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Effect analysis of using tall oil pitch (TOP) to partially extend bitumen in asphalt pavements: comparison of different TOPs

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ABSTRACT
Tall oil pitch (TOP) is widely available as a by-product, but not sufficiently valorized and has potential as a bitumen extender. In this research, three types of TOP were used to prepare bio-extended binders of two grades based on the penetration and softening point of target neat binders. The chemical, rheological, and fatigue behaviour of bio-based binders and reference binders were investigated and compared. The optimum TOP contents were inversely determined based on penetration and softening point fitting equations. New C = O and C–O–C stretching can be found in bio-based binders. Different TOPs can significantly increase the viscous response of asphalt binders, resulting in better low-temperature properties but reduced high-temperature properties. A high amount of TOP can significantly reduce fatigue resistance. This work reveals that TOP can work as extenders for bitumen, but the performance of bio-based binders is affected by the TOP type and amount.

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KEYWORDS
Tall oil pitch; bio-based binders; chemical components; rheological properties; fatigue resistance

1. Introduction

As the global energy crisis and environmental pollution problems become increasingly serious, the use of traditional petroleum resources has brought about tremendous ecological and economic pressure (Czúcz et al., 2010; Ma et al., 2025; Zhang et al., 2024b). Asphalt road is a main carrier of the modern transportation and asphalt mixtures are usually composed of petroleum-based bitumen and natural aggregates. With the continuous construction and maintenance of roads, the demand for raw materials, especially bitumen, the main material that bind the aggregates together (Zhou et al., 2024; Zhou et al., 2025), constantly increases. As an important road construction material, although petroleum bitumen has an irreplaceable position in infrastructure construction, the environmental burden caused by its extraction and use cannot be ignored (Liu et al., 2020). The production of petroleum bitumen consumes significant amounts of non-renewable resources and generates greenhouse gases and pollutants although it can avoid sending short residues to crackers (Thives & Ghisi, 2017), which are associated with even higher greenhouse gas emissions. Therefore, in order to reduce the consumption of petroleum bitumen, it is urgent to find alternatives to it while extending the service life of roads.

To address this issue, the scientific and engineering communities are actively exploring sustainable materials to reduce their dependence on petroleum resources such as reclaimed asphalt pavement

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(RAP) and biomass materials (Wang et al., 2025; Gaudenzi et al., 2023; Musco et al., 2024). With this background, biomass materials have received more and more attention in recent years as an ideal modifier or extender for bitumen. Bio-oil extracted from biomass as a renewable resource has the advantages of wide availability, large reserves, low prices, and easy handling (Dugan et al., 2020). Bio-oil can be divided into animal and vegetable oils, which are mainly derived from crop residues, wood wastes, waste oils and fats, animal manure and so on (Barzegari & Solaimanian, 2020; Jayasinghe & Hawboldt, 2012). They are usually obtained by pyrolytic, catalytic or other transformation processes of biomass resources and have a composition similar to bitumen (Pandey et al., 2015; Yang et al., 2017).

The new binders prepared by mixing neat bitumen with a larger amount of biomaterials (usually above 5%) are known as bio-extended binders. Depending on the role of the biomaterialsl in the binder and mixture, it might be categorised as a modifier or rejuvenator as well. Fini et al. attempted to prepare bio-oil from swine manure and use it to improve asphalt binders' properties and found that the low-temperature cracking temperature of bio-binders reduced by 5.3°C compared to neat binders, but the high-temperature complex modulus also reduced significantly when the bio-oil content was above 5% (Fini et al., 2011; Fini et al., 2012). Yang et al. investigated the use of a wood-based bio-oil containing a large number of oxygen-related functional groups and concluded that the aging speed of bio-binders was faster than neat binders (Yang et al., 2017). Alattieh et al. claimed that the high-temperature rutting resistance of bio-binders containing date-seed-oil was reduced but the fatigue resistance was enhanced (Alattieh et al., 2020). These studies pointed to the fact that bio-oil increases penetration and reduces the high-temperature properties of asphalt binders, but improves the low-temperature properties of bio-based binders. The difference in performance of bio-based binders is attributed to the type of bio-oil, and the reduced properties can be further improved by other modifiers, such as styrene–butadiene–styrene polymer together with bio-oil (Kazemi et al., 2024; Liu et al., 2021; Meng et al., 2022; Yang et al., 2022). In addition, bio-oil also can act as rejuvenator to recycle aged asphalt mixtures. Mirhosseini et al. concluded that the use of 5%—10% date-seed oil significantly restored the performance of asphalt mixtures after eight years of service, especially the fatigue properties. The authors also claimed that the rutting resistance was close to that of the initial asphalt mixtures, although it was slightly reduced (Mirhosseini et al., 2019). A study by Foroutan et al. showed that the use of bio-oil restored 37% of the fatigue performance and 4% of the moisture resistance of asphalt mixtures containing 90% reclaimed asphalt pavement (RAP) (Foroutan Mirhosseini et al., 2020).

These studies on biomaterials used different bio-oils, as dictated by the biogeography of the region in which the study was conducted. For example, the study by Fini et al. was based on the background that only 5% of U.S. farmland uses swine manure as a fertiliser (Fini et al., 2011). Forest is a valuable resource in the Nordic countries, where the forest coverage is much higher than in the rest of the European Union, e.g. 73% in Finland, and 68% in Sweden (Brizga & Räty, 2024). While forest raw materials are primarily used for manufacturing daily supplies such as wood and paper, various forest-derived by-products are also generated, including crude tall oil (CTO). CTO is obtained as a by-product of the kraft pulping process and undergoes further distillation to produce fractions such as tall oil rosin, tall oil fatty acids, and tall oil pitch (TOP), the latter being a low-value residue with potential applications in various industries (Bajwa et al., 2019; Mahmood et al., 2016). New utilisation of these wood-based by-products, especially in large volumes, would increase the added value of the forest raw materials. Cavalli et al. proposed to use TOP as a rejuvenator to reclaim RAP (Cavalli et al., 2019), and the results indicated that TOP can improve the mechanical and fracture resilience of RAP. This shows that it is feasible to use TOP in road engineering. However, the related research is still not sufficiently developed according to documented literature (Lu et al., 2020; Peltonen, 1992; Porot & Haslam, 2020). Past studies mainly used biomaterials as bitumen modifiers or rejuvenators, while the high-temperature performance is generally reduced, making it necessary to use other modifiers to balance the performance of bio-based binders. Biomaterialss usually have a similar composition as bitumen, which makes the two substances compatible (Ameri et al., 2023; Yang et al., 2017). If the bitumen of a harder grade can be directly extended by TOP to prepare bio-based binders of certain softer grades, it will not

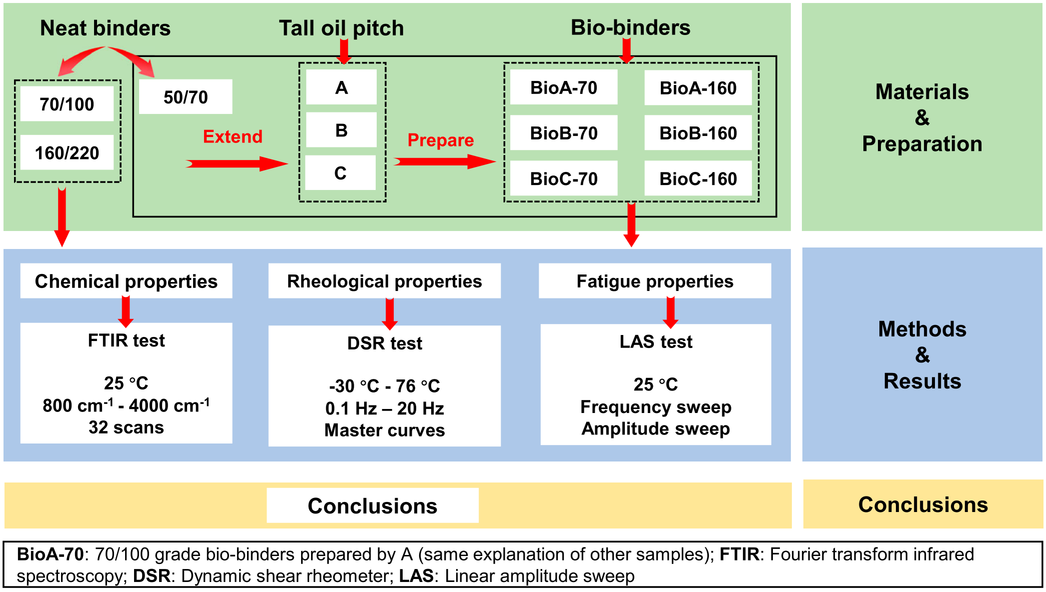

Figure 1. Flow chart of this work.

only reduce consumption of the petroleum resource, add values to the wood-based by-product, but also solve the problem of unbalanced performance of bio-based binders.

With this objective, this work aims to extend 50/70 penetration-grade neat bitumen with three different types of TOPs (A, B, and C) to prepare 70/100-grade and 160/220-grade bio-based binders. The preparation is conducted based on the penetration and softening point of the target neat binders. Different properties of bio-based binders including chemical, rheological, and fatigue behaviour are also studied and compared to the target neat binders to ensure that the properties of bio-based binders do not differ too much. Following are the results of these experiments and the corresponding discussions, and the conclusions are drawn based on the results and discussions. Finally, this work also summarises the research and recommends future work. The flow chart of this work is shown in Figure 1.

2. Materials and methods

2.1. Raw materials

The raw materials in this work include neat asphalt binders (conventional bitumen) and Tall Oil Pitch (TOP). 50/70 neat bitumen was used as the base for preparing bio-extended binders. 70/100 – and 160/220-grade asphalt binders are the common binders for road paving in the Nordic region due to their special geographical and environmental characteristics, so they served as target reference binders, which were labelled as Ref-70 and Ref-160. Three different TOPs (TOPA, TOPB, and TOPC) were employed to partially extend the 50/70 grade bitumen to prepare the bio-based binders. TOP-A and TOP-B were provided by Finland, and TOP-C was provided by Sweden. TOP is solid or semi-solid at room temperature depending on its viscosity, and it has a good flowability after heating. TOPA is very soft and close to a fluid state at room temperature compared to TOPB and TOPC. The selected TOPs in this research are dark brown and all semi-solid, they were first heated to a fluid state and blended with the base bitumen. The basic physical properties of TOPs are presented in Table 1. The bio-extended binders with the target grades were marked as BioA-70, BioB-70, BioC-70, BioA-160, BioB-160, and BioC-160.

Table 1. Physical properties of different TOPs.

Properties	TOP-A	TOP-B	TOP-C
State	Dark amber viscous liquid	Dark amber viscous liquid	Dark amber viscous liquid
Colour	Dark brown colour	Dark brown colour	Dark brown colour
Odour	Mild	Mild	Mild
Solubility in water	Insoluble	Insoluble	Insoluble
Flash point /°C	224	261	> 200
Pour point /°C	36	-	-
Ignition point /°C	-	283	-
Boiling /°C	210	-	-
Auto-ignition point /°C	370	392	-
Softening point /°C	> 19	> 19	-
Ash / %	0.5	-	-
Viscosity / mPa∗s	-	-	1060 (60°C)
Acid number / mg KOH/g	20	70	33
Density / kg/m^3	1017 (15°C)	1000–1050 (20°C)	995 (50°C)

2.2. Methodology

2.2.1. Penetration and softening point

The penetration and softening point tests were conducted to determine the optimum content of TOPs based on European standards (EN-1426, 2015; EN-1427, 2015). The penetration was measured at 25°C, and the softening point was determined with the Ring and Ball method. Detailed steps are described in the standards.

2.2.2. Fourier transform infrared spectroscopy (FTIR) test

FTIR test is used to characterise the chemical composition of bituminous binders and TOPs based on spectra. The different chemical bonds and functional groups of test samples show different intensities of absorption of infrared light, which can be reflected as different peaks in their spectra (Porot et al., 2023; Zhang et al., 2024a). The testing range in this work was from 800 cm^{-1}–4000 cm^{-1} with 32 times of scanning. Nine replicates were used to perform the FTIR test to minimise testing errors.

2.2.3. Temperature-frequency sweep test

The rheological properties of bituminous binders were evaluated based on the temperature-frequency sweep test, conducted by dynamic shear rheometer (DSR) machines. Three temperature ranges, low, intermediate and high temperatures, were selected to perform the test based on different sizes of parallel plate clamps. The clamps with a 4-mm diameter and 1.75-mm gap (PP04) were used for low-temperature tests. Clamps with an 8-mm diameter and 2-mm gap (PP08) and clamps with a 25-mm diameter and 1-mm gap (PP25) were used for intermediate- and high-temperature tests, respectively. A DSR machine and relevant parallel clamps are presented in Figure 2. The testing temperatures were selected from −30°C to 76°C and the testing frequencies were selected between 0.1 and 20 Hz, based on different laboratories. The detailed parameters for temperature-frequency sweep are presented in Table 2. In addition, all the temperature-frequency sweep tests were performed within the linear viscoelastic range, the recommended strains for PP25, PP08, and PP04 are 1%, 0.1%, and 0.05%, respectively.

2.2.4. Linear amplitude sweep (LAS) test

LAS is employed to estimate the fatigue properties of asphalt binders using a DSR device with 8-mm-diameter parallel plate clamps. This test consists of two procedures. A frequency sweep was first conducted to evaluate the linear viscoelastic properties of asphalt binders. The frequency was from 0.2 Hz to 30 Hz at 0.1% shear strain level in this stage. Then, an amplitude sweep was performed with linearly increasing amplitudes from 0.1% to 30% at a constant frequency of 10 Hz. This procedure was used to accelerate the cumulative damage of asphalt binders. The detailed procedures can

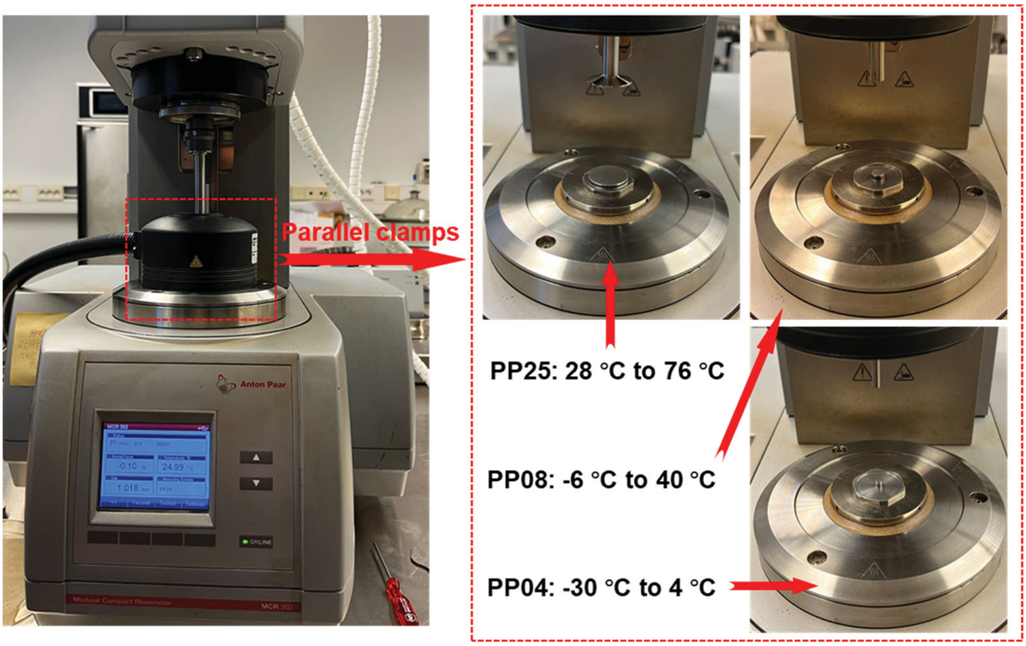

Figure 2. DSR machine and its parallel clamps.

Table 2. Temperature-frequency sweep parameters of different laboratories.

Laboratories	Low temperatures/°C	Mid temperatures/°C	High temperatures/°C	Frequencies/Hz/
VTI, Sweden (70/100 grade)	–	5, 10, 15, 20, 30, 40	40, 50, 60, 70	0.0159, 0.04, 0.06, 0.08, 0.1, 0.2, 0.4, 0.6, 0.8, 1, 2, 4, 6, 8, 10, 20
VTI, Sweden (160/220 grade)	–	5, 10, 15, 20, 30	30, 40, 50, 60	0.0159, 0.04, 0.06, 0.08, 0.1, 0.2, 0.4, 0.6, 0.8, 1, 2, 4, 6, 8, 10, 20
Aalto, Finland	−30, −24, −18, −12, −6, 0, 4	−4, 0, 6, 12, 18, 24, 28, 34, 40	28, 34, 40, 46, 52, 58, 64, 70, 76	0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1, 1.59, 2, 3, 4, 5, 6, 7, 8, 9, 10

be found in AASHTO T 391–20 (AASHTO-T391-20, 2020). The testing temperature was 25°C, and the binder samples were after long-term aging based on the AASHTO standard (AASHTO-R28-12, 2012).

The fatigue life (N_f) can be calculated as follows based on the Visco-Elastic Continuum Damage (VECD) Model (Hintz et al., 2011):

$$N_f = A_{35} \cdot (\gamma_{\max})^B \tag{1}$$

where, A_{35} and B are the model coefficients. A_{35} represents the ability of the material to maintain its integrity during cumulative damage. B means the sensitivity of the material to changes in shear strain. Hence, a higher A_{35} and lower B can make the material show better cumulative damage resistance.

3. Results and discussion

3.1. Formulation of bio-extended binders

In order to obtain bio-extended binders close to the target grades, 50/70 neat bitumen is partially extended by TOPs with contents of 5%, 10%, 15%, and 20% by weight of the blend, respectively. Then, the penetration and softening points of these bio-based binders are measured, as shown in Figures 3(a) and 3(b). These two parameters show the basic performance of the bitumen binders, especially penetration, which is used in Europe for the classification of bitumen (EN-12591, 2009). It can be observed that the penetration of the bio-based binders increases with the TOP content. BioB and BioC binders show very similar increasing trends, while BioA binders have higher penetration values. TOPA presents more viscous components at room temperature, and the penetration above 220 (0.1 mm, 25°C) could not be measured due to the small size of the sample container, which would damage the needle of the penetration device. The penetration values of BioA binders containing 15% and 20% TOP-A are much higher than 220 (0.1 mm, 25°C), so the contents of 4%, 5%, 10%, and 12% are re-selected. From Figure 3(b), the softening point of the bio-based binders reduces with the TOP content. The penetration and softening point results in Figures 3(a) and 3(b) show exponential and linear trend changes, respectively, so we fit them with the correlation functions. All the R^2 of the fitting curves are 0.98 or higher, suggesting that the fitting results are accurate. Therefore, the optimum content of TOPs can be deduced inversely from the penetration and softening point of the target reference bitumen by the fitting formula. With a 50/70 base bitumen, the optimum contents of TOPA, TOPB, and TOPC to match Ref-70 are 5%, 6%, and 5%, respectively; similarly, the optimum contents of TOPA, TOPB, and TOPC to match Ref-160 are 11%, 20%, and 21%, respectively. In order to validate the results, the penetration and softening points of the bio-based binders with the optimum TOP contents are measured and shown in Figures 3(c) and 3(d). The penetration of BioB and BioC binders in Figure 3(c) is very close to the target reference binders. Their penetration does not differ by more than 2 (0.1 mm, 25°C). The softening point in Figure 3(d) also has a small error, varying within a range of 1.6°C. However, the two properties of BioA binders show inconsistency, especially in penetration value. It can be observed that BioA-70 is softer than Ref-70 and BioA-160 is harder than Ref-160. The appropriateness of the dosage of TOPA needs to be discussed in the following section based on the analysis of other properties.

3.2. Chemical properties

The chemical properties of the bio-extended binders are evaluated based on the results from FTIR tests. The FTIR spectra of the binders are shown in Figures 4 and 5, associated with 70/100 target reference bitumen and 160/220 target reference bitumen, respectively. The FTIR spectra of bio-extended binders remained essentially the same as those of neat binders because the TOP and neat binders are all hydrocarbons and the TOP contents are limited in the binder. However, there are some differences in their absorbance peaks. From the figures, the main molecular vibrations of functional groups are the stretching and/or bending vibrations of C–H, S = O, and C = C bonds. The C–H stretching vibrations in $-CH_2$ and $-CH_3$ groups occur from 2800 cm^{-1}–3000 cm^{-1}, and the peaks at 1375 and 1450 cm^{-1} are associated with the C–H bending in $-CH_3$ and $-CH_2$ groups (Ma et al., 2023). The S = O stretching vibrations occur at the absorption peaks with around 1030 cm^{-1} of wavenumber. This peak indicates the relative content of oxidised sulphide (Ma et al., 2023). This peak area turns lower after adding the TOPs compared to neat binders, revealing a decrease in oxidised sulphide or sulfoxide compounds of bio-based binders. The peak at 1600 cm^{-1} is caused by C = C stretching (Ma et al., 2023). The area of this peak in Figures 4 and 5 does not change too much, indicating a similar relative C = C content in bio-based binders and neat binders. In addition to the changes in functional group content, the bio-based binders also generated some new absorption peaks, which are presented in Figure 4(b), Figure 5(b) and 5(c). For 70/100 and 160/220 grade bio-based binders, they both generated new absorption peaks of C = O stretching from 1650 cm^{-1} to 1800 cm^{-1}. However, this does not mean that a chemical reaction has occurred between the TOP and the bitumen to produce a new substance. The FTIR spectra of TOPs are presented in Figure 6. It can be found that TOP contain large amounts of fatty acids and/or fatty acid esters (Suota et al., 2019), which are not present in neat binders. This explains the new absorption peaks here. From Figure 5(c), more new peaks can be observed between 1050 and 1300 cm^{-1}, which can be assigned to the C–O–C stretching vibrations in oxygen-containing functional groups (e.g. esters) in TOPs from Figure 6 (Yang et al., 2017). These peaks are not observed in 70/100-grade bio-based binders with less TOP in Figure 4, which means that C–O–C stretching vibrations in bio-binders are related to the bio-oil content in bio-based binders.

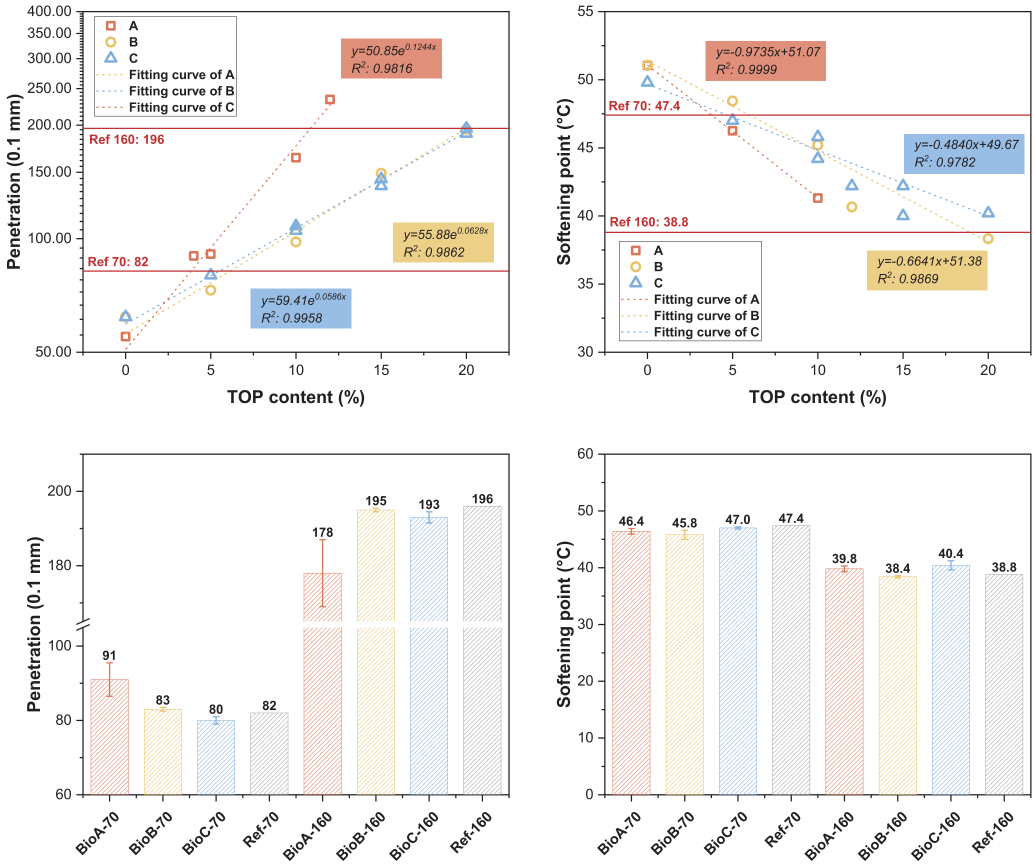

Figure 3. Penetration of bio-based binders, with different TOP contents (a), with optimum content (c), at 25°C; (b) Softening point of bio-based binders, with different TOP contents (b), with optimum content (d), using ring and ball method.

3.3. Rheological properties

3.3.1. Cole–Cole plot

The Cole–Cole plot is the preliminary analysis of asphalt binders' viscoelastic properties, which is plotted by storage modulus (G') versus loss modulus (G''), as shown in Figure 7. In a Cole–Cole plot, the elastic properties can be characterised by G', and the viscous properties can be represented by G''. When G' is higher than G'', it means that the composition of the asphalt binders is dominated by elasticity, and conversely, it is dominated by viscosity (Yuan et al., 2022). The elastic and viscous zones in Figure 7 are marked by blue and yellow backgrounds.

The Cole–Cole plots are presented in the low- (−30°C – 0°C), intermediate- (4°C – 40°C), and high-temperature (46°C – 76°C) ranges, respectively, due to the significant effect of temperature on binder modulus. At low temperatures, the storage and loss modulus of binders increases with the decline of the temperature in the initial period. The bio-extended binders are prepared by low-grade binders (50/70) and TOP, and the low-temperature performance of 50/70 binders is typically worse than high-grade binders. However, the addition of TOP significantly improves the viscosity of the bio-extended binders; it can be found that a small amount of TOP can make the bio-extended binders show similar or more viscous responses. Among these TOPs, TOPA shows the best improvement effect, the Cole–Cole plot of BioA is higher than that of other samples, revealing a larger G''. The viscoelastic transition of most of the binders occurs at intermediate temperatures, as presented in Figure 7(b). The viscoelastic transition temperatures of 70/100-grade binders are around 4°C. Meanwhile, the Cole–Cole plots in

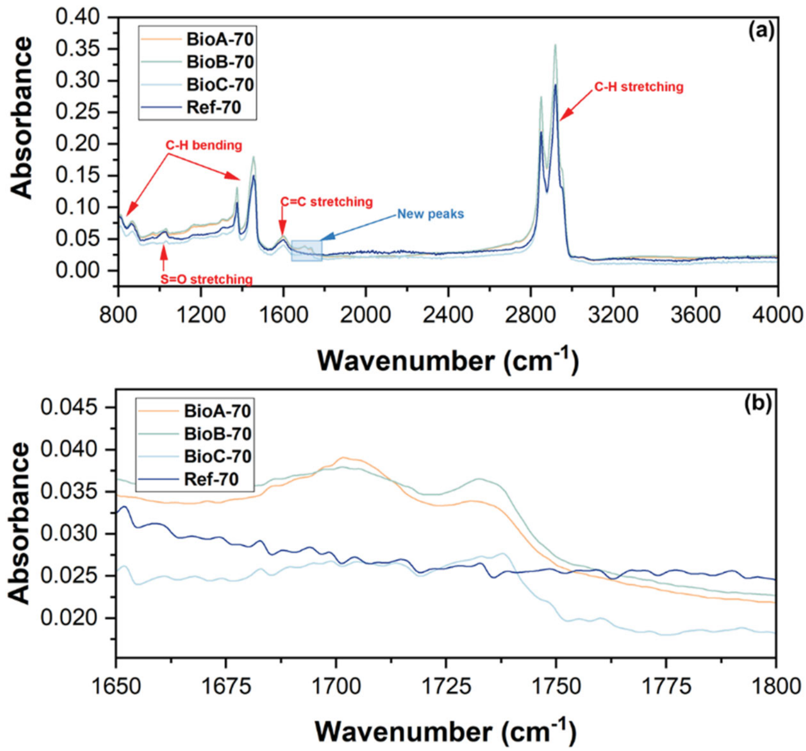

Figure 4. (a)FTIR spectra of 70/100 grade binders; (b)enlarged spectra of new peaks.

Figure 7(b) present a nearly linear trend with the temperature and the viscoelastic responses of bio-based binders are close to the target reference binders. At high temperatures, all the binders are in the viscous zone, and the viscoelastic responses are very similar.

3.3.2. Black diagram

Different TOP contents and binder types may result in different results of complex shear modulus (G^*) and phase angle (δ), causing various rheological behaviours. As reported in some previous works (del Barco Carrion et al., 2020; Wang et al., 2020; Wang et al., 2022), G^* varies in the range between 1 kPa and 1 GPa, while δ varies between 0° and 90°. Hence, the black diagram of binders constructed by G^* against δ can visually observe the rheological trends of bituminous binders. It should be noted that different types and contents of additives, modifiers, and alternative materials can lead to different black diagram shapes. Based on previous research, the black diagram can be divided into three types: neat binders, modified binders, and complex modified binders (Wang et al., 2022). All binders in Figure 8 have a similar high G^* (around 1 GPa) and low δ in the very low-temperature region, which means that the bio-based binders reach the glassy state at around −30°C. The G^* gradually turns lower and δ increases as the temperature increases. When the temperature reaches around 70°C, all the binders' δ is close to 90°. This characteristic is consistent with the trend of the black diagram of neat binders, suggesting that the viscoelastic characteristic of bio-extended binders is essentially the same as neat binders. The observed consistency in rheological behaviour implies that TOPs integrate into the bitumen without disrupting its structural integrity or flow properties. This is a critical finding, as it demonstrates that TOPs can be incorporated into bitumen formulations without compromising their performance in applications where rheological stability is essential, such as in pavement construction. Furthermore, this stability may be attributed to the chemical compatibility between TOPs and the

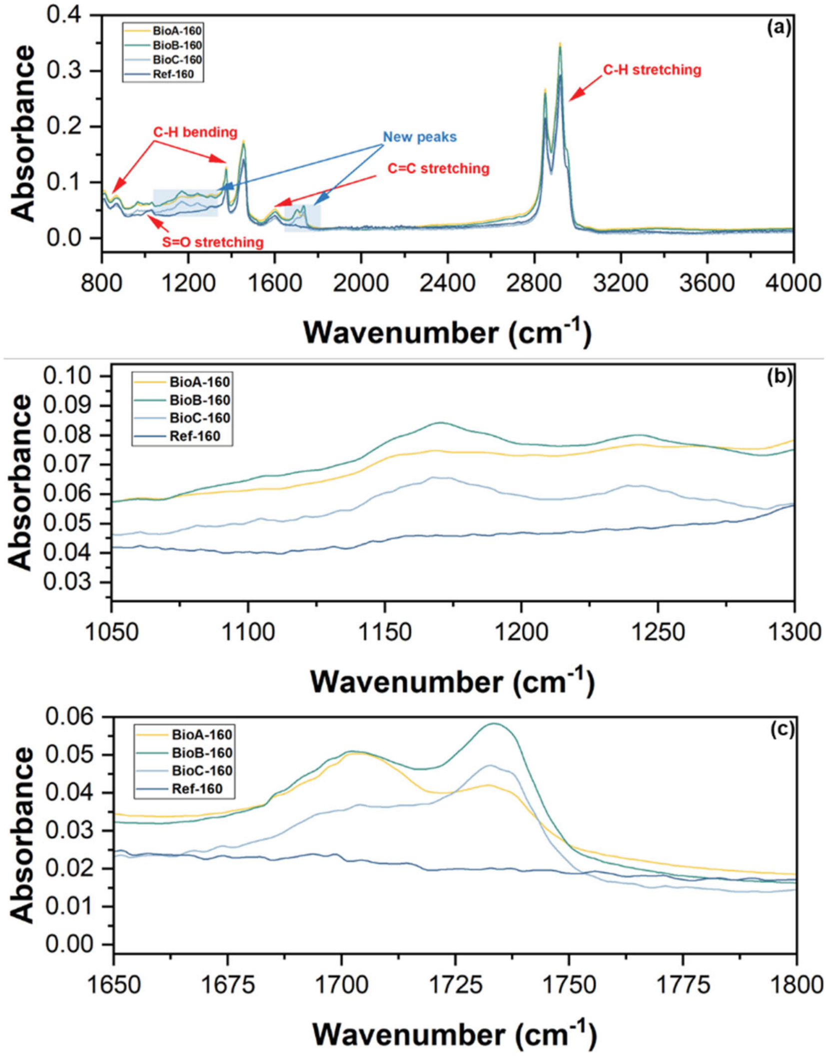

Figure 5. (a)FTIR spectra of 160/220 grade binders; (b)enlarged spectra of new peaks between 1050 and 1300 cm^{-1}; (c)enlarged spectra of new peaks between 1650 cm^{-1} and 1800cm^{-1}.

bitumen components, which allows for uniform dispersion and minimal interference with the bitumen's viscoelastic response. These findings highlight the potential of TOPs as a sustainable additive that enhances specific performance attributes without adversely affecting the bitumen's rheological signature.

The asphalt binders will fail when the complex modulus is lower than 1 kPa (Wang et al., 2022), so this part of the data is not in analysis. The temperature and modulus when δ is equal to 45° called crossover temperature and modulus at a given frequency, which reportedly can evaluate the anti-cracking and anti-aging properties of binders at intermediate temperatures (Ferrotti et al., 2018; Garcia Cucalon et al., 2019; Wang et al., 2022). The high PG temperature ($|G^*|/\sin\delta \geq 1000$ Pa), crossover temperature, and crossover modulus are shown in Table 3. It can be found that all 70/100-grade bio-extended binders are in the same PG temperature range, but they are all one PG grade lower than Ref-70. This suggests that for 70/100-grade binders, although the rheological behaviour of the prepared bio-extended binders is close to target binders, their high-temperature rutting resistance is

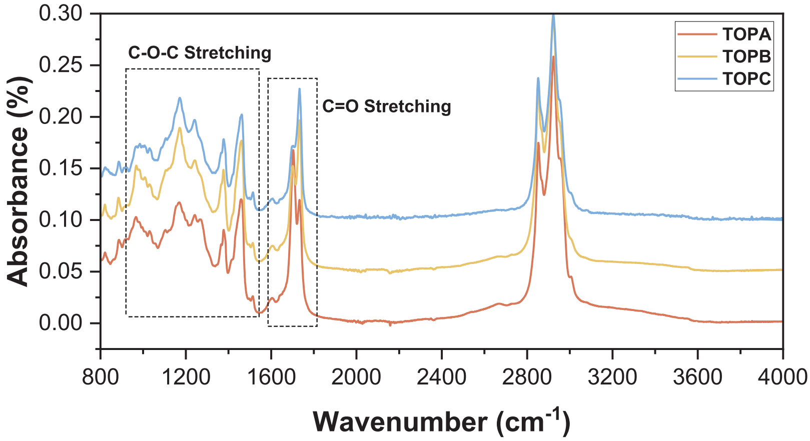

Figure 6. FTIR spectra of TOPs.

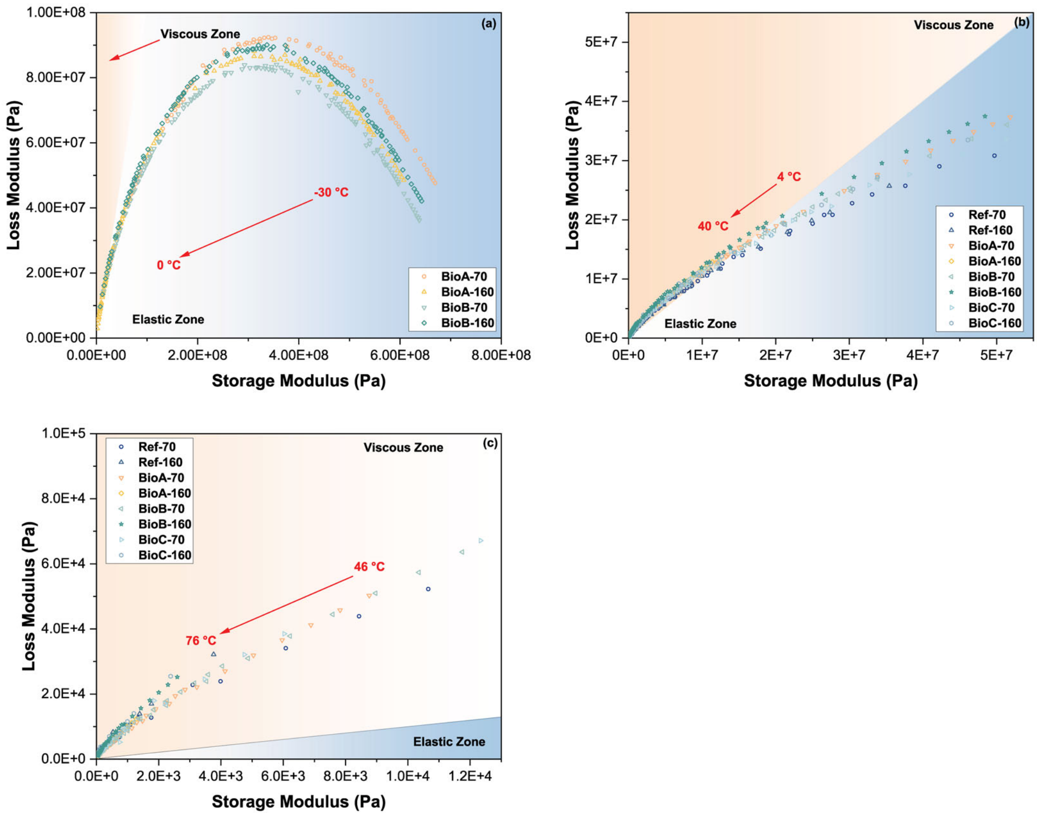

Figure 7. Cole-Cole plot of all binders: (a)low temperatures; (b)intermediate temperatures; (c)high temperatures.

reduced, directly impacting driving safety and pavement performance (Han et al., 2025). For 160/220-grade binders, all the bio-extended and target binders are the same PG temperatures, this means that TOP is more suitable for the preparation of high-grade binders. All the crossover temperatures of bio-extended binders in Table 3 are lower than target binders, especially for BioA binders. Similarly, the crossover modulus of bio-extended binders is higher than that of target binders. Hence, the prepared

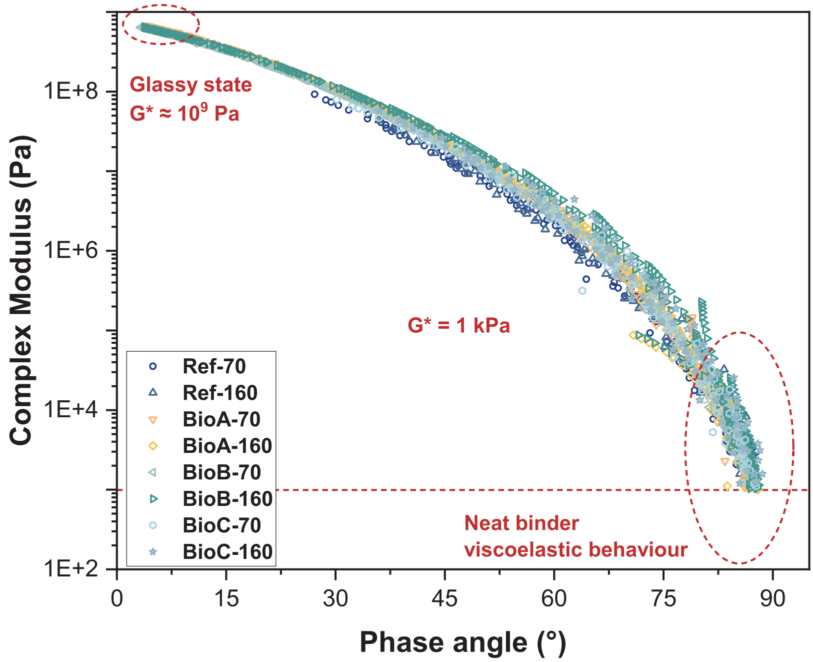

Figure 8. Black diagrams of bio-extended binders.

Table 3. PG temperature of bio-binders.

Binder type	PG Temperature /°C	Crossover Temperature /°C	Crossover modulus / kPa
Ref-70	64	11.4	14173.4
BioA-70	58	5.3	14912.2
BioB-70	58	9.5	14352.1
BioC-70	58	8.1	18776.5
Ref-160	52	5.0	14967.7
BioA-160	52	−2.3	15876.4
BioB-160	52	4.5	19876.4
BioC-160	52	3.9	22685.6

bio-extended binders in this research exhibit better low-temperature performance but unsatisfactory high-temperature performance.

3.3.3. Master curves

It is time-consuming to analyse the rheological properties of binders at each temperature, master curves provide a convenient method to evaluate the rheological behaviour by shifting the curves at different temperatures to a reference temperature based on the time-temperature superposition theory (Cao et al., 2022; Wu et al., 2022; Zhang et al., 2024a), thus generating the master curve. In this research, the Williams – Landel – Ferry (WLF) model (Bahia et al., 2001; Wang et al., 2022) is applied to construct the master curves of G^* and δ. The formulations of the master curves are as follows:

$$|G*(f,T)| = |G_e| + \frac{|G_\infty| - |G_e|}{[1 + (f_c/\alpha_T f)^k]^{m_e/k}} \tag{2}$$

$$\delta = 90I - (90I - \delta_m)\left\{1 + \left[\frac{log(f_d/\alpha_T f)}{R_d}\right]^2\right\}^{-m_d/2} \tag{3}$$

$$I = \begin{cases} 1, \text{ for asphalt mastic and mixture} \\ \left.\begin{cases} 0, & \text{if } f' > f_d \\ 1, & \text{if } f' \le f_d \end{cases}\right\} \text{ for asphalt binders} \end{cases} \tag{4}$$

where, $|G_e|$ (Pa) is zero for neat binders in this research; $|G_\infty|$ (Pa) is glassy modulus; f (Hz) is frequency; f_c (Hz) is the frequency when G' is equal to G''; δ_m the phase angle constant; f' (Hz) is the reduced frequency; f_d is the location parameter; α_T is the shift factor at T temperature; k, m_e, R_d, and m_d are the fitting coefficients.

The reference temperature in this research is selected as 22°C, and the G^* and δ at other temperatures are shifted horizontally to the results at 22°C based on shift factors (α_T). The shift factor can be calculated as follows:

$$log\alpha_T(T) = -\frac{c_1(T - T_0)}{c_2 + (T - T_0)} \tag{5}$$

where, T_0 (°C) is the reference temperature; c_1 and c_2 are the fitting coefficients.

The G^* master curves of bio-based binders are constructed in Figure 9 based on the above model. The time-temperature superposition theory (Cao et al., 2022; Wu et al., 2022; Zhang et al., 2024a) is applicable to the rheological properties of asphalt binders. So, the high-temperature performance can be evaluated at low frequencies, and low-temperature performance can be evaluated at high frequencies, based on shift factors. The master curves of 70/100-grade binders are shown in Figure 9(a), and they do not completely overlap, suggesting that bio-based binders have slightly different rheological behaviour from that of the target reference binder. Specifically, Ref-70 has the highest complex modulus G^* compared to BioA-70, BioB-70, and BioC-70, suggesting that Ref-70 is stiffer than bio-based binders. In other words, the TOP content is higher in bio-based binders although their needle penetration is already very close. This means that it is insufficient to use penetration and softening points as criteria for the preparation of bio-based binders with TOP. G^* begins to increase as temperature decreases/frequency increases, which is consistent with the G^* tendency of viscoelastic materials with temperature. All the master curves of G^* converge into a single line at low temperatures. The prepared bio-based binders show similar low-temperature rheological properties with the targeted neat binders. The master curves of 160/220 grade binders are presented in Figure 9(b). The G^* of the binders versus temperature/frequency is the same as the 70/100-grade binders in Figure 9(a). Their G^* values all increase with the decline in temperature or increase in frequency. The 160/220-grade bio-based binders show consistent rheological behaviour with the target reference binder, except BioA-160. The master curves of BioB-160 and BioC-160 almost overlap with Ref-160, revealing that the preparation of BioB-160 and BioC-160 is ideal and acceptable. Nevertheless, the rheological behaviour of BioA-160 is different from that of the target reference binder of Ref-160. The G^* of BioA-160 is always lower than that of Ref-160 even at low temperatures, indicating that BioA-160 is much softer compared to Ref-160. Although the penetration of BioA-160 is lower than that of Ref-160, BioA-160 shows more viscous rheological behaviour compared to the reference binder. Hence, the type of TOP has a significant effect on the rheological properties of bio-based binders.

The δ of binders is mainly affected by the viscous components of binders, and the master curves of δ are shown in Figure 10 with the same shift factors as for G^*. The δ of all binders is close to 90° at high temperatures/low frequencies, which means that the binders show pure viscous liquid behaviour in that region. δ turns lower and binders show more elastic responses as temperature reduces/frequency increases. At intermediate temperatures, the master curves of δ are dispersed in Figure 10. Specifically, bio-based binders have higher δ values compared to reference neat binders in Figure 10(a), which means that the bio-based binders show more viscous rheological responses at intermediate temperatures. As for the 160/220-grade binders, BioB-160 and BioC-160 have similar master curve trends as Ref-160 in Figure 10(b), but BioA-160 shows higher δ values. As the δ decreases gradually, the elastic response increases. When the temperature is low or frequency is high, the δ master curves of 70/100-grade binders converge into the same line, showing similar viscoelastic responses and rheological properties at low temperatures. The δ master curves of 160/220-grade binders exhibit two different

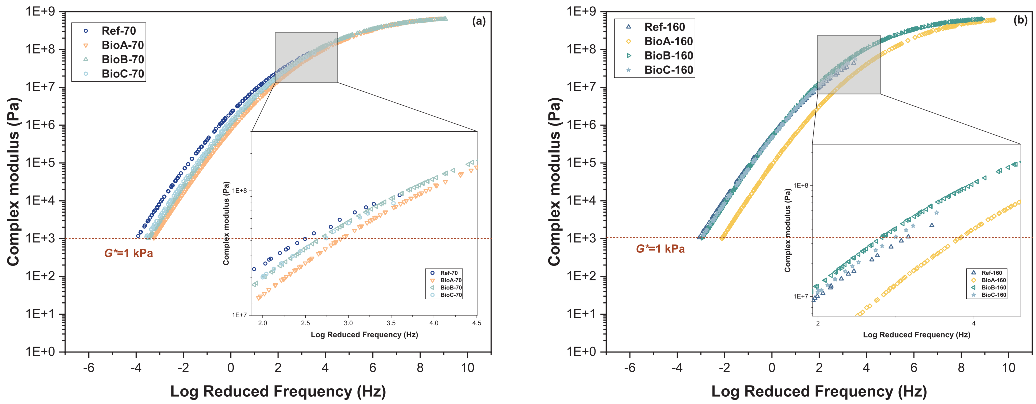

Figure 9. Master curves of G^* at reference temperature 22°C: (a)70/100-grade binders; (b) 160/220-grade binders

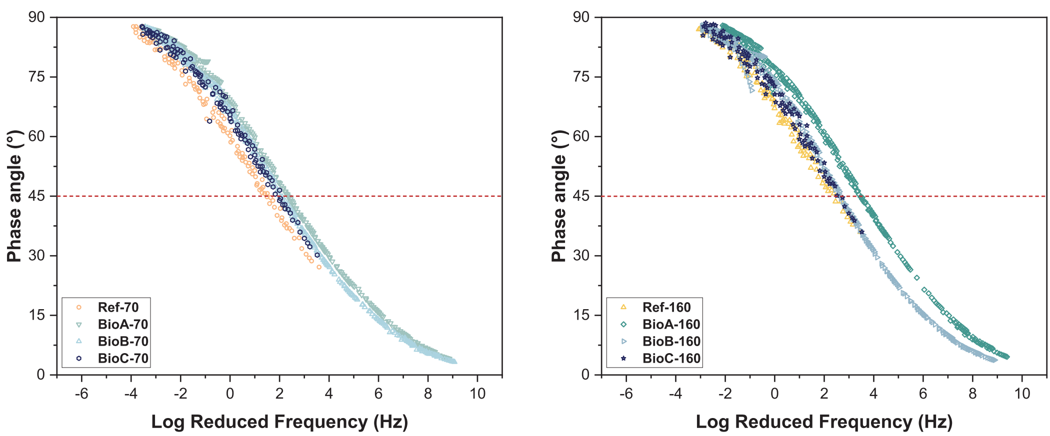

Figure 10. Master curves of δ at reference temperature 22°C: (a)70/100-grade binders; (b) 160/220-grade binders.

lines: BioB-160, BioC-160, and Ref-160 are closely gathered, while BioA-160 separates out of them. This indicates that BioA-160 shows more viscous responses and a different rheological behaviour at low temperatures.

3.4. Fatigue resistance

3.4.1. Yield strain and stress

The long-term aged asphalt binders are subjected to shear stress during the LAS test, making the stress–strain curves (Figure 11) first increase rapidly. The stress of binders then gradually reduces when the binders are damaged, hence stress–strain curves all show typical peaks. The peak stress and its corresponding strain are known as yield stress and strain. The asphalt binders can be considered as damage when the strain reaches the yield strain. Asphalt binders may not appear to yield without peak on stress–strain curves due to the addition of polymers according to Wu et al.'s report (Wu et al., 2024). Hence, TOP does not essentially have an effect as a polymer. The addition of TOP makes the yield stress of harder binders (70/100-grade binders) lower but increases the yield stress of softer binders (160/220-grade binders). This indicates that the amount of TOP has a different effect on the yield stress and strain.

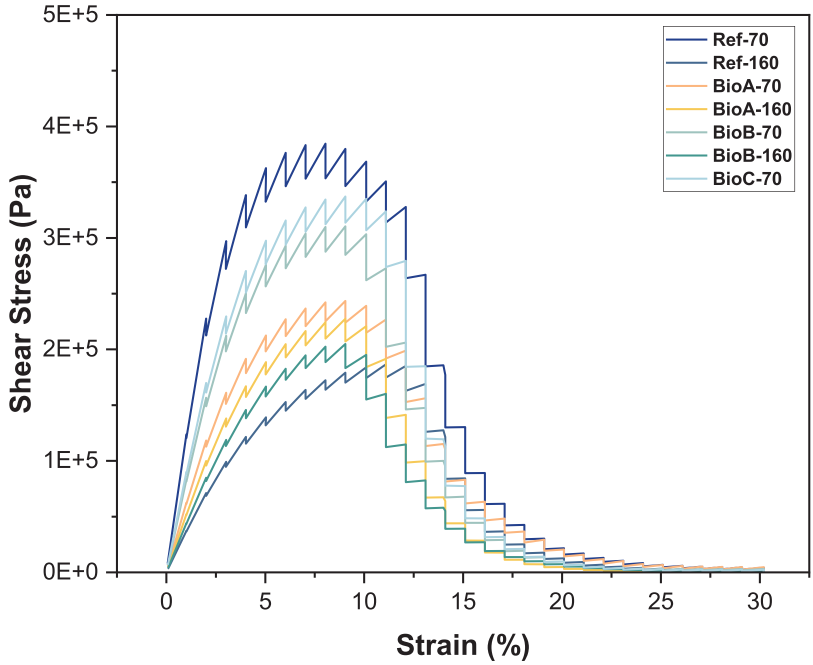

Figure 11. Stress-strain curves of bio-based binders at 25°C.

3.4.2. Integrity of bio-extended binders

The damage characteristic curve (DCC) is constructed by the integrity (*C*) and damage number (*D*) of the binders based on VECD theory, as shown in Figure 12. All the 70/100-grade bio-based binders show a lower *C* at the same *D*, compared to Ref-70. This indicates that the integrity of materials declines more rapidly compared to reference binders. Larger *D* at the same *C* reveals a better fatigue resistance (Wu et al., 2024), and 70/100-grade binders show higher *D* compared to 160/220-grade binders. This observation appears counterintuitive at first glance, as softer binders are typically expected to demonstrate better fatigue resistance due to their higher strain tolerance. This trend may be attributed to the specific interaction between the TOPs and the asphalt binders, which could influence the binders' ability to dissipate energy and resist crack propagation under cyclic loading. *D* increases after the addition of TOP at the same *C* for softer binders, suggesting that the same TOP does not show the same effect on the the fatigue resistance of different grades of binders due to the higher amount of TOP in softer binders. The above analysis of the DCC primarily can not fully capture the fatigue resistance and it is needed for further investigation to better understand the fatigue resistance of different TOP-based binders (Cao et al., 2018; Wang et al., 2017).

3.4.3. Fatigue life

Fatigue life (N_f) can be defined by the parameters *A* and *B*. The parameter *B* is affected by the storage modulus of asphalt binders, as shown in Figure 13. The addition of TOPs can reduce the storage modulus for harder binders, this is consistent with the rheological analysis. Additionally, the logarithmic curve of storage modulus has a strong linear behaviour, and the scope of fitting curves is used for calculating parameter *B* based on the VECD model.

The parameter *B* is calculated in Figure 14(a), and it can be found that all the bio-based binders show a lower parameter *B* compared to reference binders. Specifically, the parameter *B* of harder binders (70/100 grade) has minimal reduction, the maximum reduction is only 0.59. For softer binders (160/220 grade), the parameter *B* of BioA-160 and BioB-160 reduced by 6.59% and 11.05% compared to Ref-160.

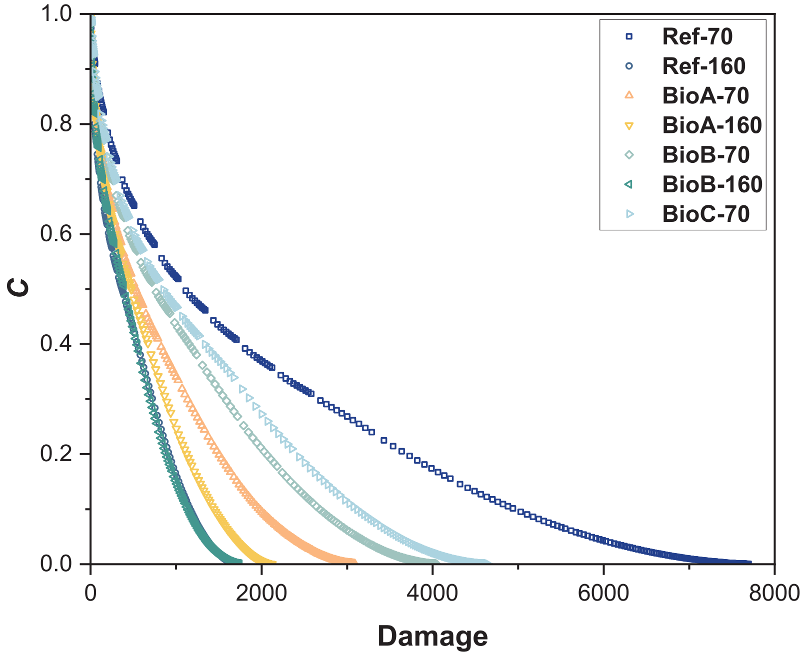

Figure 12. DCC of bio-based binders at 25°C.

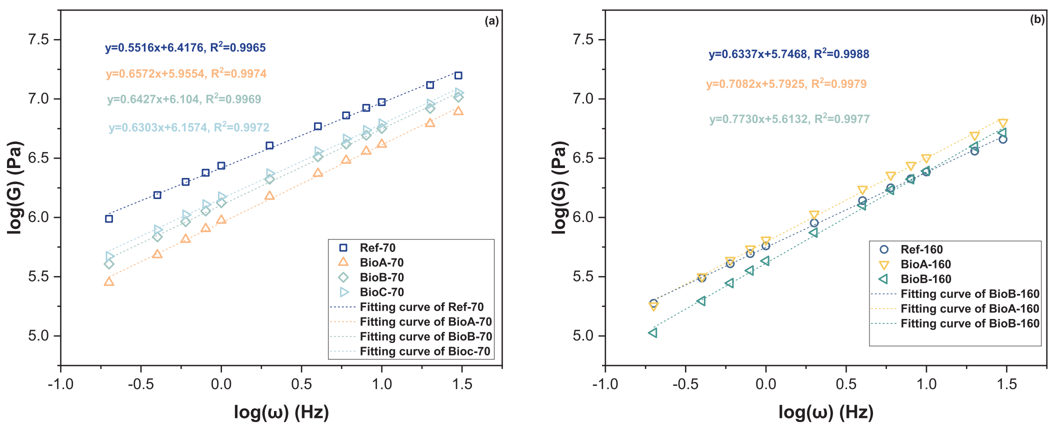

Figure 13. The storage modulus of bio-based binders at 25°C: (a)70/100 grade; (b)160/220 grade.

This suggests that TOP can slightly reduce the sensitivity of the material to changes in shear strain (Nivitha et al., 2024).

The parameter A is calculated according to the DCC, as shown in Figure 14(b). All the bio-based binders show a lower parameter A compared to reference binders. For harder binders, parameter A of bio-based binders are slightly reduced; however, for softer binders, parameter A is significantly reduced. This further indicates that the amount of TOP mainly affects the damage characteristics (reflected by parameter A) of the material rather than stress sensitivity (reflected by parameter B). The fatigue life (N_f) at three strain levels can be calculated based on parameters A and B, as shown in Figure 15. When the strain level is 2.5%, bio-based binders show similar N_f with reference binders. However, a high amount of TOP can significantly reduce the N_f for softer binders. It can be found that BioA-160 and BioB-160 can reduced by 2.7 and 2.8 times compared to Ref-160 at 2.5% strain level. When the

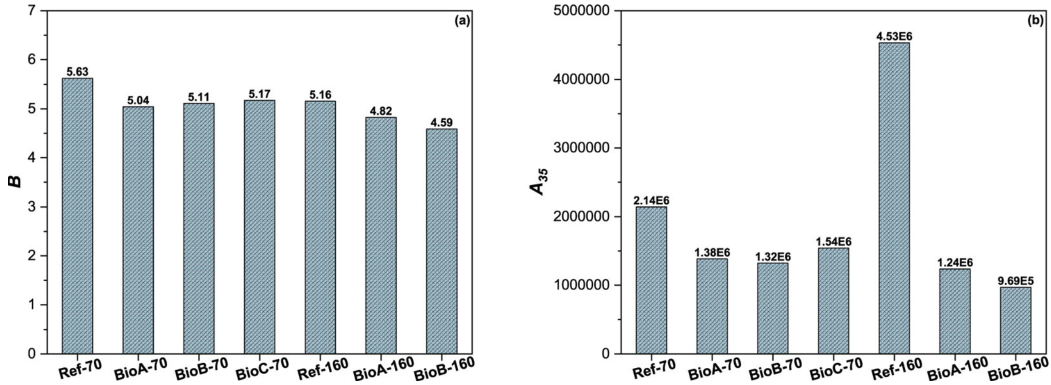

Figure 14. Parameters of bio-based binders at 25°C: (a)*B*; (b)A_{35}.

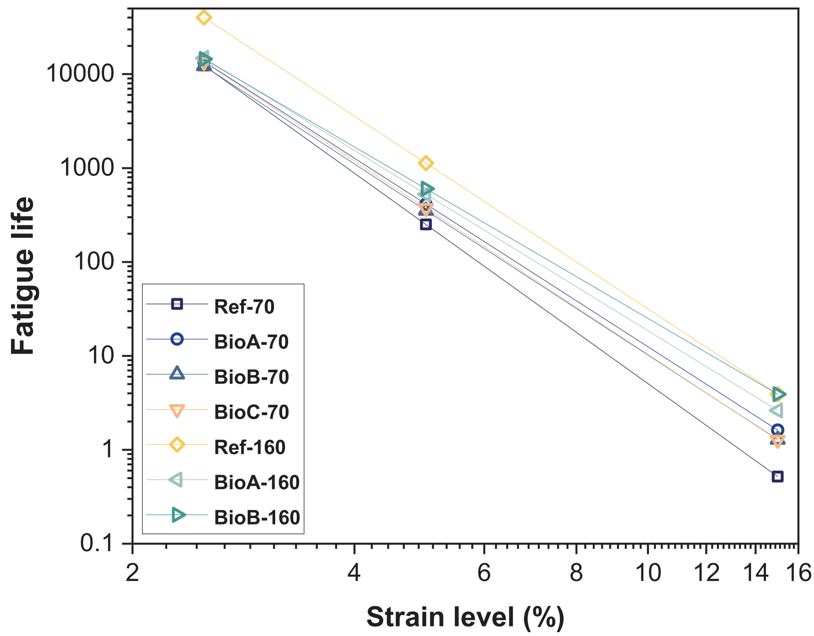

Figure 15. Fatigue life of bio-based binders at 25°C.

strain level increases to 5%, the N_f of 70/100-grade bio-based binders can increase by a maximum value of 64.86% and a minimum value of 40.96% compared to Ref-70. Nevertheless, the N_f of 160/220-grade bio-binders is still lower around 50% than that of reference binders. Additionally, the number of cycles to failure corresponding to a strain level of 15% is representative of the actual fatigue resistance of asphalt binders based on Chen et al.'s research (Chen et al., 2022). It is found that almost all the binders show a N_f below 10, indicating that TOP amount and strain level can significantly affect the fatigue resistance of the binders.

4. Conclusions

This research aims to use different TOPs (A, B, and C) to partially replace neat bitumen to prepare bio-extended binders of two penetration grades (70/100 and 160/220). The study investigates the feasibility of bio-based binders as an alternative to petroleum-based bitumen by comparing their physical, chemical, rheological and fatigue properties. The main conclusions can be drawn as follows:

- The penetration and softening point tests revealed that TOPs generally increase the penetration (softening effect) and decrease the softening point (reduced thermal stability) of the bitumen. The exponential and linear relationships between TOP content and penetration/softening point, respectively, allowed for the derivation of optimal TOP concentrations to match target bitumen grades.
- The overall FTIR spectra of bio-extended binders remained similar to those of neat bitumen, resulting in a better compaitiy between TOP and binder. The appearance of new absorption peaks (e.g. C = O stretching at 1650–1800cm^{-1} and C–O–C stretching at 1050–1300 cm^{-1}) indicated the presence of fatty acids and esters from TOPs. These findings suggest that TOPs integrate into the bitumen without significant chemical reactions, maintaining the fundamental hydrocarbon structure of the binder.
- Bio-extended binders exhibit improved low-temperature performance, with TOPA showing the most significant enhancement in viscosity. The Black diagrams confirmed that bio-extended binders retain viscoelastic properties similar to those of neat bitumen, suggesting good compatibility between TOPs and the bitumen binder. However, bio-extended binders are generally softer than their reference target binders, particularly at high temperatures, which may limit their rutting resistance.
- Performance grading (PG) analysis showed that bio-extended binders generally have lower high-temperature grades compared to reference binders, indicating reduced rutting resistance. However, the crossover temperature and modulus analysis revealed that bio-extended binders exhibit better low-temperature performance, with lower crossover temperatures and higher crossover moduli.
- TOP amount can influence the yield stress, strain and cumulative damage of binders differently based on their grade. A higher amount of TOP and a higher strain level can reduce the fatigue resistance for binders.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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