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LinkedIn Revista Ingeniería de Construcción

Vol. 39
Special Issue 2024
DOI: 10.7764/RIC.00127.21

Effect of biochar type in the performance of biochar-modified binder

Efecto del tipo de biocarbón en el desempeño de ligantes modificados con biocarbón

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Fecha de Recepción: 11/10/2024
Fecha de Aceptación: 25/11/2024
Fecha de Publicación: 30/12/2024
PAG: 1-9

Abstract

Biochar is obtained from the thermochemical conversion of biomass and stores carbon in a stable form. Adding biochar to asphalt could enable carbon offsetting. Biomass sources and pyrolysis technologies yield biochar with different chemical and physical characteristics. This study investigated the effect of two types of wood biochar on the binder characteristics. 5% and 10% biochar were added to a PG 58S-28 asphalt. Four biochar-modified binders and one control were characterized using rotational viscosity, penetration, frequency sweep tests, and Fourier-transformed infrared spectroscopy (FTIR). The results indicate that biochar increases the viscosity and reduces the penetration. The rheological analysis suggests that biochar could increase the rutting resistance but decrease the cracking resistance. Additionally, biochar alters the oxidative ageing of the binders. This study demonstrated that the biochar type impacts the binder performance, and source and content should be considered to understand the effect and determine the dosage of biochar to asphalt.

Keywords: Biochar; asphalt additives; biochar-modified binder performance.

Resumen

El biocarbón se obtiene a partir de la conversión termoquímica de biomasa y almacena carbono en una forma estable. La adición de biocarbón al asfalto podría facilitar la compensación de carbono. Las fuentes de biomasa y las tecnologías de pirólisis producen biocarbón con diferentes características químicas y físicas. Este estudio investigó el efecto de dos tipos de biocarbón de madera en las características del ligante. Se añadieron 5% y 10% de biocarbón a un asfalto PG 58S-28. Cuatro ligantes modificados con biocarbón y un ligante de control se caracterizaron utilizando viscosidad rotacional, penetración, ensayos de barrido de frecuencia y espectroscopía infrarroja por transformada de Fourier (FTIR). Los resultados indican que el biocarbón incrementa la viscosidad y reduce la penetración. El análisis reológico sugiere que el biocarbón podría aumentar la resistencia al ahuellamiento pero disminuir la resistencia a la fisuración. Además, el biocarbón altera el envejecimiento oxidativo de los ligantes. Este estudio demostró que el tipo de biocarbón impacta el desempeño del ligante, y tanto la fuente como el contenido deben considerarse para comprender el efecto y determinar la dosificación del biocarbón en el asfalto.

Keywords: Biocarbón; aditivos asfálticos; desempeño de ligantes modificados con biocarbón.

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ISSN: 0718-5073

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Special Issue 2024
DOI: 10.7764/RIC.00127.21

1. Introduction

Plants, trees, and other photosynthetic organisms remove carbon dioxide from the atmosphere, producing carbon-containing living biomass. In most natural environments, biomass will eventually die, decompose, and return to the atmosphere as carbon dioxide. This forms the basis of the natural carbon cycle. However, biomass can also be responsibly harvested in order to be converted into materials and products that are either used in manners that prevent or significantly postpone their decomposition (such as the use of lumber in buildings), or it can be thermochemically converted into recalcitrant forms that resist biodegradation for long periods. When this is done, the carbon is sequestered, removing this carbon from the carbon cycle. This approach can be used to offset fossil carbon that is presently being added by human activities to atmospheric carbon dioxide levels.

Biochar, a black solid product obtained from the thermochemical conversion of biomass, is one such product that will resist biodegradation for long periods of time. Furthermore, it may be useful as an input to some construction materials like asphalt concrete. Biochar can be the primary desired product from thermochemical conversion (such as in slow pyrolysis) or can be a secondary byproduct during the production of biofuels or biochemicals (such as in gasification or fast pyrolysis). Although biochar can be added to asphalt in order to sequester biogenic carbon, it is imperative that the biochar addition modify the asphalt properties in predictable ways to maintain or, ideally, improve asphalt quality.

A few studies have been completed that attempt to provide insight as to how biochar can influence the properties of asphalt binders or asphalt mixes. Some studies have shown promise that using biochar in the asphalt mix could help reduce the temperature susceptibility of the binder and increase the rutting, moisture, and cracking resistance of hot-mix asphalt (Chebil et al., 2000); (Kumar et al., 2018); (Walters et al., 2014); (Zhao et al., 2014).

This study was designed to respond to the following questions: Using two distinct biochar types, what impact does biochar incorporation have on the properties of the asphalt? Furthermore, how important are the properties of biochar? Will both biochars have similar impacts on the properties, or are there important differences between greatly different types of biochar?

The study's objectives included comparing the effect of biochar on viscosity, its impact on production temperatures (i.e. mixing and compaction), and the effect of biochar on consistency at intermediate temperatures. Another objective was to determine if biochar would make the asphalt more susceptible to permanent deformation (using the Superpave rutting parameter) or more susceptible to fatigue cracking (using the Superpave fatigue parameter). The study also aimed to compare the overall response to shear at different temperatures and frequencies and observe if there is a chemical interaction between the materials using Fourier Transformed Infrared Spectroscopy (FTIR).

2. Materials and methods

2.1 Biochar

Two biochars were produced at CanmetENERGY-Ottawa using distinct production technologies and feedstock materials to obtain two contrasting biochars. This helps inform whether the physicochemical properties of the biochar are important variables for optimizing the asphalt mix. One biochar was selected to exemplify a high-quality biochar from an uncontaminated stemwood feedstock processed in a slow pyrolysis process. Typically, the most important product from slow pyrolysis is the solid product, i.e. the biochar. The feedstock used was white birch wood chips from Ontario, which were converted to biochar in a temperature-controlled oven in closed 1L metal containers with tiny perforations made for pyrolysis vapours to escape. The temperature program plateaued at 450°C, which was held for 12 hours. In this article, this biochar is hereafter referred to as the slow pyrolysis biochar. The second biochar is an example of a byproduct biochar stream resulting from a low-quality forestry industry residue through a production method whose primary goal is normally to produce a liquid bio-oil. This biochar was produced using poplar bark from a mill in Eastern Quebec, which was processed by a fluidized bed fast pyrolysis pilot plant operated at 480°C. Details of the fast pyrolysis pilot plant can be found in (Mazerolle et al., 2019). This biochar is hereafter referred to as the fast pyrolysis biochar. For the analysis of each biochar, a representative sample was obtained by coning and quartering the parent lot of biochar. Representative biochar samples were ground to 0.075 mm (200 mesh) prior to analysis. The amount of ash in the samples (mineral matter after combustion) was completed according to ASTM D7582. Elemental composition was determined according to ASTM D5373 for carbon, hydrogen, and nitrogen, whereas ASTM D4239 was used for sulphur, and oxygen was computed by difference. The major differences in composition between the two biochars are the much greater content of ash (mineral matter), the lower overall carbon content, and the greater amount of oxygen and hydrogen relative to the amount of carbon in the fast pyrolysis biochar (Table 1).

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Table 1. Biochar Elemental Composition.

Parameter	Slow pyrolysis biochar (BS)	Fast pyrolysis biochar (BF)
ash (mass basis, dry)	1.47%	31.7%
C (mass basis, dry)	85.2%	55.1%
H (mass basis, dry)	3.0%	2.9%
N (mass basis, dry)	0.5%	0.5%
S (mass basis, dry)	<0.05%	<0.05%
O (mass basis, dry)	10.0%	9.9%
O/C ratio (molar basis, dry)	0.09	0.13
H/C ratio (molar basis, dry)	0.42	0.63

These two chars represent two extremes in terms of the quality of biochar that could be produced as an asphalt binder modifier. In the slow pyrolysis case (BS), residence times were long to maximize yields, the ash content in the feedstock was low (0.6%), and no mineral media was used in the process. These conditions produce a char with very low ash content. On the other hand, BF is a byproduct in fast pyrolysis, the starting feedstock was bark which contains considerably more ash (6.4%), and fast pyrolysis uses a mineral bed material, which when worn, contributes some additional inorganic material (ash) to the biochar.

2.2 Testing protocols

A single straight-run binder graded PG 58S-28 was used for the experiments. For the blends, the biochar was further sieved with a pestle over the No. 200 sieve, and only a fraction finer than 0.075 mm was used. Two biochar contents, 5% and 10%, by weight of the binder were selected (Table 2). For example, in a 10% content, 100 g was fresh run asphalt and 10 g biochar, yielding a total of 110 g modified binder. The binder was heated in the oven at the manufacturer-recommended mixing temperature of 148 °C; then, it was moved to a hot plate where the biochar was slowly added and stirred with a spatula for several minutes until the blended material was homogeneous.

Table 2. Samples Identification.

ID	Type of Biochar	Biochar Content
CM		0%
BS5	Slow pyrolysis	5%
BS10	Slow pyrolysis	10%
BF5	Fast Pyrolysis	5%
BF10	Fast Pyrolysis	10%

Testing of the modified binders was completed in three stages. The first stage evaluated the viscosity of the binders at elevated temperatures as per the ASTM D4402 standard using a rotational viscometer. The two temperatures selected were 135 °C and 148 °C, typical compaction and mixing temperatures, respectively, for the straight-run PG 58S-28 binder. Additionally, measurements of the penetration of the binder at an intermediate temperature of 25 °C were obtained, following the ASTM D5 standard to compare the consistency of the modified binders. The second stage comprised a frequency sweep test (15 frequencies from 0.20 to 100 rad/s) using a dynamic shear rheometer (DSR) to characterize the unaged (i.e. original binders), followed by repeating the same procedure on the same binders aged using Rolling Thin Film Oven (RTFO) and a Pressure Aging Vessel (PAV) (Table 3). In the third stage, Fourier Transform Infrared Spectroscopy (FTIR) was employed to monitor the changes in the composition of the binders through the different ageing conditions.

Table 3. DSR Parameters.

Condition	Temperature (°C)	Plate diameter (mm)
Original	45, 55, 65, 75	25
RTFO Aged	45, 55, 65, 75	25
RTFO + PAV Aged	5, 15, 25, 35	8
RTFO + PAV Aged	0, -6, -12, -18, -24, -30	4

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DOI: 10.7764/RIC.00127.21

3. Results

3.1 Biochar increased viscosity at elevated temperatures

As expected, the modified binder's viscosity changes with the addition of biochar (Figure 1). At production temperatures, biochar increases the viscosity of the binder, which is proportional to the percentage of biochar. 5% biochar has a lower increase compared to 10% biochar. However, fast pyrolysis biochar-modified binder (BMB) has a lower increase than slow pyrolysis BMB. A slight increase in mixing and compaction temperature would be necessary to achieve the recommended viscosities (0.17 Pa·s for mixing and 0.28 Pa·s for compaction). An average increase of 2.8% and 5.8% would be required, which means an increase from 150 to 154 °C for mixing and from 137 to 145 °C for compaction.

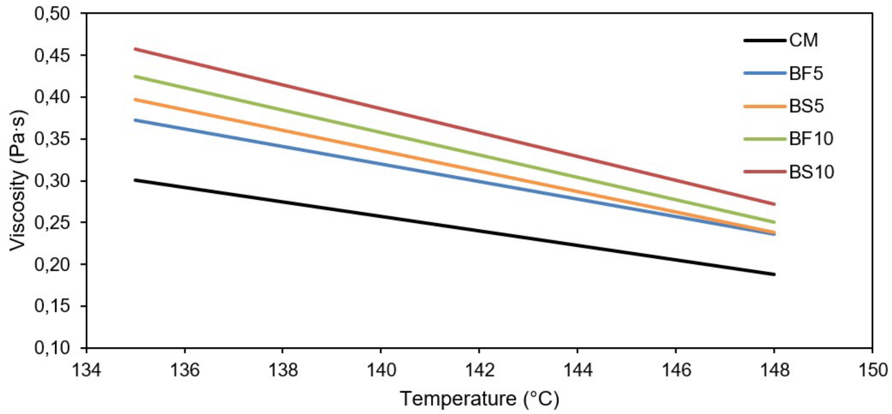

Figure 1. Viscosity vs Temperature Chart.

3.2 Biochar reduced penetration at intermediate temperatures

Consistency at intermediate temperatures is affected, and biochar hardens the asphalt based on the penetration test (Figure 2). Slow pyrolysis biochar hardens the asphalt more than fast pyrolysis biochar at 25 °C. Penetration decreases with the biochar content. A decrease of penetration depth of up to 50% was achieved with 10% slow biochar.

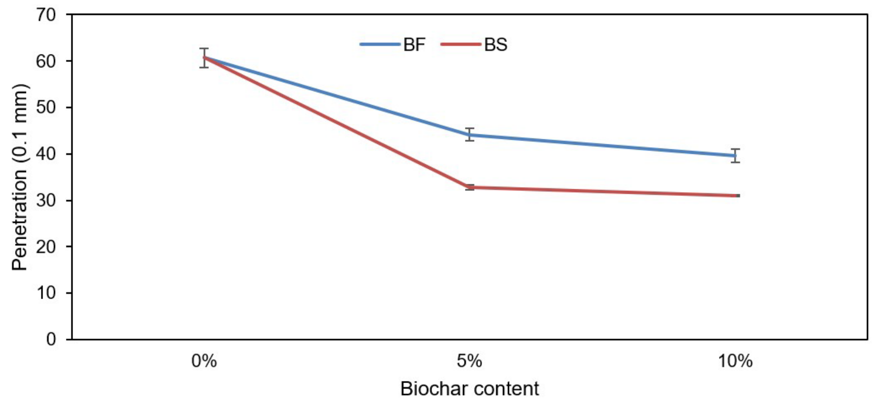

Figure 2. Penetration at 25°C.

3.3 Biochar increased the rutting resistance of binders

The rutting resistance of the BMB is higher than that of the control binder. The increase is proportional to the percentage of biochar (Figure 3). Slow pyrolysis BMB showed a higher value of the rutting parameter for the original (unaged) binder, but after RTFO ageing, the values of slow pyrolysis BMB and fast pyrolysis BMB are closer. Nonetheless, at this temperature, all binders exceeded the minimum Superpave suggested $G^*/\sin\delta$ threshold of 1.0 kPa and 2.2 kPa for original and RTFO-aged binders, respectively.

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DOI: 10.7764/RIC.00127.21

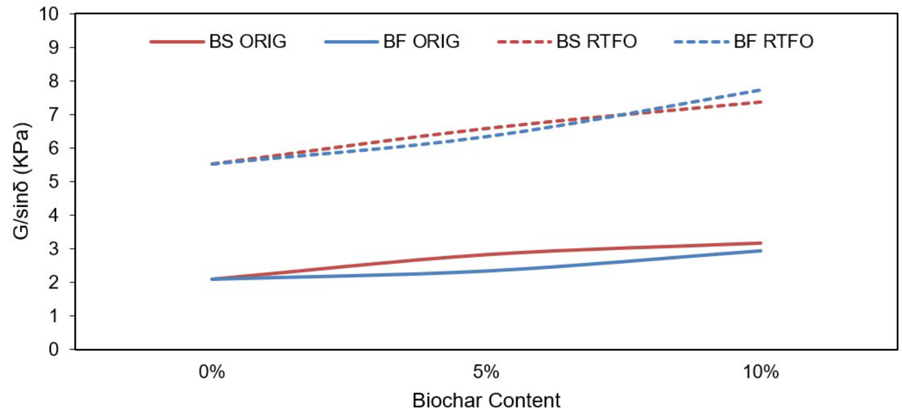

Figure 3. Superpave Rutting Parameter at 55°C and 10.9 rad/s.

This suggests that biochar would not adversely impact the high PG grade of the base binder. However, due to time and materials constraints, further testing was not conducted to determine the true grade of the BMB. This result is consistent with other studies reporting an increase in the rutting factor after incorporating mineral fillers such as limestone, silica fume, steel slag, Portland cement, hydrated lime, and fly ash (Chen et al., 2022).

3.4 Biochar reduced the fatigue cracking resistance of binders

The fatigue parameter at an intermediate temperature of 25 °C was <=5000 kPa for all the samples (Figure 4). The control binder shows a lower G* sinδ parameter, which translates into better fatigue resistance than BMB. It seems that fast pyrolysis BMB binders gain stiffness at a higher rate with the increase of biochar content compared to the slow pyrolysis BMB. It can be observed that the slow pyrolysis BMB gained more stiffness at a lower percentage and then stiffness plateaued at 10%, while the fast pyrolysis BMB gained more stiffness at the 10% biochar content but was not as stiff at the 5% content. It is essential to acknowledge that the fatigue parameter is widely used but has been criticized for not correlating well with field performance (Gibson et al., 2012).

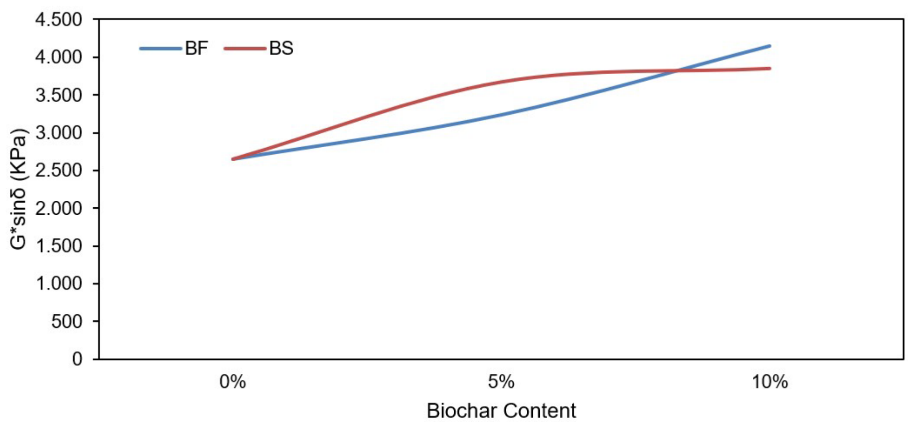

Figure 4. Superpave Fatigue Parameter of PAV residue at 25°C and 10.9 rad/s.

3.5 Similar performance trend of modified binders

Overall, at different temperatures and ageing states, the BMB with 5% or 10% content shows similar behaviour to the control binder. The results for the PAV binders at an angular frequency of 10.9 rad/s were selected for demonstration since they simulate typical highway traffic speeds. The CM binder generally shows the lowest stiffness, and BF5 is the binder consistently closer to the control (Figure 5). The phase angle did not see significant changes with the modification, and overall, the deviation of the modified binders compared to the control was under one degree for most of the conditions. However, the results are more dispersed as the temperature increases.

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The master curves were determined for the original and RTFO binders based on the results with the 25 mm plate and temperatures between 45 and 75 °C. At the original state, the control binder and the BF5 modified binder master curves seem to overlap; however, the control binder has the lowest stiffness values (Figure 6). The highest stiffness is observed for the 10% slow pyrolysis BMB. It can be observed that after ageing, the binders seem to have a closer complex shear modulus to each other. The RTFO aged binder results showed that 5% fast pyrolysis BMB has the lowest stiffness, while the control binder has the second lowest stiffness and overlaps with the 5% slow pyrolysis BMB. After ageing, the 10% slow pyrolysis BMB maintained the highest stiffness.

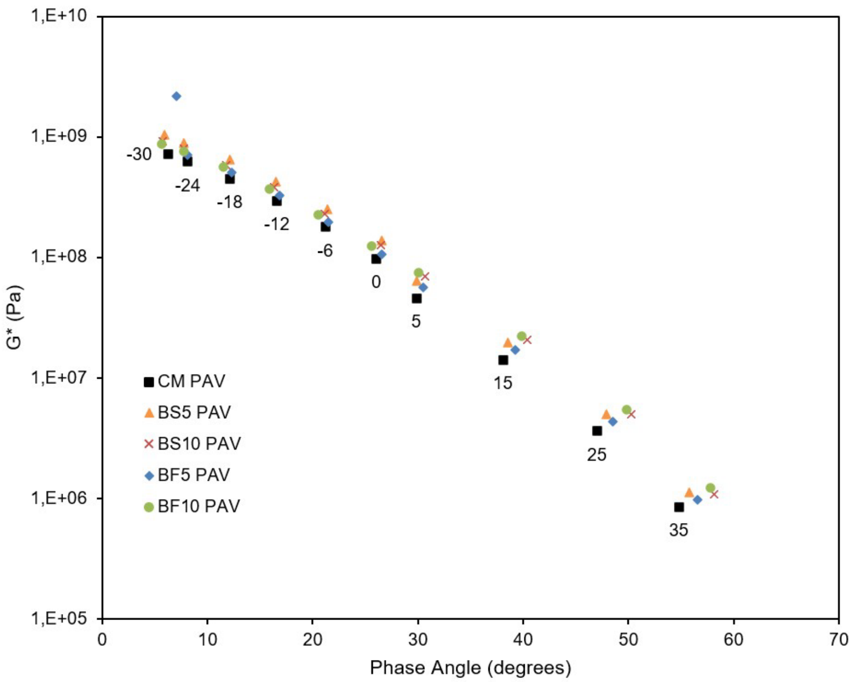

Figure 5. Black Space Diagram at 10.9 rad/s at varying temperatures in °C.

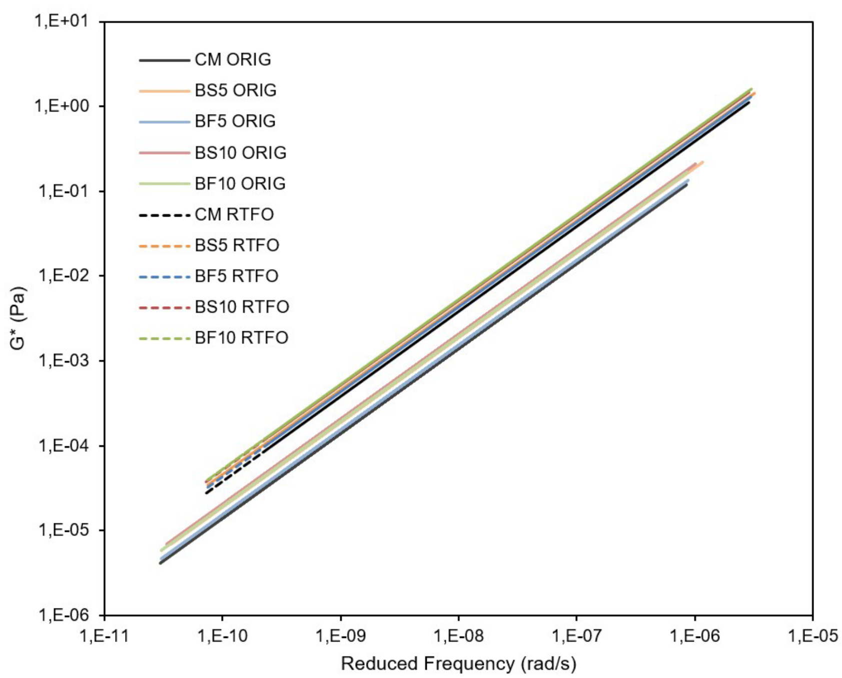

Figure 6. Master Curves at a reference temperature of 58 °C before and after RTFO.

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3.6 Impact on Ageing Rate

The ageing index can be used to compare the effect of short-term ageing on the binders. The ageing index is defined by the (Equation 1):

$$AI_{G^*} = \frac{G^*_{RTFO}}{G^*_{ORIG}} \quad (1)$$

The results at a frequency of 10.9 rad/s (Table 4) indicate that slow pyrolysis BMB has a lower ageing rate than the control. Fast pyrolysis biochar could increase the ageing rate and make the binder stiffer after RTFO. A reduction of up to 12% in the ageing index was obtained with slow pyrolysis BMB (BS10 at 45 °C), while a maximum increase of 4% was observed with fast pyrolysis BMB (BF5 at 65 °C).

Table 4. Ageing Index (AI_{G^*}).

Temp (°C)	CM	BS5	BS10	BF5	BF10
45	2.67	2.37	2.35	2.75	2.70
55	2.60	2.31	2.31	2.68	2.62
65	2.43	2.21	2.21	2.52	2.46
75	2.24	2.05	2.06	2.32	2.23

3.7 Impact on Ageing Susceptibility

Studies on the ageing of asphalt indicate that the carbonyl groups and sulfoxide indices increase after laboratory ageing where the respective indices are the ratio of the area of the C=O band (1670 to 1750 cm-1) or the S=O band (1015 to 1045 cm-1) compared to the area of adsorption bands between 1410 to 1510 cm-1. Nonetheless, (Hoftko et al., 2018) found that differences in the sulfoxide index are less significant, which is evidenced in (Figure 7b).

The original/unaged binders' carbonyl index has values closer to zero (Figure 7a). The negative results are artefacts of the shoulder of the aromatic C-C peak occurring near 1670 cm-1 relative to a nearly indiscernible C=O peak for original/unaged binders. At the RTFO state, a higher carbonyl index occurs in the slow pyrolysis BMB. This indicates that slow pyrolysis BMB could be more susceptible to oxidative hardening after short-term ageing.

The carbonyl index after long-term ageing for the BF5 is the lowest of all the BMB. Besides BF10, adding biochar did not seem to have a detrimental effect on the modified binder's long-term ageing susceptibility.

4. Discussion and conclusions

In this study, biochar-modified binders were tested to understand the effects of the biochar type on the properties of bitumen. The two biochars from different sources and different pyrolysis processes, slow pyrolysis and fast pyrolysis, were added in concentrations of 5% and 10% wt% of the bitumen. Overall, as expected, adding fine biochar to asphalt shows some similarities to the impact of adding a fine mineral filler. Nevertheless, the quality of the biochar does affect the performance of the modified binder, demonstrating that the chemical properties of the biochar are important to consider when optimizing BMB blends. The following conclusions can be drawn from the results:

- It was observed that BMB is more viscous than the control (unmodified) binder. This was expected and could be attributed to the solid nature of the biochar, meaning that it would reduce the ease of flow of the material at elevated temperatures. However, fast pyrolysis BMB was less viscous than slow pyrolysis BMB. It can be hypothesized that the presence of extra ash could help the temperature stability of the material.
- Overall, the presence of biochar decreased the penetration of the material. However, fast pyrolysis BMB was less stiff than slow pyrolysis BMB. This could be due to the lower carbon content of the fast pyrolysis biochar.
- Regarding the performance of the materials, it can be noted that both fast- and slow-pyrolysis BMB are less susceptible to rutting but more susceptible to fatigue than straight-run asphalt. Nonetheless, slow pyrolysis BMB is less susceptible to rutting, which can be

ISSN: 0718-5073

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DOI: 10.7764/RIC.00127.21

explained by the higher proportion of carbon. The trend for fatigue was dependent on the biochar content. At 5%, fast pyrolysis BMB was more resistant to fatigue than slow pyrolysis BMB, but at 10%, the effect is inverse.

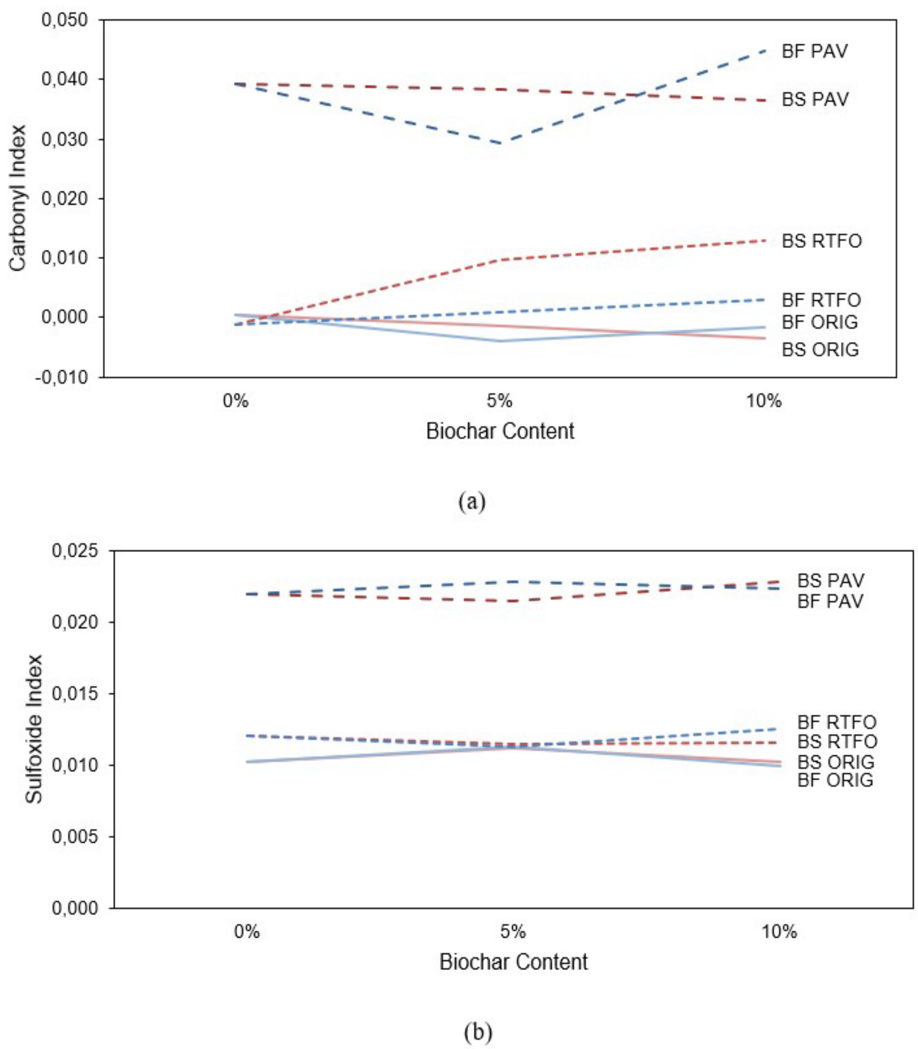

Figure 7. FTIR Carbonyl Index (a) Sulfoxide Index (b) Results.

- The results from FTIR indicate that a fast pyrolysis BMB is less susceptible to short-term aging than a slow pyrolysis BMB. This is related to the lower available carbon in the fast pyrolysis biochar. Nonetheless, the ageing rate was higher for fast pyrolysis BMB. However, a dosage of 5% fast pyrolysis biochar could reduce the long-term ageing susceptibility of the binder, which is an incentive for the use of this additive.
- In general, 5% fast pyrolysis BMB had the closest performance to the control binder. The use of biochar could improve the resistance to permanent deformation, and it was found that even though the complex shear modulus could increase, the phase angle does not seem to be compromised.

Future research should consider studying the low-temperature performance of BMB. Additionally, due to the particulate nature of the biochar, the applicability of traditional rheological measurement methods needs to be established. It is also critical to explore the storage stability of the modified binders and perform a comprehensive physical characterization of biochar, which could also substantially impact the properties of the modified binder.

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5. Acknowledgments

The authors would like to acknowledge CanmetENERGY, Natural Resources Canada / Government of Canada, for supplying the biochars used for this study. We also acknowledge McAsphalt Industries Ltd. for the in-kind DSR and FTIR testing.

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ISSN: 0718-5073