b). The results show that the ageing indices (carbonyl index and sulfoxide index) increase with time and that the effect of ageing is more significant for the top part of the PA layer than for the bottom, which supports the results presented in earlier studies (Choquet & Verhasselt, 1994; Farrar et al., 2006).

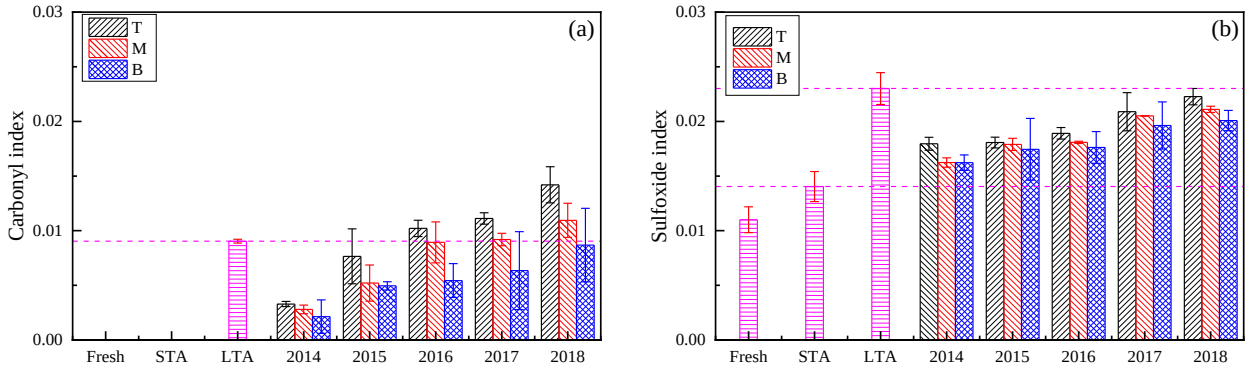

Figure 6 Ageing indices of PEN 70/100 at different ageing conditions, (a) Carbonyl index, (b) Sulfoxide index.

From a comparison between the results of laboratory-aged bitumen and recovered bitumen, it is easily observed that carbonyls were not formed in the studied bitumen after STA, whereas carbonyl groups are already present in the samples after laying in 2014, **Figure 6** (a). Moreover, the sulfoxide index of the field-aged bitumen was slightly below than the index of the bitumen after LTA, **Figure 6** (b). Overall, it appears that the standard STA and LTA protocols cannot describe properly the short- and long-term (5-10 years) ageing behaviour of porous asphalt both in terms of chemistry.

In addition, the quantitative indices for carbonyls and sulfoxides of laboratory-aged and field-recovered bitumen of PA_2018 and SMA_2018 are shown in **Figure 7**. Overall, the results show that the formation of carbonyls and sulfoxides were higher for the bitumen samples recovered from the PA than those from SMA. These results suggest that PA has a higher ageing sensitivity than SMA, which agrees with the stiffness results of the core samples presented in the previous section.

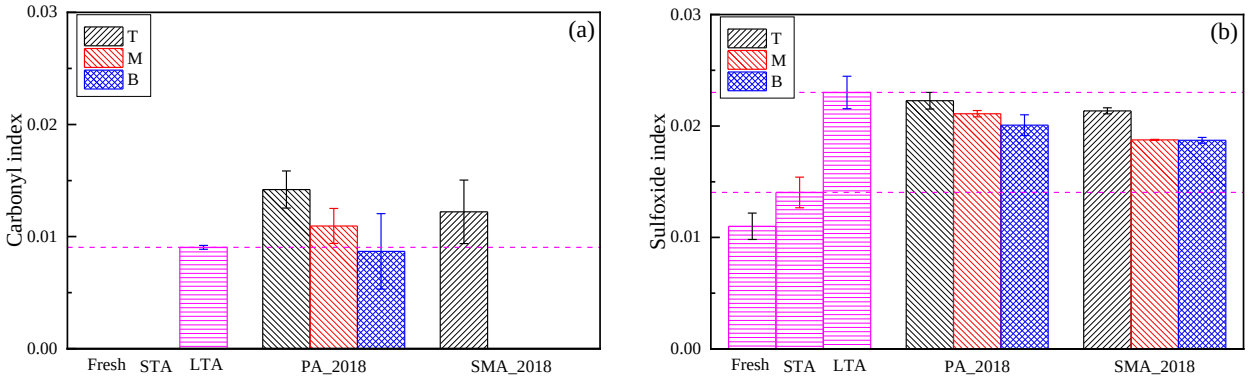

Figure 7 Ageing indices of laboratory-aged and field-recovered bitumen of PA_2018 and SMA_2018, (a) carbonyl index, (b) sulfoxide index.

Moreover, the ageing indices across the pavement depth are significantly different between PA and SMA mixtures. For both mixture types, ageing at the top slice (pavement surface) is more severe as indicated by the higher values of the ageing indices in comparison to those at lower pavement depths, due to the direct exposure to the atmospheric oxygen and temperature. For the PA mixture, it can be observed that the ageing indices gradually decreases from top to bottom. However, for the SMA mixture, the top slice has the highest ageing indices compared to the middle and bottom slices, which have similar values but significantly lower values than the top. These findings suggest that ageing has a full-depth effect on PA pavements, but its impact on SMA pavement mainly occurs at the surface.

From a comparison between the results of laboratory-aged and field-recovered bitumen for both the PA and SMA mixtures, the carbonyl index of the recovered bitumen is higher than that of LTA. This can be explained by the different conditions that are present during laboratory and field ageing. For instance, laboratory ageing is performed at relatively high temperatures (100 °C in this study), while field ageing of pavement occurs at lower temperature and is accompanied by moisture and UV effects. On the other hand, the sulfoxide index of the recovered bitumen is smaller than that of LTA. This may also be caused due to the combined ageing-moisture effects. According to some studies, the sulfoxides are water soluble and can be removed by water in the field condition (Green et al., 1993; Menapace et al., 2018).

Change in the rheological properties of bitumen

The complex shear modulus and phase angle values were determined during DSR tests. To obtain the visco-elastic behaviour in a wider range of frequencies, master curves of complex shear modulus and phase angle were generated at reference temperature 20 °C on the basis of TTS principle. **Figure 8** shows the master curves of PEN 70/100 bitumen in the unaged (fresh) state and after the application of the STA and LTA protocols.

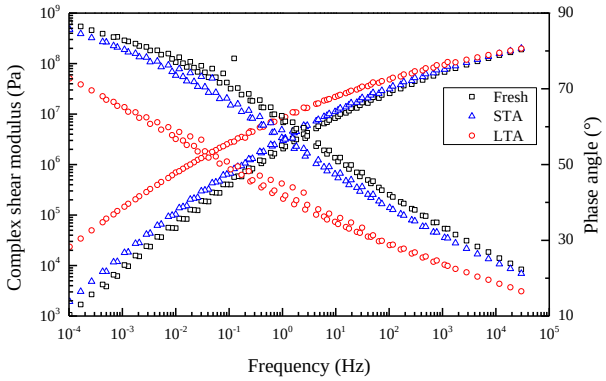

Figure 8 Master curves of complex shear modulus and phase angle of PEN 70/100 at Fresh, STA and LTA state.

In **Figure 8**, the observed differences on the master curves of complex shear modulus are more pronounced at low frequencies where bitumen is in the rubbery flow region, whereas minor changes were observer at high frequencies where bitumen behaves more as solid. Specifically, the variation of complex modulus between fresh and STA/LTA materials was about 0.1 and 1.1 orders of magnitude at low frequencies, respectively. At the highest frequency, all samples tend to reach an asymptote at a value of 10^8 Pa. The STA sample results in a slight difference with respect to the rheological response of fresh bitumen. On the contrary the modulus of the LTA sample shows a significant difference with that of fresh bitumen. This denotes that bitumen ageing mainly occurs after placement and during the lifetime of a pavement. On the other hand, the phase angle decreases substantially for the whole frequency range, indicating a reduction of its viscous flow characteristics.

Figure 9 shows the evolution of the rheological properties of recovered bitumen from PA with time for top and bottom slices. It can be seen that the complex shear modulus and the phase angle for the top parts of the pavement change significantly with time. On the other hand, the changes are less obvious for the bottom part of the pavement; the values indeed change with time but at a slower rate. This finding supports the hypothesis of a stiffness gradient across the depth of the pavement. A stiffness gradient leads to higher tensile stresses at the surface and, as noted in the NCHRP 1-37A Design Guide (NCHRP, 2004), ageing hardening near the surface can play a major role in causing top-down cracking.

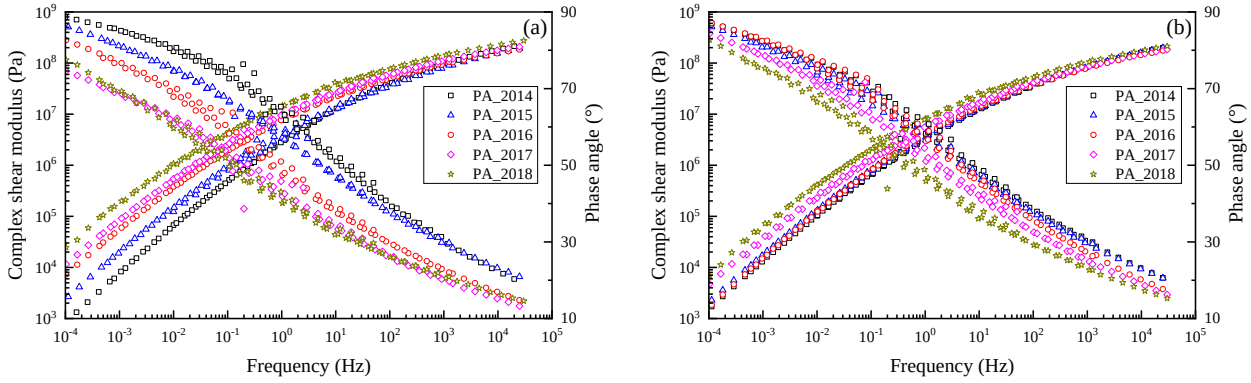

Figure 9 Evolution of complex shear modulus and phase angle master curves with time at different slices of the PA mixture, (a) top slices, (b) bottom slices .

To compare the effect of ageing at different depths, the rheological properties of recovered bitumen from the PA and SMA cores of 2018 were studied. **Figure 10** shows that PA and SMA mixtures have higher complex shear modulus and lower phase angle at the top slice compared to the middle and bottom slices. As expected, such results indicate that ageing is more severe for the top part of the pavement. In addition,

for the PA mixture (**Figure 10** (a)), it can be observed that the complex shear modulus decreases and the phase angle increases from the top to the bottom slices. While, for SMA mixture (**Figure 10** (b)), the middle and bottom slices have consistent complex shear modulus and phase angle values across the frequency spectrum. These results point out that ageing affects the PA mixture throughout its depth, whereas for SMA mixture the ageing effects are mainly found on the surface. This observation agrees well with the chemical profiles of the PA and SMA mixtures as discussed in the previous section.

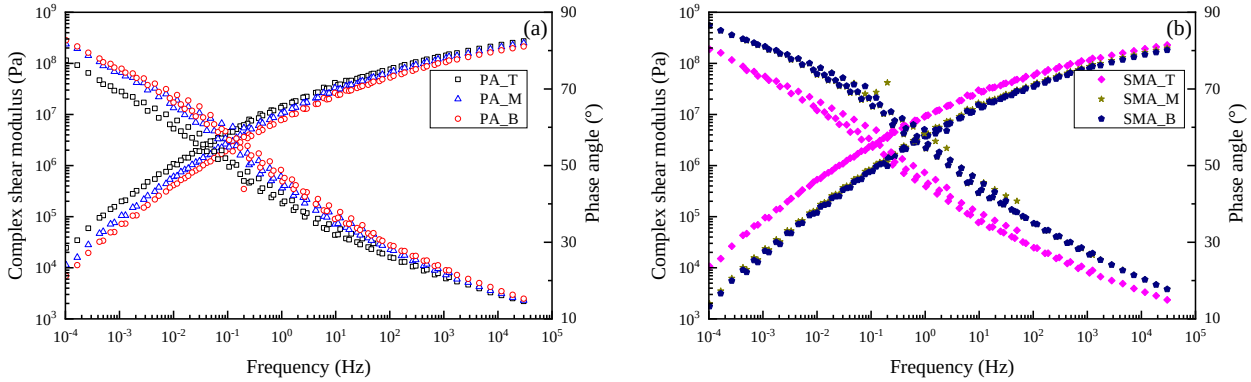

Figure 10 Master curves of complex shear modulus and phase angle of field-recovered bitumen of, (a) PA_2018, (b) SMA_2018.

For comparison, the master curves of complex shear modulus and phase angle of laboratory-aged and field-recovered bitumen are illustrated in **Figure 11**. The results show that PA has a higher complex shear modulus and lower phase angle than SMA at the corresponding slices. As expected, it suggests that PA has a higher ageing rate due to its high porosity. In addition, the master cures show that the stiffness of the bitumen recovered from the top slice (PA_T) is higher than that of LTA sample, whereas the top slice of SMA (SMA_T) has approximately the same stiffness as the LTA sample. It appears that the ageing state of the top slices of the PA and SMA is more severe than or similar to the LTA sample. These results imply again that the long-term field ageing condition cannot be simulated accurately by the PAV protocol. Moreover, the middle and bottom slices of the SMA (SMA_M and SMA_B) have slightly higher stiffness and lower phase than the STA samples. This denotes that the SMA mixture has a better ageing resistance due to its high density that prevents oxygen flow into the pavement structure and slows down ageing inside the pavement structure.

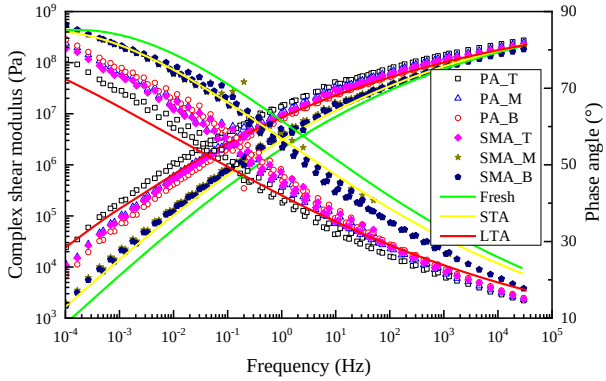

Figure 11 Comparison of master curves (complex shear modulus and phase angle) of laboratory-aged and field-recovered bitumen.

CONCLUSIONS AND RECOMMENDATIONS

This study has investigated the changes in the chemical composition and rheology of laboratory-aged and recovered field-aged bitumen and focused on multiscale characterization of field aged PA and SMA

mixtures. Test sections of PA and SMA were constructed in 2014 and continuously exposed to environmental conditions. The rheological and chemical profile of the recovered bitumen was compared with that of laboratory-aged bitumen after STA and LTA. The following conclusions are drawn based on the results obtained from laboratory experiments:

- Bitumen ageing is more severe for the top part of the pavement, as it is in contact with the atmospheric air and the pavement temperature is higher.
- Field ageing at the pavement surface is far more severe than standard laboratory ageing protocols and cannot be simulated by standard RTFOT and PAV ageing.
- Field ageing has more influence on PA mixtures than on SMA mixtures, because of the high void content of the PA mixtures, which leads to a larger binder area directly exposed to oxygen and temperature.
- A significant difference in the field-ageing behavior of the PA and SMA mixtures is observed along the pavement depth. Specifically, an ageing gradient exists in the PA mixture, whereas the ageing of the SMA mixture mainly occurs at the surface.

As a continuation of this research, an experimental testing program is currently undertaken on the recent collected asphalt cores and extracted bitumen with the aim to develop a proper ageing protocol to simulate long-term and field ageing behavior of PA and SMA mixtures. In addition, the rheological and chemical profiles of field-recovered bitumen results from the synergistic effects of thermal-oxidative ageing, photo-oxidative (ultraviolet radiation), and moisture. To fully characterize the field ageing behavior of asphalt pavement, the various environmental factors need to be coupled in the laboratory protocols.

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