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Long-term performance of asphalt mixtures containing bio-extended bituminous binders: laboratory investigation and accelerated pavement testing

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ABSTRACT

Bio-extended bituminous binders were formulated with tall oil pitch (TOP). Their corresponding asphalt mixtures were prepared. Various laboratory tests and full-scale accelerated pavement testing (APT) were conducted to evaluate their long-term performance. It is indicated that, at 25°C, the aged bio-extended 70/100 binder with 5% TOP had similar fatigue resistance to its reference. At 10°C, the aged bio-extended 160/220 binder with 21% TOP was marginally less resistant to fatigue at small strains. The asphalt mixture with bio-extended 160/220 binder had higher stiffness at 10°C and similar water sensitivity to its reference. Although the aged mixture with bio-extended binder showed higher strength to resist cracks at 5°C, it was more brittle, leading to faster crack propagation. After further moisture conditioning, the mixture became weaker but less brittle. The full-scale APT indicated that the section with bio-extended binders was more resistant to permanent deformation. Overall, the results suggest potential for larger-scale testing.

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KEYWORDS
Bio-bitumen; bio-asphalt; bio-oil; tall oil pitch; long-term performance; accelerated pavement testing

1. Introduction

Asphalt mixture is the predominantly used pavement material for road surfaces all over the world. Bitumen is commonly used as the binder to hold the asphalt mixture together. As concerns about climate and environmental issues grow, however, the use of traditional petroleum bitumen in asphalt mixtures has been questioned due to its dependence on non-renewable resources, high energy consumption and significant greenhouse gas (GHG) emissions (Corona et al., 2022; Mattinzioli et al., 2021; Shirzad & Zouzias, 2024). This has resulted in the development of alternative binders that are either partially or fully derived from renewable resources, offering a more sustainable solution for road construction (Ingrassia et al., 2019a; Penki & Rout, 2023). Among others, bio-extended bituminous binders, which usually contain bio-based components partially replacing petroleum bitumen, present an opportunity to reduce the carbon footprint and decrease the dependence on fossil resources of the asphalt paving industry (Gaudenzi et al., 2023; Riccardi & Losa, 2024).

Among the related investigations so far, the use of plant-based bio-oils as bitumen extenders has shown the most promising results (Ingrassia, 2021; Wang et al., 2020; Zhu et al., 2024a). Raouf and Williams (2010) investigated the potential of using bio-binders derived from switchgrass bio-oil

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fractions as a sustainable alternative to petroleum bitumen. Their study concluded that the fractionated switchgrass bio-oils exhibited similar characteristics to traditional bituminous binders. Grilli et al. (2019) examined the effects of five bio-based additives derived from the distillation process of crude tall oil (from pine trees) on bitumen properties. The researchers found that the different additives could either harden, soften or change the temperature susceptibility of bitumen. Ingrassia and Canestrari (2022) studied the fatigue behaviour of an asphalt mixture with bio-extended bituminous binder containing a wood-based bio-oil (10% replacement in the binder). Their study suggested potential fatigue performance benefits of using the bio-extended bituminous binder in the long term.

The success of using plant-based bio-oils to partially replace bitumen can be attributed to several factors. First, the incorporation of bio-oils, especially at low content, has not demonstrated a significant negative impact on the properties of bituminous binders. Second, bio-oils allow for the replacement of a relatively large portion of bitumen compared to other approaches, thereby maximising the environmental and sustainability benefits. Lastly, replacing petroleum-derived bitumen (rather than other components in asphalt mixture) is a more effective strategy for addressing the associated climate and environmental concerns.

Plant-based bio-oils can be derived from a wide variety of renewable biomass sources, including wood, agricultural residues and waste cooking oil (He et al., 2023). By blending bio-oils with conventional bitumen, the resulting binder offers a more sustainable and environmentally friendly alternative to solely using petroleum bitumen in asphalt pavements. In addition to the environmental advantages, previous studies have shown that certain plant-based bio-oils can enhance specific properties of bituminous binders. For example, it has been reported that some bio-oils can improve the flexibility, adhesion and workability of the binder (Ingrassia et al., 2019b, 2020), which are critical to the performance and durability of asphalt pavements. These findings demonstrate the potential of plant-based bio-oils not only to reduce the environmental impacts of asphalt production but also to contribute to improved pavement performance.

However, despite their potential benefits, further research is required to fully understand the long-term performance and durability of asphalt mixtures containing bio-extended bituminous binders under various traffic and environmental conditions. This is to ensure that the plant-based bio-oils can adequately replace traditional petroleum bitumen in asphalt pavements. For this purpose, the present study investigated a specific type of plant-based bio-oil, tall oil pitch (TOP), for its use as an extender in bituminous binders (namely partial replacement). The TOP bio-oil is sourced from a biorefinery that processes bio-based by-products from the pulp and paper industry, making it a sustainable alternative derived from forest resources. Chemically, TOP consists mainly of high-boiling esters and free acids.

In this study, bio-extended bituminous binders were formulated with the plant-based bio-oil (TOP) and asphalt mixtures were prepared with the binders. Various laboratory tests were conducted to evaluate the long-term performance of the bio-extended bituminous binders and their asphalt mixtures. Additionally, accelerated pavement testing (APT) was carried out to demonstrate and validate the long-term performance of the materials in full scale. The results of this study are expected to enhance our understanding of the long-term performance of asphalt mixtures containing bio-extended bituminous binders.

2. Materials and methods

2.1. Materials

The bio-oil investigated in this study was TOP, a plant-based material derived from by-products of the pulp and paper industry. Its Fourier-transform infrared (FTIR) spectrum is shown in Figure 1. In addition to the methylene and methyl groups that commonly exist in organic matters, the spectrum presents strong signals of C = O and C–O chemical bonds, confirming the presence of a large amount of esters and acids in the bio-oil. This plant-based bio-oil was used to formulate bio-extended bituminous binders in the present study. Different amounts of TOP bio-oil were blended with a base bitumen

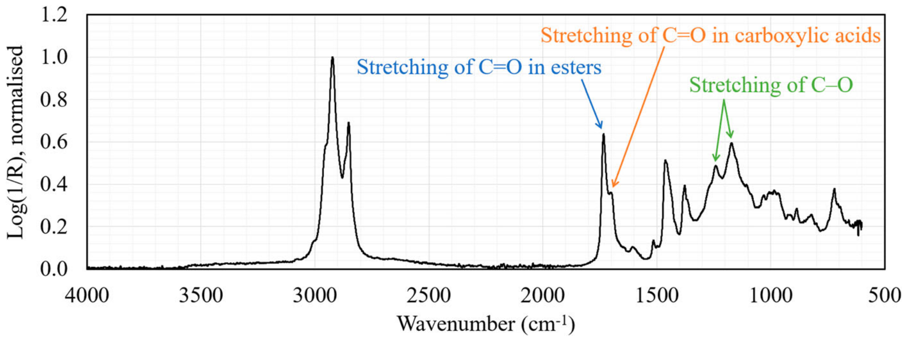

Figure 1. Fourier-transform infrared spectrum of the plant-based bio-oil (TOP).

Table 1. Formulation of the bio-extended bituminous binders.

Bio-extended bituminous binders	Base bitumen (% by weight of the blend)	Content of plant-based bio-oil TOP (by weight of the blend)
Bio 70/100	50/70 bitumen (95%)	5%
Bio 160/220	50/70 bitumen (79%)	21%

Table 2. Basic properties of the bituminous binders.

Bituminous binders	Bio 70/100	Ref 70/100	Bio 160/220	Ref 160/220
Penetration @ 25°C (0.1 mm)	80	82	193	196
Softening point, original (°C)	47.2	46.8	40.4	38.8
Softening point, after RTFOT* (°C)	51.2	52.8	43.6	45.0
Softening point, after RTFOT + PAV# (°C)	57.8	62.4	50.4	53.8

Notes: * RTFOT: Rolling Thin Film Oven Test at 163°C for 75 min according to EN 12607-1:2024; # PAV: Pressure Ageing Vessel ageing at 100°C and 2.1 MPa for 20 h according to EN 14769:2023.

of penetration grade 50/70 to prepare bio-extended binders. As the result, two bio-extended bituminous binders were formulated, matching the penetration grades of 70/100 and 160/220 respectively in terms of needle penetration and softening point. These bio-extended binders are denoted as 'Bio 70/100' and 'Bio 160/220' in this paper and their formulation is presented in Table 1.

The penetration grades of 70/100 and 160/220 are commonly used in Sweden. The two bio-extended bituminous binders were formulated to obtain consistency equivalent to these grades, with their penetration and softening point values as close to the reference counterparts as possible. In addition to the two bio-extended bituminous binders, their reference counterparts were also analysed in this study, namely a conventional 70/100 bitumen (denoted as 'Ref 70/100') and a conventional 160/220 bitumen (denoted as 'Ref 160/220'). All four binders are listed in Table 2 with their penetration (EN 1426:2024, average of three determinations) and softening point (EN 1427:2015, average of two specimens, before and after ageing) values. It shows that the original binders have very similar basic properties and the softening point changes after ageing are smaller for the bio-extended variants than their reference counterparts. For more information about these binders, please refer to a previous publication by the authors (Zhu et al., 2024b).

These four binders were used to prepare dense graded asphalt mixtures: typical Swedish wearing course mixtures (ABT16) with the 70/100 binders and base course mixtures (AG16) with the 160/220 binders. For each binder grade, the mix design procedure was the same and the basic characteristics such as volumetric parameters of mixtures were kept as close as possible between the bio-extended variant and reference counterpart. Figure 2 shows the aggregate gradation curves of the two types of asphalt mixtures. The aggregates were 100% crushed rock, consisting mainly of gneiss, amphibolite and granite. No reclaimed asphalt or anti-stripping agent was used in the mixtures. The binder content

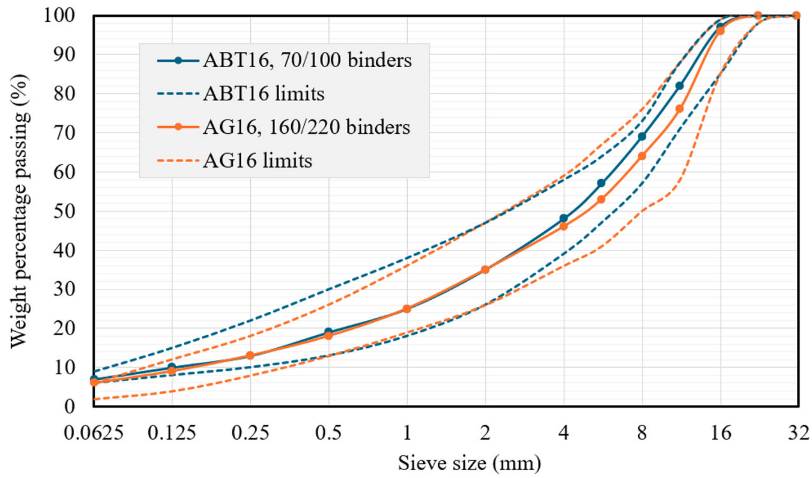

Figure 2. Aggregate gradation curves of the asphalt mixtures.

of ABT16 mixtures with 70/100 binders was 5.9%; and that of AG16 mixtures with 160/220 binders was 4.5%. The target air void of ABT16 mixtures with 70/100 binders was 2.1% while that of AG16 mixtures with 160/220 binders was 4.2%.

2.2. Methods

To evaluate long-term performance, the four bituminous binders were aged in laboratory with the Rolling Thin Film Oven Test (RTFOT at 163°C for 75 min according to EN 12607-1:2024) followed by Pressure Ageing Vessel (PAV) ageing at 100°C and 2.1 MPa for 20 h according to EN 14769:2023. RTFOT is a method for conditioning bituminous binders to measure the combined effects of heat and air on a thin moving film of the binder. It simulates the short-term ageing that most bituminous binders undergo during mixing in an asphalt plant. PAV ageing is an accelerated long-term ageing procedure for bituminous binders, which involves ageing trays of binder at an elevated temperature under a pressurised condition in a PAV instrument. After the long-term ageing, the conditioned binders were analysed with the Linear Amplitude Sweep (LAS) test method according to AASHTO T391-20, to evaluate their fatigue resistance. The LAS tests were carried out using a Dynamic Shear Rheometer (DSR, 8 mm plates) at 25°C for the 70/100 binders and 10°C for the 160/220 binders. The selection of LAS test temperatures considered the specific service conditions of the different binders in asphalt pavements.

As for asphalt mixtures, slabs of AG16 mixtures (160/220 binders) for asphalt base course were prepared with an asphalt roller. Core specimens were drilled from the slabs. The compacted and drilled AG16 specimens were aged in laboratory according to CEN/TS 12697-52:2017 (Procedure B.1 at 65°C for 15 days, see the Appendix) and subsequently analysed for fatigue resistance at 10°C according to EN 12697-24:2018 Annex E (indirect tensile test on cylindrical shaped specimens of 100 mm in diameter and 50 mm thick) and stiffness modulus at the same temperature according to EN 12697-26:2018 + A1:2022 Annex C (indirect tensile test on cylindrical shaped specimens of 100 mm in diameter and 50 mm thick) with load time 0.1 s and rest time 2.9 s. In addition, compacted specimens of AG16 mixtures were also conditioned with a Moisture Induced Stress Tester (MIST) according to a customised practice after ASTM D7870/D7870M-20. The MIST conditioning was at 40°C with 12,000 cycles and 0.28 MPa pore pressure (Rahman et al., 2022). Both before and after the MIST conditioning, the asphalt mixture specimens were tested in wet state for stiffness modulus at 10°C with the same test method as described above.

Besides the separate analyses on impacts of individual factors, the combined effect of ageing and water was also assessed. For this, the laboratory aged AG16 mixtures were further conditioned with

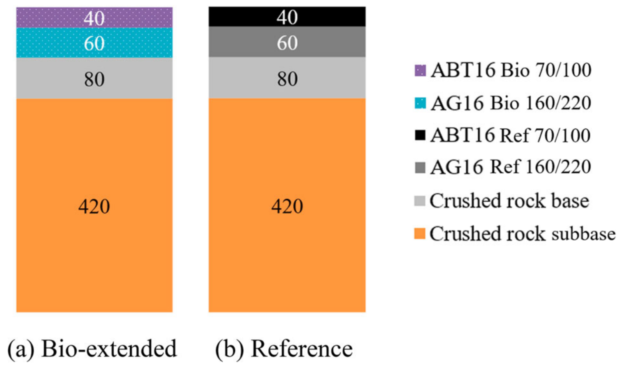

Figure 3. Cross-sections of the pavement structures for APT – four layers over a sand subgrade (numbers indicate the layer thickness in mm): (a) bio-extended and (b) reference.

MIST following the same MIST conditioning procedure as described above. These specimens after both laboratory ageing and MIST, together with their reference counterparts, were tested in dry state using dynamic mechanical analyses according to EN 12697-26:2018+A1:2022 Annex F (cyclic indirect tensile test on cylindrical shaped specimens of 100 mm in diameter and 50 mm thick) to assess their mechanical property changes due to both ageing and water. Furthermore, for the evaluation of crack propagation potential, the laboratory aged AG16 mixtures were also analysed with the Semi-Circular Bending (SCB) test method at 5°C according to EN 12697-44:2019. The specimens for SCB test were 150 mm in diameter and 50 mm thick. The notch depth was 10 mm. Some aged specimens were further conditioned with MIST and these specimens were also analysed with the SCB test method at 5°C after both laboratory ageing and MIST conditioning.

At last, two pavement sections with the analysed asphalt mixtures were tested by APT in full scale. As illustrated in Figure 3, the tested pavement sections consisted of four layers over a sand subgrade, namely an asphalt wearing course layer (ABT16), an asphalt base course layer (AG16), a crushed rock base layer and a crushed rock subbase layer. The difference between the two sections was in the material for asphalt layers – a bio-extended section with bio-extended binders in asphalt mixtures and a reference section with conventional bitumen in asphalt mixtures. The two sections were paved next to each other in a concrete test pit that is 3 m deep, 5 m wide and 15 m long, as shown in Figure 4(a). After paving, the sections were subjected to wheel loading using a Heavy Vehicle Simulator (HVS at VTI – the Swedish National Road and Transport Research Institute, see Figure 4b) in both directions with dual wheels at 12 km/h. First, 20,000 cycles of 30 kN uniform preloading (wheel load) were applied at 10°C. At the same temperature, after about 1000 cycles of response measurement, 240,000 cycles of 60 kN loading (wheel load) with normally distributed lateral positions were applied to the APT sections. This was followed by another 240,000 cycles of the same loading at an elevated temperature of 20°C. During the APT test, the deformation of the sections, namely the rut depth, was regularly measured with a laser sensor from the pavement surface.

3. Results and discussion

3.1. *Fatigue resistance of bituminous binders*

The LAS test according to AASHTO T391-20 included two steps: a frequency sweep followed by an amplitude sweep. Both steps were run at the same given test temperature. The frequency sweep at 0.1% shear strain was for determining the ‘alpha’ parameter for damage analysis, while it also disclosed the basic linear viscoelastic properties of the binder. Figures 5 and 6 present the frequency sweep results of the analysed binders in this study. It is indicated that the laboratory aged Bio 70/100 binder, containing 5% plant-based bio-oil (TOP), showed slightly lower dynamic shear modulus |G*|

(a) Paved sections for APT (b) Heavy Vehicle Simulator at VTI

Figure 4. Accelerated pavement testing at VTI: (a) paved sections for APT and (b) heavy vehicle simulator at VTI.

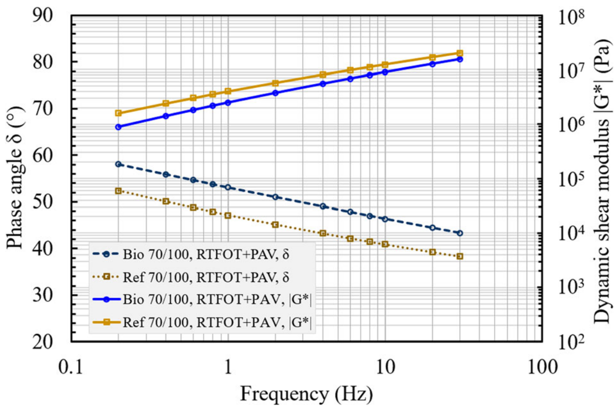

Figure 5. Frequency sweep results of the aged 70/100 binders during LAS tests at 25°C.

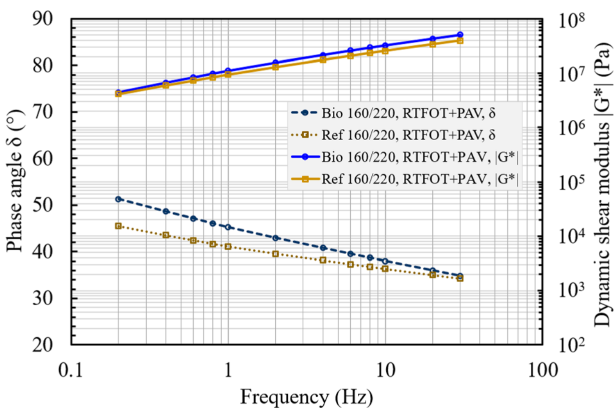

Figure 6. Frequency sweep results of the aged 160/220 binders during LAS tests at 10°C.

and significantly higher phase angle δ than its reference counterpart at 25°C. At a lower temperature, 10°C, the laboratory aged Bio 160/220 binder with 21% bio-oil replacement showed slightly higher |G*| and higher δ than its reference counterpart.

For amplitude sweep, the LAS test applied a stepped linear strain procedure from 0.1% to 30% shear strain. In total, 31 strain levels were applied, all at 10 Hz and for 10 s each, and 3100 loading cycles were

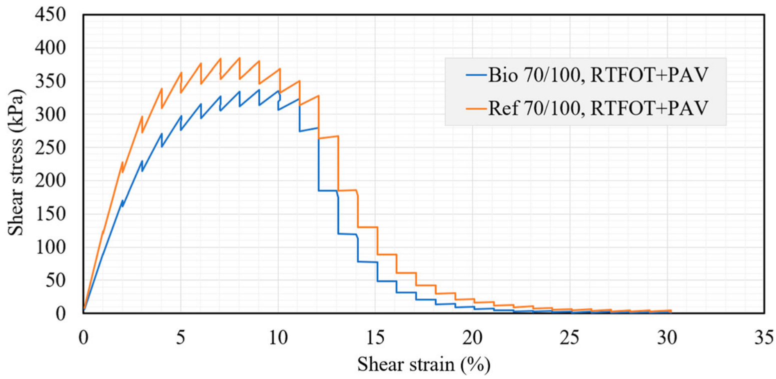

Figure 7. Shear stress–strain curves of the aged 70/100 binders during LAS tests at 25°C.

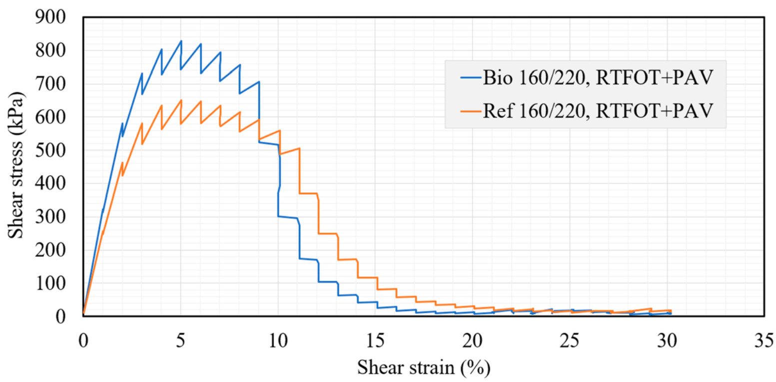

Figure 8. Shear stress–strain curves of the aged 160/220 binders during LAS tests at 10°C.

performed during the amplitude sweep. This loading scheme generated shear stress–strain curves of 'bell-like' shape for the test samples, as shown in Figures 7 and 8. At very small shear strains, the binders exhibited a linear mechanical behaviour. As the shear strain increased, the samples reached their non-linear range and damages started to occur. After reaching its peak, the shear stress decreased significantly at high strains, indicating significant damages in the test sample (Hintz et al., 2011).

With 5% plant-based bio-oil (TOP), the laboratory aged Bio 70/100 binder showed slightly slower increase and lower peak value of the shear stress than its reference counterpart at 25°C. The limited replacement by TOP did not significantly change the shear stress–strain curve of aged binder at the temperature. However, when the replacement by TOP was higher (21%) and test temperature was lower, the laboratory aged Bio 160/220 binder showed significantly higher peak value of the shear stress than its reference counterpart at 10°C, indicating higher stiffness of the binder and possibly higher strength at the temperature. Additionally, the laboratory aged Bio 160/220 binder also showed faster decrease of the shear stress at high strains, which suggests that although the bio-extended binder can withstand higher stress, it would exhibit faster development of failure after certain damage accumulation.

Furthermore, the LAS analysis template v1.59 (developed by the Modified Asphalt Research Center at University of Wisconsin–Madison, version on March 26, 2024) was used to calculate the fatigue model parameters and binder fatigue performance parameter N_f. The binder failure criterion of 35% damage was considered, namely 35% reduction in undamaged $|G^*|\bullet\sin\delta$ at 0.1% shear strain, where $|G^*|$ is the dynamic shear modulus and δ is the phase angle. The calculation results are listed in Table 3.

Table 3. LAS test results of the bituminous binders after long-term ageing.

Bituminous binders	Temperature (°C)	Fatigue model parameters		Binder fatigue performance, N_f	
		A_{35}	B	@ 2.5% strain	@ 5.0% strain
Bio 70/100, RTFOT + PAV	25	1.538×10^6	5.172	13457	373
Ref 70/100, RTFOT + PAV	25	2.137×10^6	5.624	12351	250
Bio 160/220, RTFOT + PAV	10	1.635×10^6	5.737	8527	160
Ref 160/220, RTFOT + PAV	10	4.482×10^6	6.172	15686	218

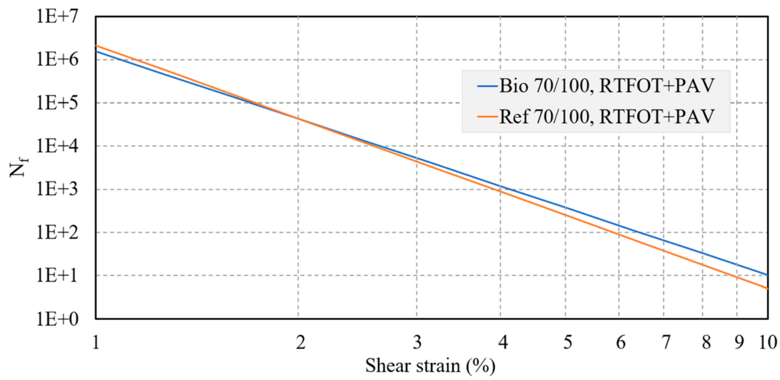

Figure 9. Fatigue resistance of the aged 70/100 binders by LAS tests at 25°C.

The results indicate that, with 5% replacement by TOP, the aged Bio 70/100 binder showed slightly better fatigue performance (higher N_f at both 2.5% and 5.0% strains) than its reference counterpart at 25°C. However, the aged Bio 160/220 binder, with higher replacement (21%) by TOP, appeared to be worse than its reference counterpart in terms of fatigue resistance at 10°C (almost halved N_f at 2.5% strain). For a more comprehensive comparison, Figures 9 and 10 show the fatigue resistance of the analysed binders over a larger range of shear strains. It is indicated that there are certain differences in fatigue resistance between the bio-extended binders and their reference counterparts, depending on the shear strain level, replacement rate by bio-oil, and temperature. The aged Bio 70/100 binder did not show significantly worse fatigue resistance than its reference counterpart at 25°C. But at 10°C, the aged Bio 160/220 binder appeared to be less resistant to fatigue damages than the reference 160/220 bitumen at small strains. Considering the possible uncertainty of LAS tests as well as the complexity and variability of asphalt material, the significance of this difference in binder fatigue resistance needed to be further assessed with an investigation of asphalt mixtures to verify if it is significant enough to affect the asphalt mixture performance.

3.2. Fatigue resistance of asphalt mixtures

To verify the effect of difference in fatigue resistance between binders described above, AG16 asphalt mixtures that are mainly used for asphalt base course layers were prepared with the 160/220 binders. The motivation for investigating this type of mixture and this grade of binder was that they are more commonly exposed to high risk of fatigue damages and the binder analyses had raised more concerns about their fatigue resistance. Compacted specimens of AG16 mixtures were first aged in laboratory (see the Appendix) and then analysed for fatigue resistance at 10°C according to EN 12697-24:2018 Annex E (indirect tensile test on cylindrical shaped specimens). The test results are presented in Figure 11.

The fatigue resistance results of asphalt mixtures agreed well with the binder analysis results in Figure 10. At 10°C, the aged AG16 mixture with Bio 160/220 binder also appeared to be less

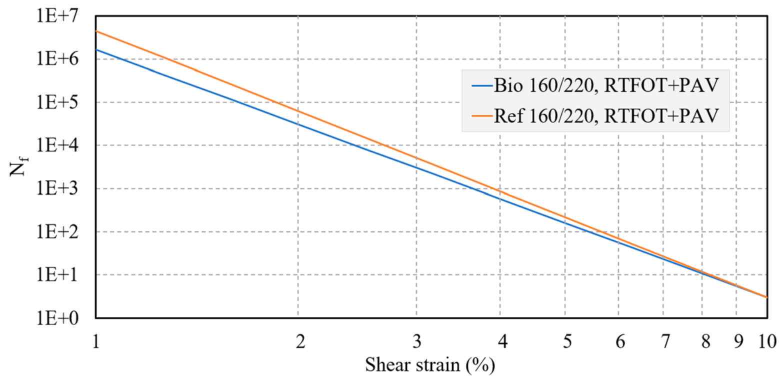

Figure 10. Fatigue resistance of the aged 160/220 binders by LAS tests at 10°C.

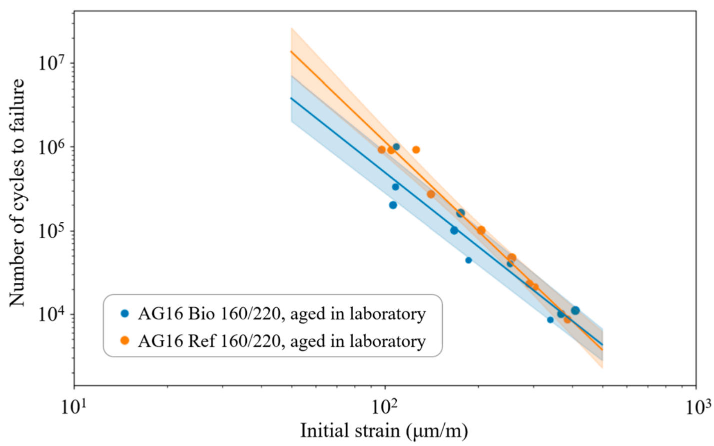

Figure 11. Fatigue resistance of the aged AG16 asphalt mixtures with 160/220 binders at 10°C (colour-shaded areas represent the 95% confidence intervals).

resistant to fatigue damages than the mixture with reference 160/220 bitumen at small strains (tensile strains for asphalt mixtures). In Figure 11, the colour-shaded areas represent the 95% confidence intervals. At high strains, the asphalt mixture with bio-extended binder did not show significantly different fatigue resistance from its reference counterpart. As the strain level reduces, their difference in fatigue resistance would start to appear. However, it is worth noting that the confidence level for this difference would be lower than 95% within the investigated range of tensile strains.

One critical factor affecting the fatigue resistance of asphalt mixture is the modulus. Increasing the mixture modulus reduces the fatigue resistance (Sudarsanan & Kim, 2022). Table 4 lists the stiffness modulus of the AG16 mixtures at 10°C before and after laboratory ageing. It is indicated that the AG16 mixture (in dry state) with Bio 160/220 binder had higher stiffness modulus than its reference counterpart at 10°C under the loading condition of load time 0.1 s and rest time 2.9 s. After laboratory ageing, the stiffness modulus of both asphalt mixtures increased, indicating the hardening due to ageing. Their increase rate was similar to each other (both about 40% increase), which led to the stiffness modulus of the aged AG16 mixture with Bio 160/220 binder remaining higher than its reference counterpart. This agreed well with the binder |G*| results in Figure 6 and might explain the slight difference between the aged AG16 mixtures in fatigue resistance in Figure 11.

Table 4. Stiffness modulus of the AG16 asphalt mixtures with 160/220 binders at 10°C before and after laboratory ageing.

	Stiffness modulus at 10°C (MPa)	
Asphalt mixtures	Fresh	Aged in laboratory
AG16 Bio 160/220 (dry state)	5868	8239
AG16 Ref 160/220 (dry state)	4565	6432

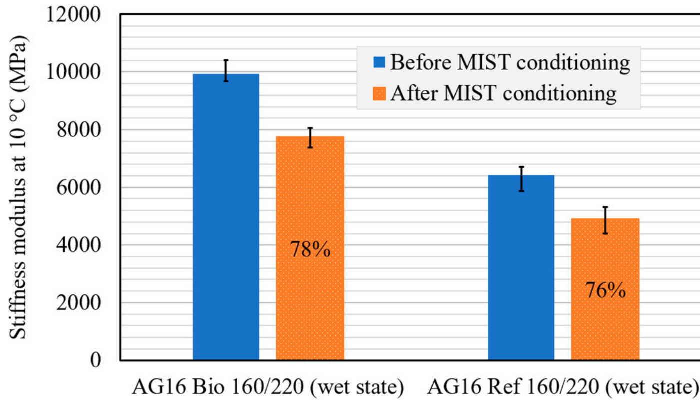

Figure 12. Stiffness modulus of the AG16 asphalt mixtures with 160/220 binders at 10°C before and after MIST conditioning (error bars represent the minimum and maximum values).

3.3. Water sensitivity by MIST

To evaluate the water sensitivity of asphalt mixtures, MIST was used to execute an accelerated moisture conditioning method with compacted specimens under cyclic pore pressure generation at an elevated temperature, thereby simulating long-term moisture damages. It is expected that the MIST conditioning would weaken the specimens and reduce the material integrity. By analysing the change in the mechanical properties before and after MIST conditioning, the sensitivity of different asphalt mixtures to water could be assessed. In this study, AG16 asphalt mixtures with 160/220 binders were conditioned by MIST and tested for stiffness modulus change in wet state at 10°C according to EN 12697-26:2018+A1:2022 Annex C (indirect tensile test on cylindrical shaped specimens). This type of asphalt mixture contains a relatively high amount of plant-based bio-oil (TOP), expectedly showing relatively significant effect of the bio-oil on water sensitivity of asphalt mixtures. The test results are presented in Figure 12.

The test results indicate that, in wet state, the AG16 mixture with Bio 160/220 binder also had higher stiffness modulus than its reference counterpart at 10°C under the specified loading condition (load time 0.1 s and rest time 2.9 s). After MIST conditioning at 40°C with 12,000 cycles and 0.28 MPa pore pressure, the stiffness modulus of both asphalt mixtures decreased, indicating the degradation of material integrity. Their reduction rate was similar to each other (22% versus 24%), which suggests similar water sensitivity of the asphalt mixtures although their initial stiffness modulus levels were different.

3.4. Combined effect of ageing and water

As both ageing and water can affect the mechanical properties of asphalt mixtures, their combined effect was assessed by dynamic mechanical analyses. Figures 13 and 14 present the master curves for dynamic modulus |E*| and phase angle φ of the AG16 asphalt mixtures with 160/220 binders at reference temperature 10°C. In the high-frequency (corresponding to low-temperature) range, the fresh

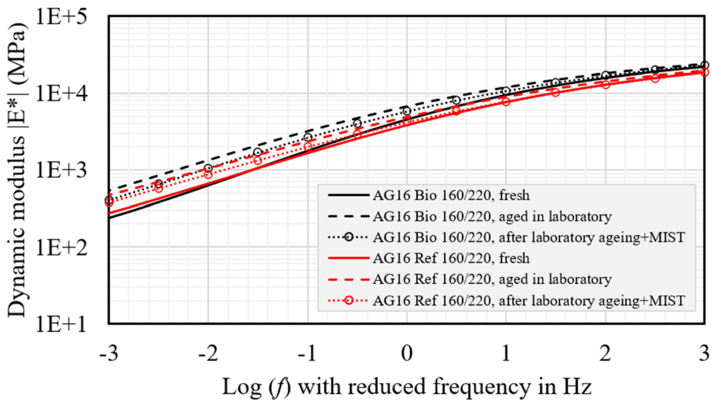

Figure 13. Dynamic modulus |E*| master curves of the AG16 asphalt mixtures with 160/220 binders at reference temperature 10°C.

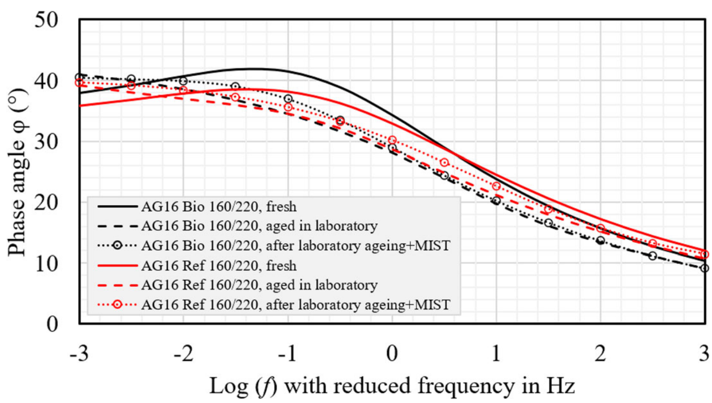

Figure 14. Phase angle φ master curves of the AG16 asphalt mixtures with 160/220 binders at reference temperature 10°C.

AG16 mixture with Bio 160/220 binder had higher dynamic modulus |E*| than its reference counterpart, indicating a stiffening effect. However, as the frequency decreases, its |E*| also decreases and is surpassed by the reference counterpart at very low frequencies (namely the stiffening effect ceases). After laboratory ageing, |E*| of the asphalt mixtures increased. The aged mixtures showed very similar relationship of |E*| as the fresh mixtures but at an increased level and with an eventual surpassing (stiffening effect ceasing) at even lower frequencies. The |E*| results of aged asphalt mixtures agreed well with the binder |G*| results in Figure 6. The further MIST conditioning reduced |E*| of the aged asphalt mixtures, but the |E*| values were still higher than that of the fresh mixtures. After laboratory ageing and MIST, the AG16 mixture with Bio 160/220 binder had higher |E*| than its reference counterpart (namely a stiffening effect) in almost the entire analysed frequency range.

As for phase angle φ (Figure 14), in the low-frequency (corresponding to high-temperature) range, the AG16 mixture with Bio 160/220 binder had higher φ than its reference counterpart. At very high frequencies (corresponding to very low temperatures), however, the bio-extended 160/220 binder led to lower φ of the asphalt mixture than the reference. After ageing, the φ of asphalt mixtures decreased in the high-frequency range and its peak (Zhu et al., 2022) moved towards to the low-frequency end. The aged mixtures showed very similar relationship of φ as the fresh mixtures but at a reduced level and with a shift towards lower frequencies. Similar as for |E*|, the further MIST conditioning also reversed some of the φ changes due to ageing. This resulted in φ master curves after laboratory ageing and MIST to appear between the fresh and aged mixtures, while the relationship of φ master curves between the

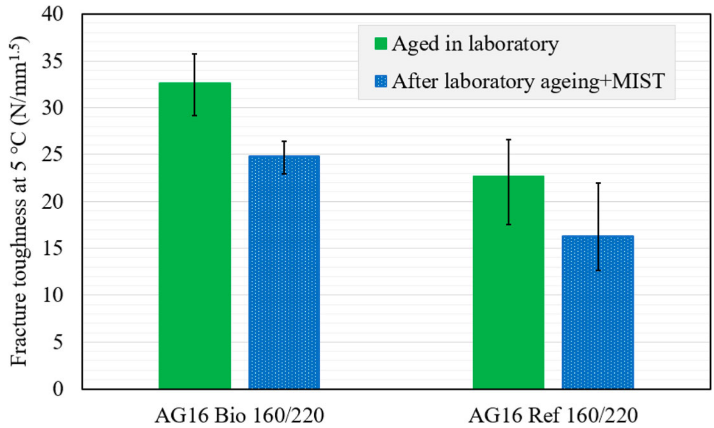

Figure 15. Fracture toughness of the AG16 asphalt mixtures with 160/220 binders at 5°C (error bars represent the minimum and maximum values).

bio-extended variant and its reference remained similar despite further changed level and reversed shift.

3.5. Crack propagation potential by SCB

The SCB test according to EN 12697-44:2019 applied a load from the top of a semi-circular specimen with a central notch. Due to the load, a tensile stress was generated and a crack started to propagate at the tip of the notch. By measuring the applied force and vertical displacement, the fracture toughness and other related parameters could be calculated as indicators for the asphalt mixture's crack propagation potential. In this study, compacted specimens of AG16 asphalt mixtures with 160/220 binders were firstly aged in laboratory (see the Appendix) and then analysed with the SCB test method at 5°C. This type of asphalt mixture and this grade of binder are mainly used for asphalt base course layers and commonly exposed to high risk of fatigue cracks. Moreover, some aged specimens were further conditioned with the MIST and these specimens after both laboratory ageing and MIST conditioning were also analysed with the SCB test method at 5°C. The calculation results of fracture toughness of the investigated asphalt mixtures according to EN 12697-44:2019 are shown in Figure 15.

The calculation results indicate that the aged AG16 mixture with Bio 160/220 binder had significantly higher fracture toughness than its reference counterpart at 5°C. After MIST conditioning at 40°C with 12,000 cycles and 0.28 MPa pore pressure, the fracture toughness of both asphalt mixtures decreased, indicating the impact of moisture damage on asphalt mixtures' crack propagation resistance. After both laboratory ageing and MIST conditioning, the fracture toughness of the AG16 asphalt mixture with Bio 160/220 binder was still higher than its reference counterpart. It is also worth noting that the fracture toughness of the AG16 asphalt mixture with Bio 160/220 binder after both laboratory ageing and MIST conditioning was even higher than that of the aged AG16 mixture with Ref 160/220 binder before MIST conditioning. However, although the European standard EN 12697-44:2019 adopts the fracture toughness as the primary test result, it does not seem to disclose any information other than the maximum force during the SCB test and the strength of the test specimen. There is a linear relationship between the fracture toughness of the AG16 asphalt mixtures with 160/220 binders at 5°C and the maximum force during the SCB test, as shown in Figure 16. Besides the strength and fracture toughness, there are other aspects and other related parameters to asphalt mixtures' crack propagation resistance (Meng et al., 2023).

Another relevant parameter is the post-peak displacement. It is defined as the displacement from the maximum force until failure. A small post-peak displacement usually indicates a fast crack propagation through the test specimen, thus suggesting a brittle asphalt mixture. In contrast, a large

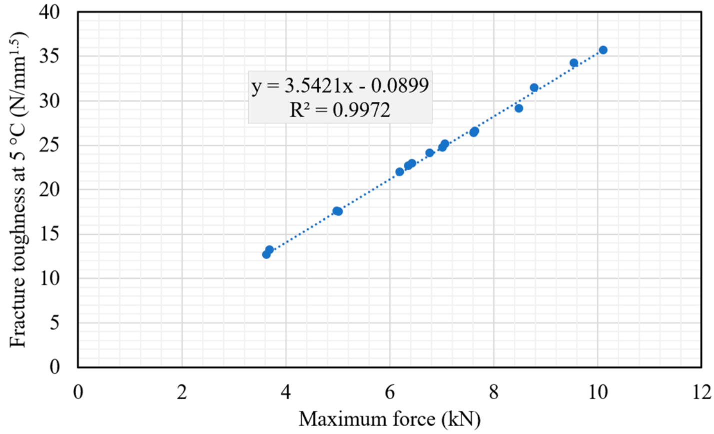

Figure 16. Linear relationship between the fracture toughness of AG16 asphalt mixtures with 160/220 binders at 5 °C and the maximum force during SCB test.

post-peak displacement is often a sign of slow crack propagation and a flexible asphalt mixture (Kaseer et al., 2018). Figure 17 presents the force-displacement curves of the analysed specimens (four replicates under each test condition) during SCB tests at 5°C. The differences in maximum force between various test conditions are evident. Furthermore, the laboratory aged AG16 mixture with Bio 160/220 binder showed very different post-peak curves – almost instant failure after the maximum force. It reveals a fast crack propagation through the mixture and a brittle failure. However, after the further MIST conditioning, the bio-extended variant was weakened (lower strength) and did not show the brittleness any longer. This might be attributed to the reduced material integrity after MIST conditioning, which made the asphalt mixture weaker but allowed larger deformation before the failure. The specific impacts of this on the pavement performance are worth future studies.

The results of post-peak displacement of all investigated asphalt mixtures are shown in Figure 18. It indicates that the aged AG16 mixture with Bio 160/220 binder had significantly lower post-peak displacement than its reference counterpart at 5°C, suggesting its brittleness. After the MIST conditioning, the post-peak displacement of both asphalt mixtures increased, possibly due to the impact of moisture damage on material integrity. After both laboratory ageing and MIST conditioning, the post-peak displacement of the AG16 asphalt mixture with Bio 160/220 binder was still lower than its reference counterpart.

Combining both the force and displacement during SCB test, the fracture energy is defined as the work density (unit: J/m^2) required to initiate and propagate the crack in the specimen until fracture. For specimens of identical dimension (such as standard specimens), it corresponds to the area under the force–displacement curve. The fracture energy of the AG16 asphalt mixtures in this study was calculated. The calculation results are shown in Figure 19. It indicates that the aged AG16 mixture with Bio 160/220 binder had slightly lower fracture energy than its reference counterpart at 5°C. After the MIST conditioning, its fracture energy increased, due to the reduced brittleness. Meanwhile, the fracture energy of the reference mixture decreased because of the weakening effect of the MIST conditioning. Overall, the SCB tests at 5°C showed that the aged AG16 mixture with bio-extended binder (21% TOP bio-oil replacement) had higher strength to resist cracks than the reference mixture, but its higher brittleness would lead to a faster crack propagation once the crack starts to propagate. After moisture conditioning, the asphalt mixture with bio-extended binder would become weaker but less brittle.

3.6. Full-scale APT

APT is a method to determine the long-term performance of pavements in a reduced period of time. In this study, a bio-extended section with bio-extended binders in asphalt mixtures and a reference

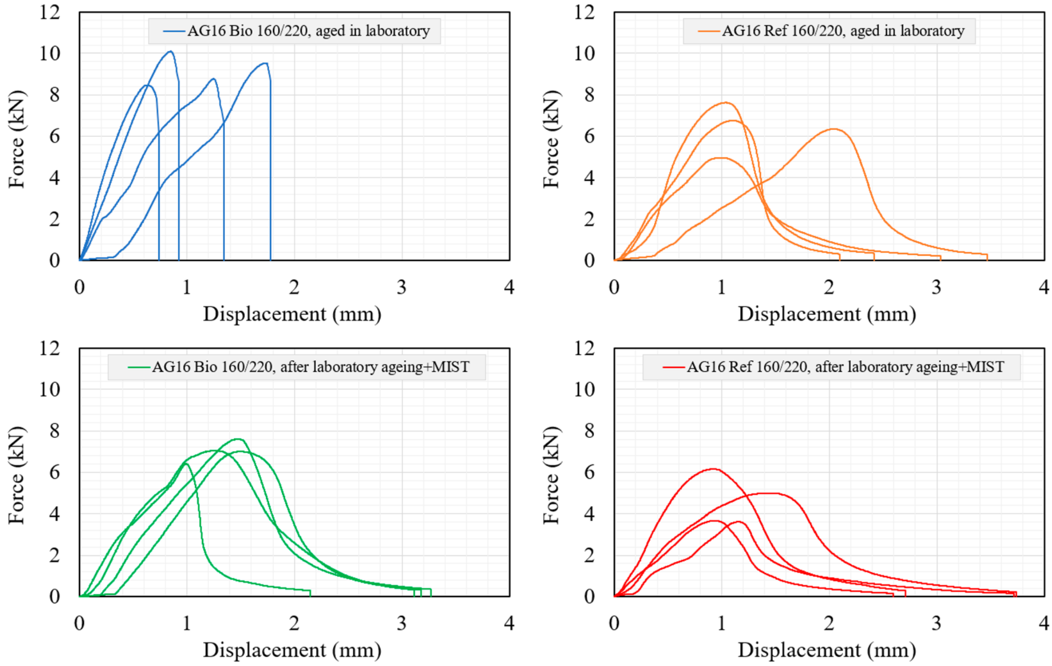

Figure 17. Force–displacement curves of the AG16 asphalt mixtures with 160/220 binders during SCB tests at 5°C.

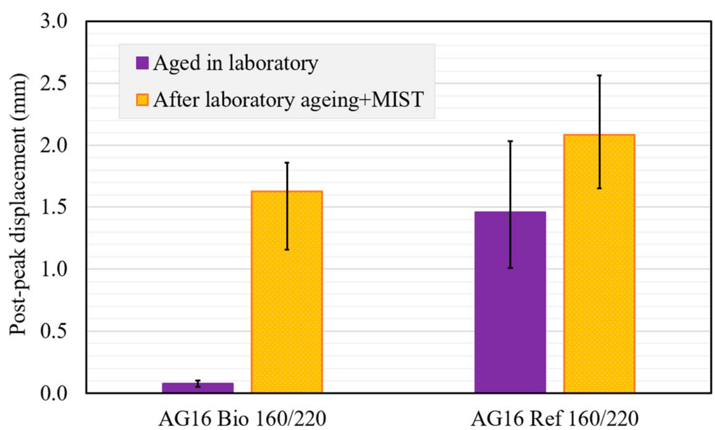

Figure 18. Post-peak displacement of the AG16 asphalt mixtures with 160/220 binders at 5°C (error bars represent the minimum and maximum values).

section with conventional bitumen were constructed and tested using APT in full scale. A total of 500,000 loading cycles was applied to the test sections. No special performance issues were identified for the two sections during the test. The average rut depth of the sections was regularly measured with a laser sensor from the pavement surface. The measurement results are presented in Figure 20.

The APT results indicate that the rut depth of the bio-extended section was constantly smaller than that of the reference section. Considering the same layer thickness and test conditions of the two sections, their difference in rut depth could be attributed to the different materials for asphalt layers. The two layers of asphalt mixtures with bio-extended binders were more resistant to permanent deformation than the reference, likely due to the higher stiffness of the bio-extended variant (primarily the asphalt base course layer with AG16 Bio 160/220 containing 21% plant-based bio-oil TOP) around the

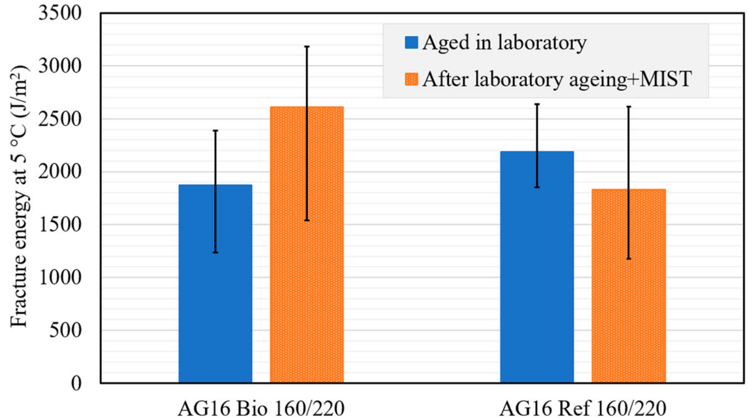

Figure 19. Fracture energy of the AG16 asphalt mixtures with 160/220 binders at 5°C (error bars represent the minimum and maximum values).

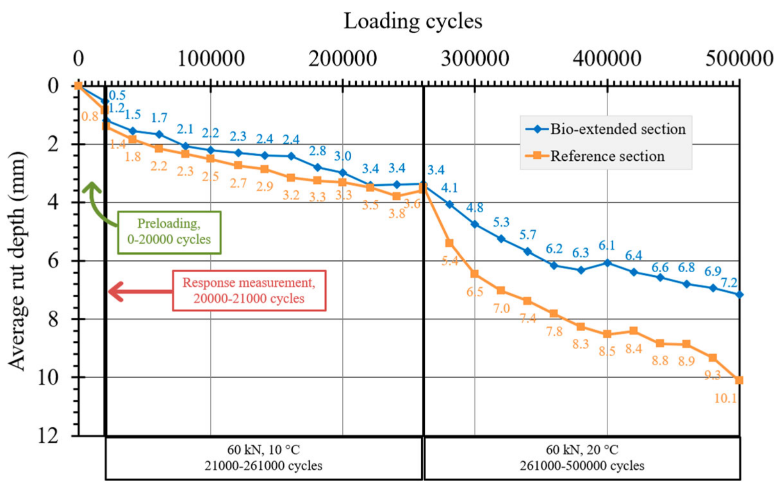

Figure 20. Average rut depth of the test sections during APT.

test temperatures. Although the APT did not involve long-term ageing, the test results provide confidence for testing the asphalt mixtures containing bio-extended bituminous binders in an even larger scale with test roads under real traffic and climate conditions.

4. Conclusion

This study evaluated the long-term performance of bio-extended bituminous binders containing a plant-based bio-oil (TOP) and their asphalt mixtures. The analyses were conducted both in laboratory and in full scale. Based on the above-described results and discussion, the following conclusions could be drawn:

(1) With 5% replacement by the plant-based bio-oil TOP, the laboratory aged Bio 70/100 binder did not show significantly worse fatigue resistance than its reference counterpart at 25°C. But at 10°C, the aged Bio 160/220 binder, which contained 21% TOP, appeared to be less resistant to fatigue damages than the reference 160/220 bitumen at small strains. This was confirmed by the fatigue resistance analyses of laboratory aged asphalt mixtures at a confidence level of

lower than 95%. At high strains, the asphalt mixture with bio-extended binder did not show significantly different fatigue resistance from its reference counterpart.

(2) The AG16 mixture with Bio 160/220 binder had significantly higher stiffness modulus than its reference counterpart at 10°C. On the one hand, the laboratory ageing increased its stiffness modulus by about 40%, a similar level as its reference counterpart. On the other hand, after MIST conditioning, its stiffness modulus decreased to a similar extent as the reference mixture. This suggests that they had similar water sensitivity, although the initial stiffness modulus of asphalt mixture with plant-based bio-oil (TOP) was higher.

(3) The laboratory ageing hardened the asphalt mixtures and reduced their flexibility. The further MIST conditioning reversed some of the changes in dynamic modulus and phase angle due to ageing. This might be due to the damage caused by water, leading to reduced material integrity after the MIST conditioning. These effects were similar for both the asphalt mixture with bio-extended binder and the reference mixture, although they had some differences in mechanical properties in fresh state.

(4) The laboratory aged AG16 mixture with bio-extended 160/220 binder (21% TOP bio-oil replacement) had higher strength to resist cracks than the reference mixture at 5°C, but its higher brittleness would lead to a faster crack propagation once the crack starts to propagate. This echoes the shear stress–strain curve of the aged 160/220 binder during LAS test at 10°C, which suggested that the bio-extended 160/220 binder after long-term ageing would withstand higher stress than the reference binder, but it would exhibit faster development of failure after certain damage accumulation.

(5) After further MIST conditioning, the aged AG16 mixture with Bio 160/220 binder was weakened (strength became lower) and did not show the brittleness any longer. This might be attributed to the reduced material integrity after MIST conditioning, which made the asphalt mixture weaker but allowed larger deformation before the failure. But its post-peak displacement was still lower than its reference counterpart while its fracture energy became higher than the reference mixture.

(6) The full-scale APT results showed that the two layers of asphalt mixtures with bio-extended binders were more resistant to permanent deformation than the reference. They support testing the asphalt mixtures containing bio-extended bituminous binders in a larger scale with test roads in future studies.

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Disclosure statement

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Appendix

Procedure for laboratory ageing of compacted asphalt mixture specimens

In this study, compacted specimens of AG16 mixtures for asphalt base course layer were aged in laboratory according to CEN/TS 12697-52:2017 (Procedure B.1 at 65°C for 15 days). The ageing temperature and duration were determined based on actual measurement data of asphalt mixtures made of similar bituminous binders as in this study. The determination is also supported by field data from test roads under real conditions. Figure A1 shows the stiffness modulus evolution of two types of asphalt mixtures made of conventional 70/100 bitumen and polymer modified bitumen (PMB, Nypol 67) respectively. The figure also shows the percent increase in stiffness modulus of the mixtures. The stiffness modulus values (each value as the average of three specimens) were determined at 10°C using the indirect tensile test method according to EN 12697-26:2018+A1:2022 Annex C (specimen diameter 100 mm and thickness 50 mm, load time 0.1 s and rest time 2.9 s). The measurements were conducted during a period of about 7 years. It can be observed in Figure A1 that, over the measurement period, the stiffness modulus of the asphalt mixtures increased by about 30% for the mixture with conventional 70/100 bitumen and by about 15% for the one with PMB. The laboratory aging procedure employed in this study attempted to attain these levels of stiffness modulus increase. Figure A2 shows the stiffness modulus increases as a function of the number of days the compacted specimens of asphalt mixtures were stored in an oven at 65°C. The aging temperature of 65°C was selected to avoid damaging the specimens made of softer binders, such as 160/220 penetration grade binders. It is obvious in Figure A2 that 15 days of ageing the compacted specimens at 65°C resulted in levels of stiffness modulus increase close to that observed in Figure A1.

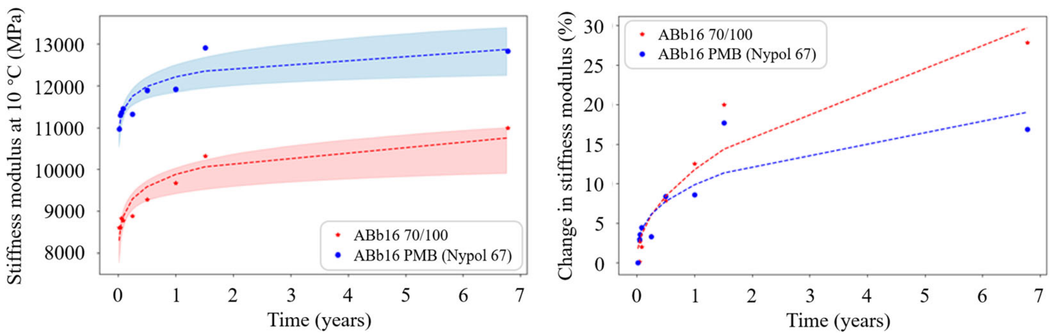

Figure A1. Stiffness modulus evolution of two types of asphalt mixtures over a period of about 7 years (colour-shaded areas represent the 95% confidence intervals).

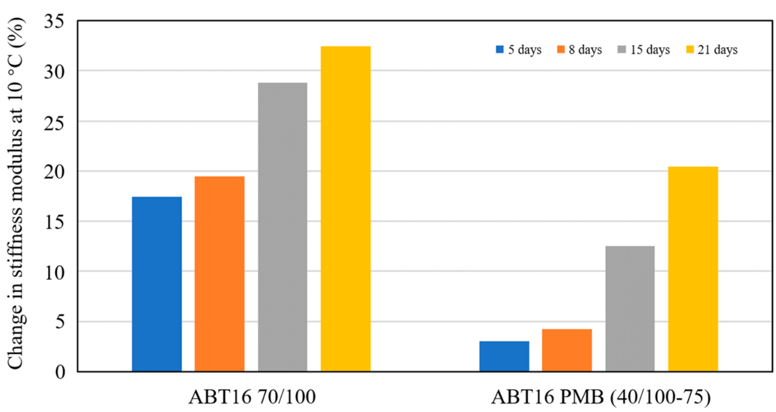

Figure A2. Stiffness modulus increases as a function of the number of days the compacted specimens of asphalt mixtures were stored in an oven at 65°C.