Laboratory Investigation of Bio-Char Modified Asphalt Mixtures

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Laboratory Investigation of Biochar-Modified Asphalt Mixture

Sheng Zhao, Baoshan Huang, Xiang Shu, and Philip Ye

The United States is promoting the establishment of a bio-based economy, generating energy and fuels from renewable organic matters rather than fossil fuels. Byproducts such as fractions of biochar not suitable for biofuel production are drawing extensive attention. ("Biochar" is defined by Oxford's online dictionary as "charcoal produced from plant matter and stored in the soil as a means of removing carbon dioxide from the atmosphere.") Recently, biochar produced with different production parameters was tried as an asphalt modifier owing to its carbon origin and proved to be positive in binder modification. This study evaluated the performance of hot-mix asphalt modified by one type of pyrolytic biochar with controlled production parameters. Typical carbon black powder pellets and microsized carbon fiber were selected as the reference additives. Multiple laboratory binder and performance tests were conducted to evaluate the rheological properties of the modified binder, rutting resistance, moisture susceptibility, and cracking resistance of asphalt mixtures. On the basis of test results, the biochar evaluated in this study proved to be an effective modifier in reducing the temperature susceptibility of the binder and thus increased the rutting, moisture, and cracking resistance of hot-mix asphalt. In addition, biochar proved to be a better asphalt modifier than did carbon black and carbon fiber.

Carbonaceous materials have long been used as asphalt additives because they are made of carbon and considered to be inherently compatible with asphalt binder that is hydrocarbon (*1*). This expectation has motivated numerous studies since the 1960s (*2*) to modify asphalt binder with carbon-based materials for improved performance. Among the many carbonaceous materials, carbon fiber and carbon black are commonly selected by researchers as asphalt additives owing to their easy availability.

The majority of carbon fiber research in the construction industry has been on its addition to concrete mixes (*3, 4*). The introduction of carbon fiber into asphalt stems from the hope that its high tensile strength may possibly increase the cracking resistance of asphalt pavement (*1*). It is also found that addition of carbon fibers produces electrically conductive asphalt mixtures (*5, 6*). Previous studies have shown that asphalt mixtures modified with mesolength carbon fibers show high resistance to permanent deformation, high tensile strength at low temperatures (*7*), and high fatigue resistance (*8*). However, addition of mesofibers might produce fiber clumps, thus leading to poor dispersion of fibers and nonuniform mixing (*7, 9*). This result would limit the application of mesofibers in asphalt industry. With the emergence of nanotechnology in the last decade or so, nanosized carbon fibers were used by highway engineers in pavement engineering (*10, 11*). The nanocarbon fibers behave more like nanoparticles rather than acting as fiber owing to the extremely small size, although a uniform mixing can be achieved.

Carbon black has been known to have a dramatic reinforcing effect on rubbers, showing potential benefits as an additive to asphalt binder owing to its carbon origin (*12*). Extensive research has shown that pelletized carbon black can reduce the temperature susceptibility of asphalt (*12–14*), improve rutting resistance at high temperatures (*14–16*), and reduce stripping potential (*16*) while maintaining similar fatigue and tensile characteristics of the mixtures (*15, 16*). The previous studies acknowledged the usefulness of carbon black as a reinforcing agent; however, the fatigue and cracking resistance of the asphalt mixtures modified by carbon black may remain a significant concern.

During recent years, the United States has been promoting the bio-based economy by requiring that energy and fuels be generated from renewable organic matters rather than fossil fuels (*17*). Some byproducts, which are not suitable for biofuel production and not used in any other industry, are drawing extensive attention. Fractions of biochar are one such byproduct from pyrolysis of biomass. ("Biochar" is defined by Oxford's online dictionary as "charcoal produced from plant matter and stored in the soil as a means of removing carbon dioxide from the atmosphere.") Most previous studies on biochar have been focused on its capability as a soil amendment that improves the physical and chemical properties of soil and may enhance nutrient cycling and plant growth (*18, 19*). Since aforementioned carbon-based modifiers have been successfully applied in asphalt modification, it is highly likely that biochar can be used as a biomodifier in asphalt mixture to improve the performance of asphalt mixtures. Earlier research work (*20*) evaluated asphalt binder modified with biochar prepared with different production parameters, demonstrating that biochar reduced the temperature susceptibility and long-term oxidation of asphalt binders, significantly increased the rutting resistance at high temperatures, and showed little or slightly better fatigue resistance. It was also found that the most effective biochar was the one with small particle size and that produced at a comparably lower treatment temperature and heating rate. However, performance of the asphalt mixtures with biochar-modified binder is still unknown and needs to be further investigated.

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TABLE 1 Asphalt Mixtures Evaluated in the Study

Asphalt Mixture	Additive	Additive Content (%)
PG 64-22 control mix	na	na
Modified mix	Biochar	5
		10
	Carbon black	5
		10
	Carbon fiber	5
		10

NOTE: na = not applicable.

OBJECTIVE AND SCOPE

The objective of the study is to evaluate the laboratory performance of hot-mix asphalt (HMA) produced with binders modified with selected biochar. The biochar used in this study was the most effective one selected from the previous research (*20*). Microsized carbon fibers and carbon black powder pellets were selected as reference additives based on previous discussion. Rheological properties of the modified binder, rutting resistance, moisture susceptibility, and cracking performance of the mixture were tested.

Six modified mixtures with various additive contents up to 10% by weight of the asphalt and one control mixture with virgin binder only were evaluated in this study (Table 1). The maximum additive content of 10% was determined on the basis of previous binder–mastic investigation (*20*) and studies on other carbon-based modifiers (*13, 16, 21*). The complex shear modulus (G^*) obtained from the dynamic shear rheometer (DSR) test was used to show the effect of additives on rheological properties of asphalt binder–mastic. The resilient modulus (M_R) and asphalt pavement analyzer (APA) rutting tests were employed to characterize rutting resistance of the modified mixture, while the AASHTO T 283 test after a freeze–thaw cycle was conducted to evaluate the moisture susceptibility pertaining to tensile strength ratio (TSR) and M_R ratio. The dissipated creep strain energy (DCSE_f) (*22*) from the Superpave® indirect tension (IDT) tests and the *J*-integral from the semicircular bending (SCB) notched fracture test (*23–27*) were used to evaluate the cracking performance.

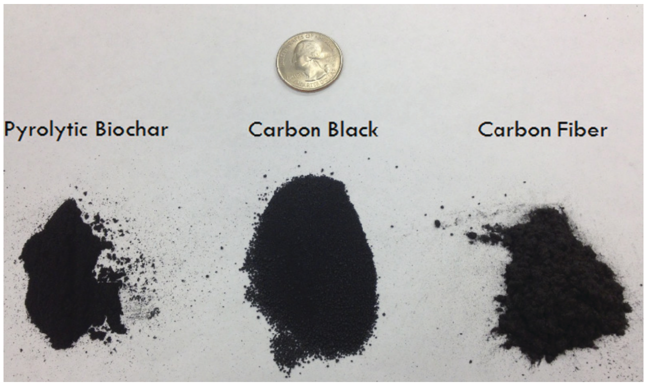

FIGURE 1 Direct view of additives.

LABORATORY EXPERIMENTS

Materials

The biochar used in this study was produced with a tube-furnace pyrolysis method (slow pyrolysis) based on a previous study (*20*). Production parameters are as follows:

- Starting temperature = 25°C,
- Pyrolysis temperature = 400°C,
- Heating rate = 15°C/min,
- Pyrolysis duration = 60 min, and
- Biochar production rate = 27.92%.

Only the particles smaller than 75 µm were used in this study to achieve homogeneous and uniform mixing. Switchgrass was used as the biomass source since considerable effort is presently being expended to make switchgrass a potential biofuel source (*28*). A commercially available carbon black with an average diameter of 80 nanometers (nm) and surface area of more than 20 $\mathrm{m^2/g}$ was used in this study. The carbon fiber was approximately 6 to 7 µm in diameter, and tensile strength and modulus of elasticity were 4,200 to 4,550 MPa and 230 GPa, respectively. Figures 1 and 2 show

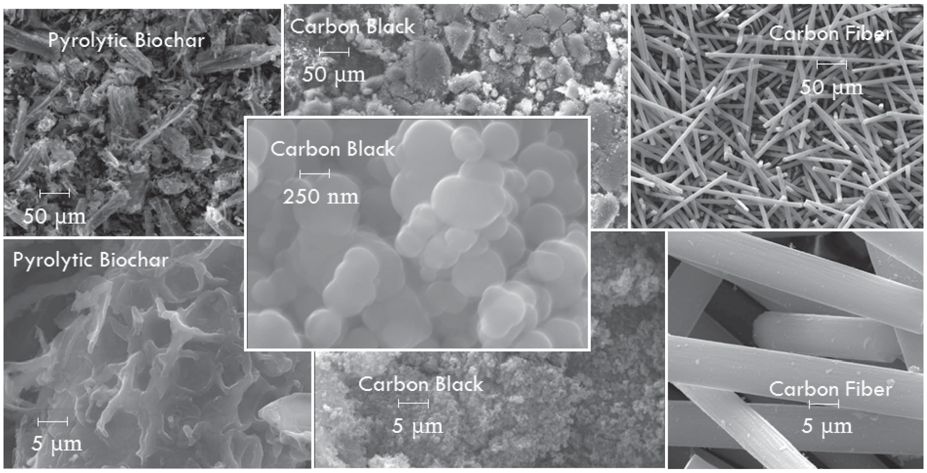

FIGURE 2 Images of additives magnified by SEM.

TABLE 2 Properties of Aggregates

Sieve Size (mm)	Gravel D-Rock (%)	No. 10 Screening (%)	Natural Sand (%)
16	100	100	100
12.5	92	100	100
9.5	71	100	100
4.75	23	93	98
2.36	15	59	76
0.60	9	22	37
0.30	6	15	17
0.15	4.0	11.4	7.0
0.075	2.5	9.5	3.2

NOTE: G_{sb} (bulk specific gravity) = 2.511 for gravel D-rock; 2.704 for No. 10 screening; and 2.498 for natural sand.

the ordinary picture and the scanning electron microscope (SEM) images of the different additives. Figure 1 shows that all three additives appeared to be similar black powder. However, SEM images show very different structures and surface textures. Carbon black was made of microcarbon pellets with a smooth texture, while carbon fiber was of a rod-like geometry and very fibrous nature. Biochar comprised irregularly shaped particles with a porous structure, which may behave like a combination of porous carbon fiber and microparticles. In addition, its porous and rough surface textures may help the interaction between biochar and asphalt binder, leading to improved performance of asphalt mixture.

One commonly used asphalt binder in Tennessee with PG 64-22 was used to make all the mixtures. The coarse aggregate was gravel D-rock with a nominal maximum aggregate size of 9.5 mm. The fine aggregates consisted of No. 10 (2 mm) screenings and natural sand. Their gradations and other properties are presented in Table 2.

Mix Design

The Marshall mix design procedure was employed to design the mixtures. The aggregates consisted of 50% gravel D-rock, 25% No. 10 screening, and 25% natural sand, and they met the gradation specifications of the Tennessee Department of Transportation (Figure 3). The optimum asphalt content was determined for control mix at 5.7% by weight of the total mix. To evaluate the effects of the modified binders, 5.7% virgin asphalt–modified asphalt was held constant for all the mixtures.

Sample Preparation

Asphalt binders and aggregates were heated for 2 h in an oven at 165°C before mixing. Meanwhile, the carbon-based additives were dried at 120°C for 2 h and then blended with heated asphalt binders at target concentrations by use of a mixing device designed for mastic research (*29*) in the laboratory. Samples for the DSR test were collected immediately after the blending, and then the modified binders were mixed in the laboratory with heated aggregates for 2 min. The newly produced mixtures were then kept in the oven for 2 h for short-term aging before compaction. Cylindrical samples 150 mm high by 100 mm in diameter were compacted with the Superpave gyratory compactor. The virgin binder for control mix was stirred for the same period of time before compaction to minimize the variations caused by mixture production. The air voids of samples for the IDT tests and SCB notch test were 4% ± 0.5%, and those for the APA and TSR tests were 7% ± 1% so as to simulate volumetric properties of pavement after several years' service and at early service stage, respectively.

Binder and Mixture Performance Testing

DSR Test

The effects of different types of biochar on binder modification were investigated in a previous study (*20*). In this study, the DSR test was conducted just to show the different properties of virgin and modified asphalt binders caused by additive modification. The complex shear modulus (G^*) of the virgin and modified binders was obtained from the DSR test by using samples of 8 mm diameter according to AASHTO T 315. Since a PG 64-22 binder was used, 64°C and −10°C were selected to characterize the modification at high and low service temperatures at a loading frequency of 10 rad/s.

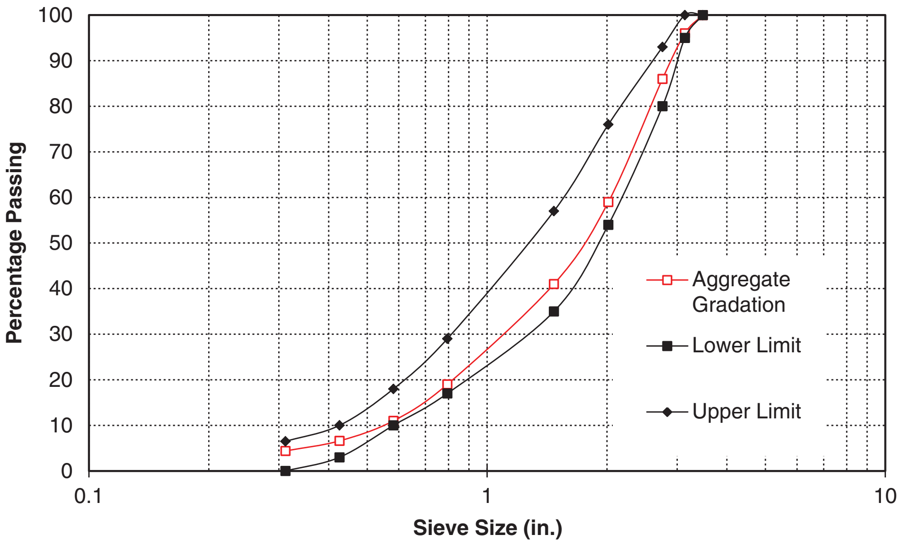

FIGURE 3 Aggregate gradation and Tennessee Department of Transportation gradation limits.

Resilient Modulus Test

The M_R (resilient modulus) test was part of the Superpave IDT tests developed by Roque and Buttlar (*30, 31*). The test was conducted at 25°C with procedures addressed elsewhere (*32, 33*). The M_R for each mixture was calculated with the following equation:

$$M_R = \frac{P \times \mathrm{GL}}{\Delta H \times t \times D \times C_{\mathrm{cmpl}}} \tag{1}$$

where

M_R = resilient modulus (psi),
P = maximum load (lbf),
GL = gauge length (in.),
ΔH = horizontal deformation (in.),
t = thickness of sample (in.),
D = diameter of sample (in.),
C_{cmpl} = nondimensional creep compliance factor, $0.6354(X/Y)^{-1} - 0.332$, and
(X/Y) = ratio of horizontal to vertical deformation.

Asphalt Pavement Analyzer Rutting Test

The APA rutting test was performed at 64°C in accordance with AASHTO T 340. Rut depths at 8,000 cycles were recorded for evaluating rutting resistance of the mixtures.

M_R Ratio and TSR Tests

Both the M_R ratio and TSR tests were used to determine the moisture susceptibility of asphalt mixtures by calculating the ratio of M_R and tensile strength of unconditioned to conditioned samples with 7% ± 0.5% air voids at 25°C (*34*). AASHTO T 283 was followed for the indirect tensile strength (ITS) and freeze–thaw conditioning procedures. A TSR value of 0.8 is recommended in AASHTO M 320 as a criterion for good resistance to moisture susceptibility.

Dissipated Creep Strain Energy Threshold

The DCSE_f was obtained from the Superpave IDT tests (*30, 31*) and used to evaluate the cracking resistance of asphalt mixtures (*35*). A higher DCSE_f value generally indicates a capability of accommodating more dissipated energy in the mixture, thus leading to a better fatigue resistance of asphalt mixtures. Figure 4 presents a schematic diagram for calculating DCSE_f, where ε_f is the failure strain, M_R is the resilient modulus, EE is elastic energy, and S_t is indirect tensile strength.

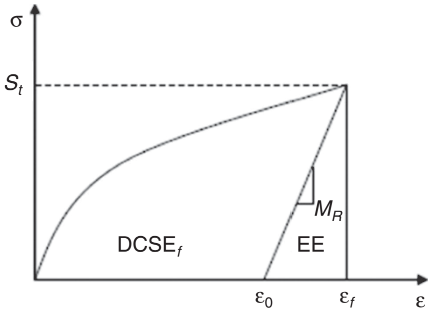

FIGURE 4 Calculation of DCSE_f (*34*).

Semicircular Bending Notched Fracture Test

The SCB fracture test was conducted on specimens with different notch depths at 25°C at a constant rate of 0.5 mm/min. The critical value of the *J*-integral was obtained and used as an indicator of fracture resistance of asphalt mixtures (*23, 27*). Three notch depths were used in this study: 7.6 mm (0.3 in.), 15.2 mm (0.6 in.), and 25.4 mm (1 in.). The strain energy to failure was calculated for each notch as the numerical number of the area under the load versus vertical deflection curve up to the peak load. Then the following equation was used to determine the critical *J*-integral:

$$J_c = -\left(\frac{1}{b}\right)\frac{dU}{da} \tag{2}$$

where

J_c = *J*-integral (kJ/m^2),
b = thickness of specimen (m),
a = notch depth (m), and
U = strain energy to failure (kJ).

All the samples evaluated in this study were made in triplicate. The summarized experimental matrix is presented in Table 3.

TABLE 3 Experimental Matrix

Property Evaluated	Test	Temperature (°C)	Parameter	Samples per Mixture
Rheology	DSR	64	G^a	3
	DSR	−10	G^a	3
Rutting resistance	Superpave IDT	25	M_R	3
	APA	64	Rut depth	3
Moisture susceptibility	Superpave IDT	25	M_R ratio	3[a]
	TSR	25	TSR	3[a]
Cracking performance	Superpave IDT	25	DCSE_f	3
	SCB	25	J_c	3

[a] For M_R ratio and TSR tests, triplicates were made for both conditioned and unconditioned samples.

RESULTS AND DISCUSSION

Rheological Properties of Modified Binders

Figures 5 and 6 show the G^* results at high and low temperatures, respectively. The observation that G^* increased with the addition of the additives is as expected. It seems that biochar showed the highest stiffening effect at high service temperatures, and that may lead to a higher rutting resistance of the modified binder, but the differences in stiffening caused by the three carbonaceous additives were not significant. For low temperatures, it can be seen in Figure 6 that biochar showed very little effect on G^* at −10°C, consistent with the findings from the previous study (*20*). However, the addition of both carbon black and carbon fiber significantly increased the G^* of the binder at low temperatures, thus potentially leading to a poor resistance of asphalt mixture to low temperature cracking. This observation indicates that biochar was a more effective asphalt modifier, compared with carbon black and carbon fiber, for reducing the temperature susceptibility of asphalt binder.

Rutting Resistance

The M_R can be used as an indirect indicator of the rutting resistance of asphalt mixtures. The higher the M_R value, the higher the rutting resistance of an asphalt mixture. Figure 7 illustrates the M_R results for the mixtures. It is observed that all three additives increased the M_R if added up to 10%. This result can be attributed to the stiffening effect of the solid powder-like additives. The fact that carbon fiber showed an insignificant effect can be related to its interaction with the binder. The rod-like microfibers may not be uniformly blended with the binder-aggregate matrix during the mixture production, thus resulting in a slightly poor structure of the matrix, although the carbon fibers were cut into microsizes. It was found that the 10% biochar-modified mixture showed the highest M_R, an indication that biochar may have a better interaction with the binder during mixing, and that may lead to a better resistance to rutting distress.

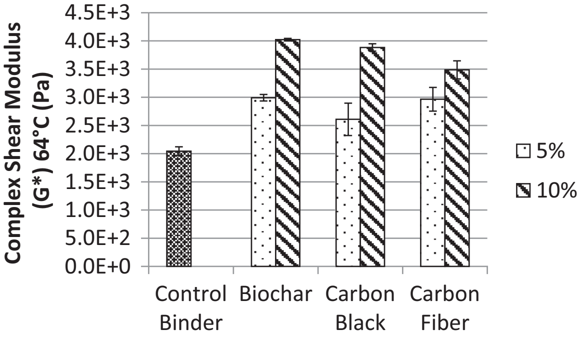

FIGURE 5 G^* results at high service temperature (64°C).

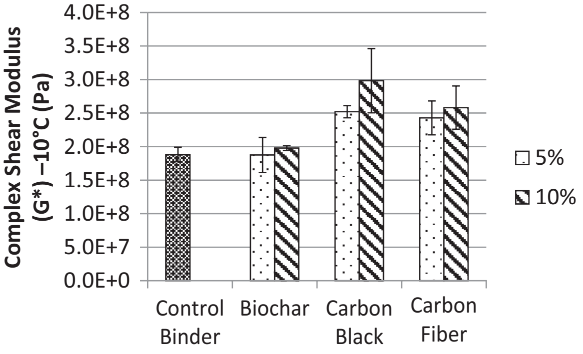

FIGURE 6 G^* results at low service temperature (−10°C).

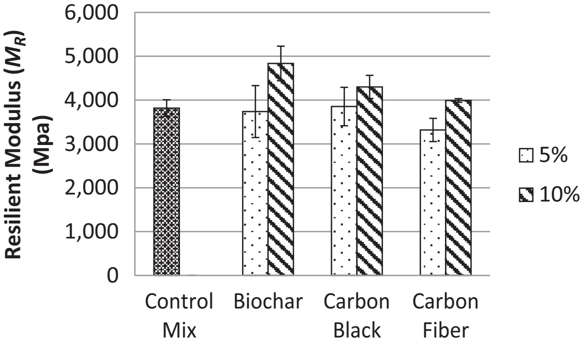

FIGURE 7 M_R results at 25°C.

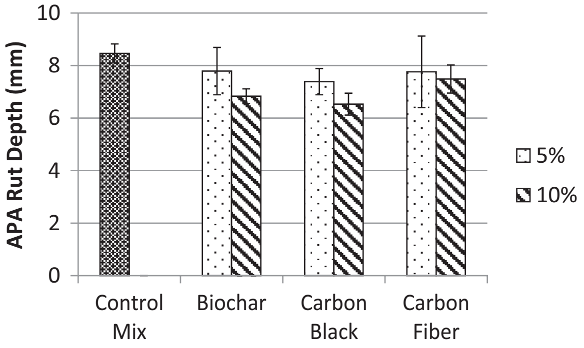

FIGURE 8 APA rut depth results.

Figure 8 presents the rut depths after 8,000 cycles from the APA rutting test. It is evident that the addition of the additives improved the rutting resistance, especially biochar and carbon black at higher contents. This observation was consistent with results from the G^* at high service temperature and M_R. It is expected that carbon black and carbon fiber could reduce the rut depths, especially carbon black (*16*). However, it was found that biochar showed a similar capability of improving rutting resistance and thus was very promising as an additive in reducing rut distress.

Moisture Susceptibility

Figures 9 through 12 present the M_R ratio, dry ITS, wet ITS, and TSR results, respectively. Generally, moisture resistance was slightly increased in most mixtures. The 10% addition of carbon black appeared to result in an obvious decrease in modulus but demonstrated that it could satisfy the AASHTO specification of TSR being over 0.8. The modifier content was found to affect moisture resistance with the following tendency: the more modifiers that were added, the more susceptible the mixture became to moisture. The mixtures modified with 10% carbon black or carbon fiber were found to be on the verge of meeting the 80% TSR criterion, while the biochar-modified mixtures could easily meet this requirement, an indication that biochar

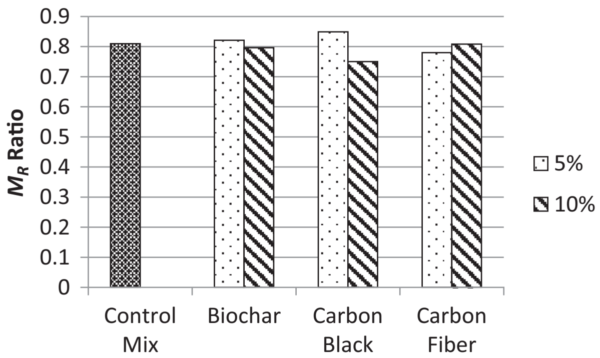

FIGURE 9 M_R ratio results.

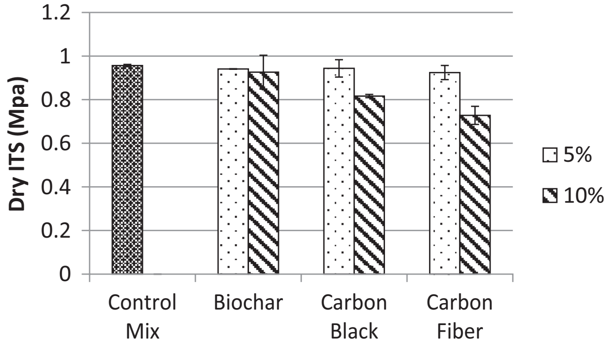

FIGURE 10 ITS for unconditioned (dry) samples.

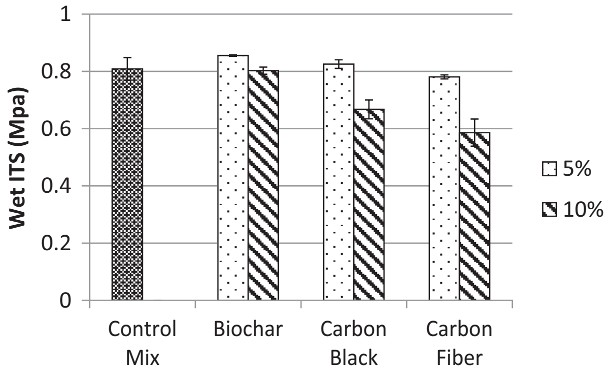

FIGURE 11 ITS for conditioned (wet) samples.

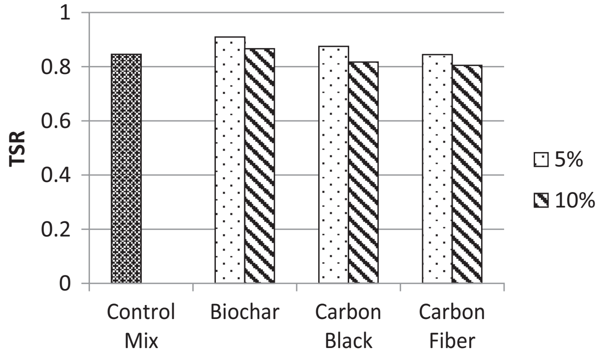

FIGURE 12 TSR results.

may have a higher capability of improving moisture resistance than the other two carbon-based additives.

Cracking Performance

The DCSE$_f$ value has been successfully used to evaluate the cracking resistance of asphalt mixtures (*35, 36*). The results are presented in Figure 13. It can be seen that high variations were observed in this study, consistent with previous research using the same method (*36*). So the numerical average of the triplicate was selected to conduct the data analysis. Unlike the effects of the additives on other properties, the effects of the three additives were found to be noticeably different from each other in regard to cracking resistance. The increase in the biochar or carbon black content reduced the DSCE$_f$ value, therefore compromising the cracking resistance of modified mixtures. However, the 5% biochar was found to increase the DCSE$_f$ value, and even the mixture with 10% biochar showed a similar DCSE$_f$ result compared with the control mix. Meanwhile, carbon black did not show beneficial effect on cracking resistance, even added in a small amount (5%). Carbon fiber behaved differently with the increase in its content. Its modification effect was insignificant when added at 5%. However, its effect became significant at a dosage of 10%. This phenomenon can be attributed to the fibrous nature of the carbon fiber. It may function like reinforcement in concrete, bridging the cracks and arresting crack propagation, thus leading to a higher cracking resistance. The carbon fiber may perform even better at higher contents. However, use of this fibrous material may be limited by such concerns as workability, dispersion, and moisture susceptibility.

The J-integral concept has been successfully used to characterize the fracture resistance of the asphalt mixtures (*23*). Figures 14 and 15 show the change of the dissipated strain energy with the notch depth. The J-integral was obtained for each mix accordingly and was reported in Figure 16. Samples with a 7.6-mm notch in 10% carbon black group were broken during the production, so the J_c value for that mixture was obtained on the basis of two points. As can be seen in Figure 16, the effects of additives in regard to the J-integral were similar to that obtained from DCSE$_f$ results. Biochar increased the J_c value so as to increase the cracking resistance of the mixture. Carbon black showed adverse effects on the mixture in regard to J_c value results, while carbon fiber proved to be positive when added in a higher amount. This consistency validates the conclusions drawn from Superpave IDT results and further indicates the effectiveness of biochar as an asphalt modifier.

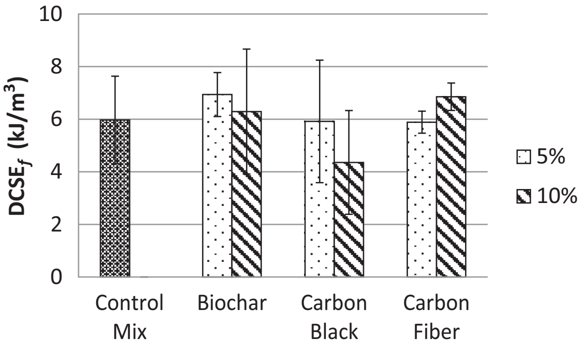

FIGURE 13 DCSE$_f$ results.

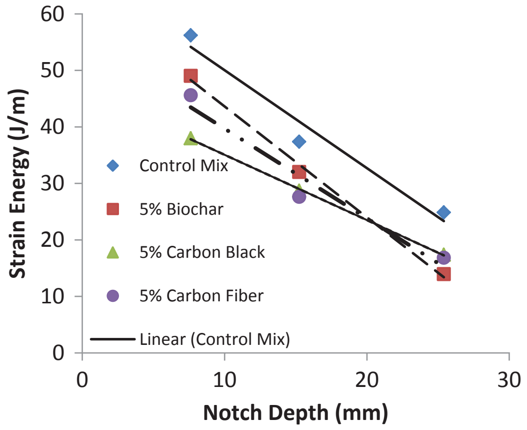

FIGURE 14 Change of strain energy with notch depth (5% additive).

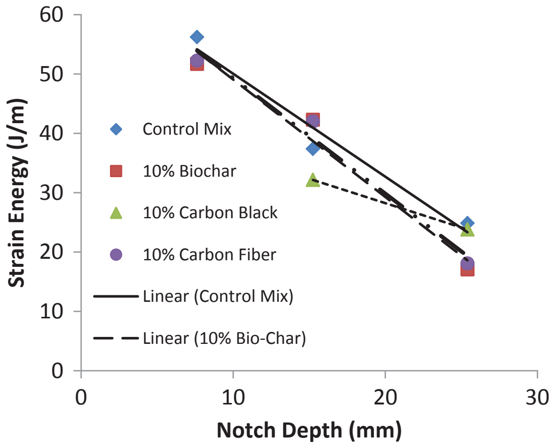

FIGURE 15 Change of strain energy with notch depth (10% additive).

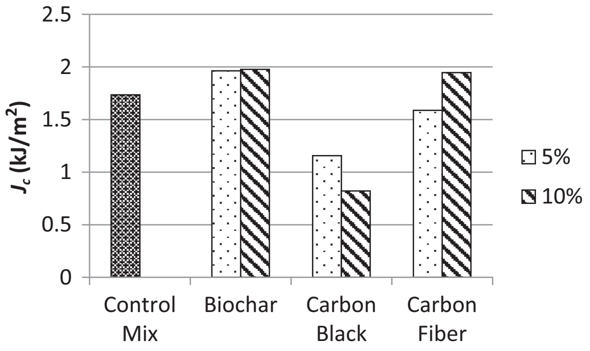

FIGURE 16 *J*-integral results.

CONCLUSIONS AND RECOMMENDATIONS

A newly developed pyrolytic biochar with controlled production parameters was introduced in this study. Multiple laboratory binder and performance tests were conducted to evaluate the rheological properties of the modified binder, rutting resistance, moisture susceptibility, and cracking resistance of asphalt mixtures. Two major carbonaceous asphalt additives, carbon black and carbon fiber, were evaluated as reference materials. According to the test results, conclusions can be summarized as follows:

1. Compared with carbon black and carbon fiber, biochar was more capable of reducing the temperature susceptibility of the asphalt binder.
2. M_R and APA rutting test results indicated that biochar increased the rutting resistance of the asphalt mixture.
3. Biochar performed in a way similar to carbon black in improving rutting resistance and did better than carbon fiber.
4. Biochar could reduce the moisture susceptibility of HMA with addition of small amounts. The modification effect would be compromised with the increase of the additive content.
5. Biochar performed better than carbon black and carbon fiber in improving the moisture resistance of HMA.
6. Both DCSE$_f$ results from Superpave IDT tests and J-integral results from the SCB notched fracture test suggested that biochar might increase the cracking resistance of HMA.
7. Carbon black might adversely affect the cracking resistance of HMA, while carbon fiber might have a positive effect if added to a certain amount. With the increase of additive content, the modification from biochar might be compromised, whereas that of carbon fiber was positively improved.
8. Results presented in this paper were only the preliminary findings of a more complete study. Further studies would be needed before biochar can be widely used in pavement engineering.

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