At the frontline for mitigating the undesired effects of recycled asphalt: An alternative bio oil-based modification approach

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Abstract

Soybean oil-derived modifiers were used for the improvement of properties of asphalt materials prepared for a pavement demonstration project. The rheological properties of base, biomodified and extracted binders were measured/compared using rheometers. The binder modification resulted in a decrease of 1.2°C and 2.3°C in, respectively, the high-and low-temperature grades of base binder, and when the effect of RAP binder was considered, the continuous performance grade (PG) became almost identical with that of base/control binder. Due to the biomodification and the presence of RAP, the binder's elastic recovery (R) increased by 8.0% and its non-recoverable creep compliance (J_{nr}) decreased by 0.13 kPa^{-1}. The tests conducted to evaluate the mechanical performance of the mixtures proved the efficacy of the bio-modifiers used in reversing the undesired effects of reclaimed asphalt pavement (RAP) and improving the performance of asphalt pavements at different temperatures. For instance, the Hamburg wheel tracking (HWT) test results revealed that the presence of bio-modifiers resulted in the increase of stripping inflection point (SIP) by 3619 passes. The disk-shaped compact tension (DCT) test proved the effectiveness of the bio-modifiers used, as these modifiers increased the fracture energy by 113 J/m^2. The master curves constructed for the asphalt binders and mixtures indicated an increased stiffness/elasticity at intermediate and high temperatures.

Keywords: bio modifiers; soybean oil-derived modifiers; polymer; rejuvenators; recycling agent; reactive restorative modifier; rheology; mechanical performance; high-and low-temperature properties

1. Introduction

The use of reclaimed asphalt pavement (RAP), due to sustainability gains [1], has been very attractive since the late 1980s [2]. RAP is often obtained from milling asphalt pavements at the end of their service lives. Due to oxidation/change in microstructure that occurs throughout the service life of asphalt pavement, the RAP binder has a poor relaxation capability [3]. Therefore, when RAP is replaced with virgin aggregate, the resulting asphalt mixture becomes stiffer/more brittle. Many different rejuvenators/recycling agents have been produced to restore the properties of this aged binder (or RAP binder) to address the brittleness issue [4], [5], [6], [7], [8]. In addition, to further enhance the resistance of asphalt mixture to distresses, it would be possible to modify the asphalt binder with polymers [9].

The rejuvenators are generally formulated to return an oxidized/aged binder's ratio of asphaltenes /maltenes to its unaged stage [10], [11]. The asphalt binder's rheology depends primarily on the asphaltenes' quantity, as they are more viscous than both resins and oils [12]. Due to oxidation, resins transform into asphaltenes while saturates/oils and aromatics transform into resins, and these transformations increase the quantity of asphaltenes leading to increased hardening in asphalt materials [13]. The rejuvenators made of saturates/oils can restore an aged asphalt binder to its original viscoelastic state, but the quality of such restoration depends on the formulation of such additives and/or modifiers and their application method [14]. Rejuvenators can restore the rheological properties of aged asphalt-based materials, e.g., RAP [15]. The less expensive and environmentally friendly bio-based rejuvenators [16], [17], [18], have lately drawn the attention of researchers. These bio-based rejuvenators are typically obtained/derived from natural oils such as distilled/crude tall oil [19], cotton seed oil [20], cashew nut oil [16], and soybean oil [21], [22], [23], [24], [25], [26].

According to a recent review study conducted by Behnood [27], the rejuvenators obtained from the oil extracted from soybean and corn oil are reported to more effectively reduce the hardness of asphalt materials, compared with the petroleum-based analogues. The Midwestern United States produces soybean at significantly large quantities, making this produce/feedstock an ideal candidate for the biomodification of asphalt materials throughout the whole country. The soybean oil-derived rejuvenators that have been successfully used for rejuvenating the RAP binder include epoxidized (or “epoxy”) soybean oil (ESO) [26], [28], [29] and epoxidized methyl soyate (EMS) [30]. Sub-epoxidized soybean oil (SESO), a reactive rejuvenator recently formulated at Iowa State University, is made from sub-epoxidized triglyceride molecules from soybean oil [31]. The SESO is theorized to react with asphaltene molecules, adding relatively long hydrocarbon chains that increase the stability of the asphaltenes within the maltene matrix. Evidence of this was shown by Podolsky et al. [32] through the use of small angle x-ray scattering testing. These results indicated that the asphaltenes are becoming de-aggregated resulting in an improvement of the miscibility within the maltenes. Due to reactive and restorative properties of SESO and lack of consensus on a name for referring to rejuvenators, in this study, this material is referred to as reactive restorative modifier (RRM).

Polymer modification of asphalt binders dates back to mid-1980s with the primary intention to mitigate rutting distress in asphalt mixtures [9]. In addition to increasing the resistance to rutting [33], polymers can also increase the asphalt mixture’s resistance to fatigue and low temperature cracking [34]. The polymers used for the modification of asphalt binders are either plastomers or elastomers [35]. Polymer modifiers conventionally used for improving the performance of asphalt binders are, for example [36] , styrene-butadiene (SB) block copolymers, ethylene vinyl acetate copolymer, glycidyl methacrylate, styrene-isoprene-styrene, polyethylene, ammonia polyphosphate, acrylonitrile-butadiene-styrene, but most of these polymers are petroleum-based. According to recent studies at Iowa State University, soybean oil-derived biopolymers with engineered formulations, can be economical and environmentally friendly alternatives to petrochemically-derived analogs [37]. Poly(acrylated epoxidized high oleic soybean oil), PAEHOSO, a biopolymer formulated at Iowa State University, is a soybean derived polymer created by reversible addition-fragmentation chain transfer (or RAFT) polymerization to obtain a thermoplastic elastomer with similar effects as polybutadiene in asphalt [37]. The RAFT polymerization technique renders the PAEHOSO thermoplastic. To date, except for the biopolymers developed at Iowa State University, there has been no report on the use/synthesis of any thermoplastic soybean oil-derived polymer with application in the modification of asphalt binders.

The objective of this research was to investigate the influence of asphalt binder modification with the SESO and PAEHOSO on changing the rheological properties and mechanical performance of asphalt binders and mixtures prepared for a pavement demonstration project. To this end, the asphalt materials were modified with the aforementioned bio modifiers in an asphalt terminal and an asphalt plant. Test samples were then procured to perform comprehensive laboratory-based investigations. The tests were performed to assess the change in the rheological properties and mechanical performance of the asphalt materials to ensure that the modification techniques and the bio modifiers used were able to improve the engineering properties of a newly-constructed asphalt pavement.

2. Materials and methods

2.1. Materials

2.1.1. Base asphalt binder

The base performance grade (PG) asphalt binder used in this study was already modified using a triblock co-polymer, styrene butadiene styrene (SBS) 3501 with sulfur pellets. This binder was PG 58-28H (or PG 65.4-31.0) with an elastic recovery of 34.8% - measured at 58°C - that was produced at an asphalt terminal. To account for the level of elasticity, due to polymer modification, a designation is added to the PG of the asphalt binder that defines the appropriate traffic level. For example, in the State of Iowa, USA, H is assigned to asphalt binders with an elastic recovery within a range of 30% - 55%.

2.1.2. Soybean oil-derived modifiers

SESO is known to slightly reduce the elastic recovery while providing favorable changes to the low temperature behavior [32]. The PAEHOSO is hypothesized to react with the double bonds on the butadiene group of the tri-block SBS. In this way, a greater polymer network is formed with the PAEHOSO adding to the elastic portion of the polymer and using the polystyrene ends as anchors. This polymer network formation is illustrated in Fig. 1 that was drawn using ChemDraw® software. Ideally, the PAEHOSO would react with two different SBS polymers forming a branched hepta-block copolymer with the four ends being polystyrene acting as anchors. To increase the reaction of the polymers, sulfur was used, and the way such reaction is done/increased is similar to vulcanization. Previous laboratory experiments have shown that PAEHOSO alone does not provide a large benefit to high temperature properties. A study done by Chen et al. [38] showed that creating a di-block copolymer with PAESO (a previous version of PAEHOSO) and styrene can form an elastomer with similar performance to styrene-butadiene

(SB) polymer in asphalt. Therefore, the current study uses PAEHOSO in an asphalt modified with SBS to achieve superior performance than what would be achieved with only SBS or only PAEHOSO.

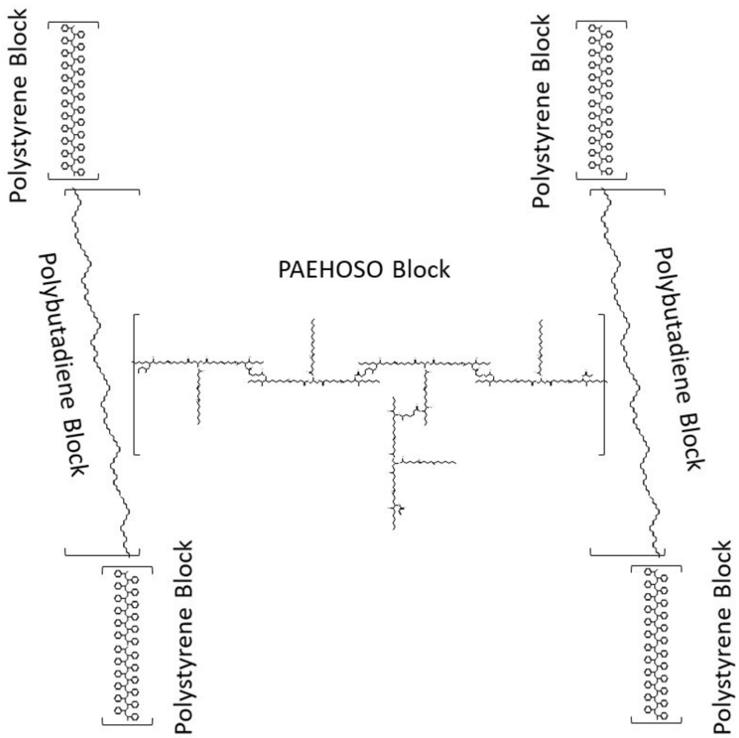

Fig. 1. Schematic of the reaction mechanism of PAEHOSO with the butadiene group of SBS.

The optimum dosage rates of SESO and PAEHOSO were determined by several trial blends in the laboratory. More information on the identification of optimum dosage rates for the modification of base asphalt binders with bio-modifiers is presented in another study [25].

2.1.3. Aggregates

Aggregates obtained from different sources (Table 1) were blended to achieve a gradation (Fig. 2) that meets the most current Superior Performing Asphalt Pavement (Superpave) specifications. In most the USA it is recommended to use about 20% RAP in a given mix design [39]. Therefore, the incorporation of RAP into the asphalt mixture was limited to 20%.

Table 1. Aggregate gradations.

Aggregate Type	Percentage in Blend	Aggregate Gradation for Each Aggregate Type										
		1"	3/4"	1/2"	3/8"	#4	#8	#16	#30	#50	#100	#200
3/4" Washed Chip	10	100	98	62	33	6	3	2.5	2.3	2	1.7	1.5
3/8" Washed Chip	30	100	100	100	92	23	3.5	2.5	1.8	1.7	1.6	1.5
Manufactured Sand	30	100	100	100	100	96	66	36	18	7	3.5	2.5
Natural Sand	10	100	100	100	100	96	83	64	41	15	2	0.7
RAP	20	100	100	96	93	71	54	41	29	16	11	9.1

Note: RAP = Reclaimed Asphalt Pavement.

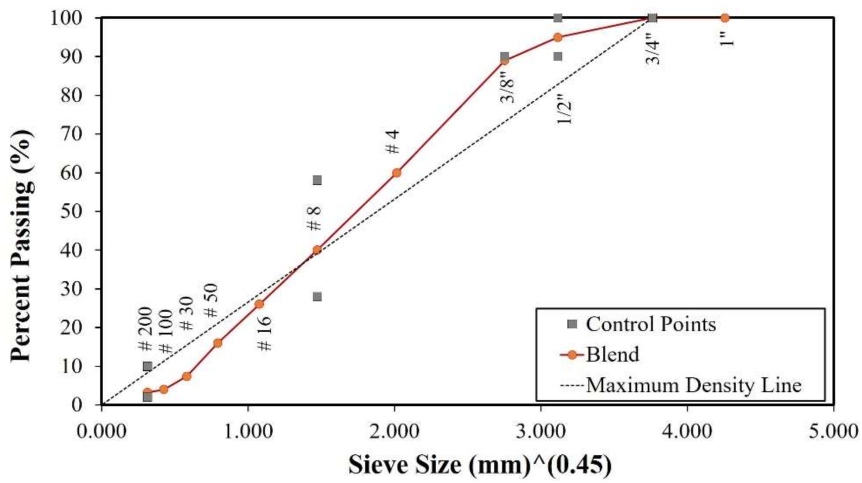

Fig. 2. Gradation of aggregate blend.

2.1.4. Asphalt mixtures

Asphalt mixtures used for fabricating specimens were procured from an asphalt plant. In this research, these mixtures were used for performing quality control and investigating the influence of BioMAG binder (a binder modified with PAEHOSO and SESO) on changing the mechanical performance of asphalt mixtures and predicting the field performance. Hereinafter, the mixtures modified with BioMAG asphalt binder are referred to as BioMAG mixtures. Two types of mixtures were procured from the asphalt plant: BioMAG and control. It is worth noting that, due to the presence of RAP in the combined aggregate blend in the asphalt plant, the

BioMAG binder was added at a content of 3.97%. This content was selected to achieve a total target asphalt binder content of 4.87% including the RAP binder. In other words, the total asphalt content of 4.87% was the summation of 3.97% BioMAG binder and 0.9% RAP binder.

2.2. Preparation of asphalt binder specimens

The BioMAG binder prepared in the asphalt terminal was blended for 10 hours before being transferred to a binder tank at the asphalt plant where it continued stirring for 4 days because of weather delays. To consider the influence of this 4-day stirring period, therefore, BioMAG binder samples were procured from the tanker truck just before mixing with the aggregates. The BioMAG binder sampled after four days is representative of the binder that was used for producing the hot mix asphalt (HMA). Therefore, extensive rheological testing was performed on this binder. Also, the asphalt binder extracted from the HMA, referred to as Ext BioMAG binder, underwent rheological testing so that the influence of presence of RAP binder could be investigated as well. This binder was extracted using toluene according to the Association of State Highway and Transportation Officials (AASHTO) T 319-15 except for filtration method. The BioMAG mixture was washed with toluene and allowed to soak before the filtrate was removed by centrifuge. The filtrate was then centrifuged again to remove any fine particles. Finally, the filtrate was recovered by distillation using a nitrogen blanket.

The BioMAG asphalt binder samples procured from the tanker truck were aged using rolling thin film oven (RTFO), to simulate the oxidation that occurs during the mixing and compaction of HMA following AASHTO T 240-13. To simulate the long-term aging that occurs throughout the service life of a pavement, both the asphalt binder types (BioMAG and Ext BioMAG) were further aged using a pressure aging vessel (PAV) following AASHTO R 28-12. This technique of long-term aging simulates 7-10 years of aging occurring in the field.

2.3. Preparation of asphalt concrete specimens

The control and BioMAG mixtures obtained from asphalt plant were compacted at 135º C to fabricate cylindrical specimens. These specimens were fabricated at two different diameters of 100 mm and 150 mm, so that they could be used for performing dynamic modulus, Hamburg wheel tracking (HWT), and disk-shaped compact tension (DCT) tests. The HWT and DCT test specimens with diameters of 150 mm were compacted to, respectively, 60.3 mm and 50 mm in height to achieve an air void content of 7 ± 0.5 %. The dynamic modulus test specimens with diameters of 100 mm were each compacted to 150 mm in height to also obtain specimens with 7 ± 0.5% air void contents.

2.4. Characterization of asphalt binder rheology

2.4.1. High-and intermediate-temperature behaviors

In this research, a dynamic shear rheometer (DSR) was used for measuring the rheological properties of asphalt binders at high and intermediate temperatures, so that the complex shear modulus (G^*) and phase angle (δ) values could be calculated. To obtain these values, a frequency sweep was performed at a strain level of 0.001% on each aging level of asphalt binder samples. The temperatures used were 15, 24, 34, 43, and 52° C. The frequencies used ranged from 0.1Hz to 10Hz. Sigmoidal function was used to both predict the complex shear modulus values and construct complex shear modulus master curves [40]. To predict phase angle values and construct the phase angle master curves, a model developed and proposed by Podolsky et al. was adopted [41]. More details regarding the construction of master curves for the asphalt-based materials investigated in this study are provided later in the text. The phase angle can be plotted against G* to obtain a Black diagram. This plot contains useful information on the occurrence of phase transition due to asphalt binder modification [42]. The elastic properties of the asphalt at high temperatures was also determined using DSR by performing multiple stress creep recovery (MSCR) test according to AASHTO T 350-14. This test was performed at the high environmental temperature that the pavement was expected to experience. Since the climate grade was a PG 58, the test was conducted at 58°C. Elastic recovery (R) and non-recoverable creep compliance (J_{nr}) are the values obtained from this test.

2.4.2. Low-temperature and cracking (or Delta T_c) behaviors

The low temperature performance of the asphalt binder was tested using a bending beam rheometer (BBR) following AASHTO T 313. In this test the stiffness and m-value are reported at 60 seconds to determine the critical low temperature. Delta Tc (ΔT_c) is another parameter that can be calculated based on BBR test results. This parameter is determined by the critical low temperature calculated from the stiffness and then subtracted by the critical low temperature calculated from the m-value. ΔT_c is a term that is correlated to non-load associated (or thermal) cracking of HMA [43]. An asphalt binder that is more aged has a lower ΔT_c. This is because the rate of change in stiffness (m-value) is too low, meaning that the asphalt cannot dissipate internal stresses quickly, which often leads to cracking. Adding polymers does not have a clear effect on the ΔT_c, as this value can sometimes underestimate the quality of polymer-modified binders [44], especially when these modifiers are used at high dosage rates. Therefore, to provide a more conclusive remark on thermal cracking performance, the low temperature performance of the mixtures was also evaluated.

2.5. Characterization of asphalt mixture performance

2.5.1. Dynamic modulus behavior

A universal testing machine (UTM) was used for applying sinusoidal loads [45], [46] to perform dynamic modulus test and determine the dynamic modulus ($|E^*|$) and phase angle (δ)

values of asphalt mixture specimens. At varying frequencies and different temperatures, the value of |E*| represents the resistance to deformation [45], and the value of δ represents the extent to which a material exhibits viscous and elastic behaviors [47]. The dynamic modulus test was performed according to AASHTO T 342, except for the frequencies and temperatures suggested by this standard. AASHTO T 342 requires testing 3 replicate specimens at five temperatures of -10, 4.4, 21.1, 37.8, and 54.4°C and six loading frequencies of 0.1, 0.5, 1., 5, 10, and 24 Hz. However, performing the test in such manner would be very time consuming and costly [48], especially given the fact that each mixture group tested in this research had 5 replicates. The reason for increasing the number of replicates in this research was to compensate for the variability in test results due to presence of RAP and enable capturing the influence of bio modifiers used more accurately. To save time, reduce the testing costs, and make the dynamic modulus test more practical, Dougan et al. [49] proposed/found that the time required for performing dynamic modulus test must be shortened. This was included in a final technical report resulting from a research that was sponsored by Connecticut Department of Transportations and performed in cooperation with Federal Highway Administration (FHWA) [49]. Bonaquist et al. [50] were among other researchers who made modifications to reduce the temperatures and frequencies suggested by AASHTO TP 63 (the previous version of AASHTO T 342). They confirmed that the specifications that were included in AASHTO TP 63 resulted in the overlap of dynamic modulus data when constructing the master curves; therefore, Bonaquist et al. proposed reducing the temperatures and frequencies to three temperatures of 4.4, 21.1, and 46.1°C and four frequencies of 0.01, 0.1, 1, 10 Hz. In another study on identification of a practical dynamic modulus protocol - that was conducted by researchers from Iowa State University and FHWA -, it was proved that the use of three test temperatures of 4.4, 21.1, and

37.8°C and six frequencies of 0.1, 0.5, 1, 5, 10, and 25 Hz is adequate enough to build a smooth master curve to characterize the properties of asphalt mixtures in a satisfactory manner. Performing the dynamic modulus following such protocol can result in the reduction of testing time, associated costs, and, in addition, preventing the detachment of Linear Variable Displacement Transducers (LVDTs) from specimens at high temperatures. Therefore, in this research, the dynamic modulus test was performed at temperatures of 4.4, 21.1, and 37.8°C and frequencies of 0.1, 0.5, 1, 5, 10, and 25 Hz. At each temperature, the specimens were temperature conditioned to ensure reaching thermal equilibrium [51].

Sigmoidal function [52] (Eq. 1) can be used for the construction of dynamic modulus and phase angle master curves.

$$\log(|E^*|) = \delta + \frac{\alpha}{1+e^{\beta+\gamma \log\left(\frac{1}{f_r}\right)}} \quad (1)$$

where: δ and $\delta + \alpha$ are, respectively, minimum and maximum modulus values, β and γ are the sigmoidal function shape factors/parameters, and f_r is reduced frequency.

The asphalt research team of Iowa State University, through the advancement of models proposed by Booij and Thoone [53] and Yang and You [54], recently developed a newer model (Eq. 2) [41] to further increase the accuracy of construction of dynamic modulus and phase angle master curves.

$$\delta(f_r) \approx c\frac{\pi}{2}\,\frac{\alpha\gamma}{\left(1+e^{\beta-\gamma\log f_r}\right)^{x}}\,e^{(\beta-\gamma\log f_r)} \quad (2)$$

where:, and C and X are parameters added to the previous models.

In this research, the C, X, and shape parameters were determined by minimizing the sum of squared errors (SSE). This value was calculated for the predicted and measured values of log $|E^*|$ and δ.

2.5.2. Rutting and stripping performance

The resistance of asphalt mixture specimens to rutting and stripping was evaluated using a HWT machine, a machine that has been used since 1970s for this purpose [55]. The HWT machine can be used for testing the specimens in either dry or wet condition, and in this research the test was conducted in the wet condition (AASHTO T 324 -16). Following the guidelines set by Iowa Department of Transportation, the test was performed at a temperature of 50°C. The data obtained were plotted (Fig. 7) to determine the slopes representing the rutting and stripping behaviors and then calculate the stripping inflection points (SIPs).

2.5.3. Low-temperature cracking performance

The DCT test, being a fracture energy-based test, can be used for the evaluation of asphalt mixture resistance to thermal (or low temperature) cracking [56] (ASTM D7317-13). It is known that the presence of RAP results in the reduction of fracture energy [57], and therefore the DCT test can be a dependable characterization method for the evaluation of effectiveness of rejuvenators and polymers. The DCT test is conducted at a displacement-controlled mode, and the tensile force applied causes crack propagation throughout the ligament area of a notched asphalt mixture specimen [56]. The crack mouth opening displacement (CMOD) and load data acquired during the test are used for the quantification of crack propagation [56]. Because of the low temperature grade (e.g., -28°C) of base asphalt binder used in this research, the test was performed at -18°C, a temperature 10°C higher than that of the base binder's low temperature grade. After the analysis of DCT test data (i.e., load versus CMOD data), the peak load and fracture energy, G_f (Eq. 3), values could be identified/calculated.

$$G_f = \frac{W_f}{Area_{lig}} \quad (3)$$

where: W_f is the work of fracture, and $Area_{lig}$ is the ligament area.

3. Results and discussion

3.1. Binders' performance grade (PG), thermal cracking resistance and mass loss evaluation

The asphalt binder samples tested were: the styrene-butadiene-styrene (SBS) modified asphalt binder (referred to as control) that was procured from an asphalt supplier; the asphalt binder sampled from the tanker truck (referred to as BioMAG) in the field before mixing; and the asphalt binder extracted from the field mix (referred to as Ext BioMAG) that was sampled from the asphalt plant. The properties of each binder type are presented in Table 2.

Table 2. Properties of asphalt binders.

Binder	**Formulation**	**PG**	**ΔTc**	**Mass Loss**
Control	58-28H	65.4-31.0	-1.6	0.10%
BioMAG	58-28H + 2.5% PAEHOSO + 3.7% SESO + 0.13% Sulfur	64.2-33.3	-3.4	0.16%
Ext BioMAG	58-28H + 2.5% PAEHOSO + 3.7% SESO + 0.13% Sulfur + RAP	66.2-30.8	-2.2	N/A

Note: all the percentages are calculated as the percent by mass of the control binder (58-28H), and the PGs presented in this table are representative of continuous gradings of asphalt binders that would be graded as PG 64-34 based on Superpave method of mix design.

The addition of poly(acrylated epoxidized high oleic soybean oil) (PAEHOSO), sub-epoxidized soybean oil (SESO) and sulfur before the addition of reclaimed asphalt pavement (RAP) binder shows a softening effect resulting in a lower continuous performance grade (PG). The reduction in the continuous PG is due to the presence of SESO (see Table 2). However, with the addition of RAP the continuous PG increases slightly. This is in accordance with the findings of Wei et al. [29] who investigated the influence of epoxidized soybean oil (ESO) on the properties of asphalt binders subjected to aging. In their study, Wei et al observed a stiffening effect with the increase of aging (similar to the influence of RAP in this research) and identified a softening effect when ESO was used to modify their asphalt binder. The reduction in Delta T_c (ΔT_c) value is indicative of decreased strain tolerance that is not the case in this research, as the significantly improved low-temperature cracking resistance of the BioMag mixture proves the effectiveness of the soybean oil-derived modifiers used (see Table 4). According to Elwardany et al. [44], the measurements performed in the linear viscoelastic response region – especially the ones inducing no damage to asphalt binder – "cannot be used to fundamentally and rigorously predict failure properties and strain tolerance for complex and modified binders." Such shortcoming has been the reason for proposing the use of Delta T_f (ΔT_f) [44] in the ongoing National Corporate Highway Research Program (NCHRP) 09-60 project. After the addition of the RAP binder, the PG temperatures increased and the ΔT_c increased as well that is in contradiction with the findings of Anderson et al. [43]. Typically, the addition of RAP binder causes a significant decrease in ΔT_c. This may be due to the presence of PAEHOSO in the base binder as well as the SBS and unknown additives in the RAP that collectively increase the complexity of the binder and affect the ΔT_c in an unexpected way. Therefore, it would be necessary to test the low temperature cracking resistance of mixture made with BioMAG binder to draw a more conclusive remark.

3.2. Binders' rutting performance evaluation

According to an FHWA Technical Brief, the multiple stress creep recovery (MSCR) is a reliable test method for evaluating the high temperature performance of asphalt binders as it correlates with the rutting performance of asphalt mixtures at a high degree of accuracy [58]. The reason for this reliability is due to a much better correlation of non-recoverable creep compliance (J_{nr}), rather than $|G^*|/\sin\delta$, with pavement rutting performance [58]. The "R" of an asphalt binder, another parameter obtained from the MSCR test, is also critical in increasing the rutting resistance of an asphalt mixture [59]. Adding polymers increases R and decreases the J_{nr}. An increase in R is indicative of a better resistance against rutting, but an increase in J_{nr} indicates a higher rutting potential [60]. The increase in the elastic recovery of BioMAG asphalt binder (see Table 3) is due to the presence of sulfur and PAEHOSO. It is known that the increase in the value of R is indicative of formation of a higher quality/larger polymer structure/network [61]. However, the reason for the slight increase in J_{nr} can be attributed to the presence of SESO, and there is a unanimous agreement on the influence of rejuvenators on increasing the value of non-recoverable creep compliance due to the softening effect of these modifiers [27]. Therefore, it is very important to be cautious when adding RRMs, so that an overdosage would not result in an increase in rutting potential. The addition of RAP, however, resulted in a significant reduction in value of J_{nr} for the Ext BioMAG binder that can be attributed to the high stiffness of RAP binder [62]. The elastic recovery (or R) also increased further with the addition of RAP. This may be due to either the presence of some polymer in the RAP or further curing of the RAP asphalt binder through time and heat during the manufacturing and paving processes.

Table 3. MSCR test results measured at 58° C.

Binder	**Formulation**	**R (%)**	**Jnr (1/kPa)**
Control	58-28H	34.8	0.94
BioMAG	58-28H + 2.5% PAEHOSO + 3.7% SESO + 0.13% Sulfur	36.1	1.16
Ext BioMAG	58-28H + 2.5% PAEHOSO + 3.7% SESO + 0.13% Sulfur + RAP	42.8	0.81

Note: all the percentages are calculated as the percent by mass of the control binder (58-28H)

3.3. Binders' master curve data analyses

According to Fig. 3b, the modification of the binder using PAEHOSO, SESO, and sulfur resulted in a general softening of the binder, that can be attributed to the presence of SESO. Wei et. al, [29] also observed a general softening effect, e.g., decrease of G* and increase of δ, due to the modification of their asphalt binder with a soybean oil-derived rejuvenator. At very high testing frequencies, representing very low temperatures, the stiffness values are comparable. The extracted binder, Ext BioMAG, shows a stiffening effect due to the presence of RAP binder that results in obtaining complex shear modulus values higher than those of the control binder. The phase angle data presented in Fig. 3a show the peak in the curve shifts significantly to the right for the modified asphalt binder, BioMAG. This shows that at high temperatures, or low frequencies, the binder will have more elastic behavior, and at low temperatures, or high frequencies, the binder will have more viscous behavior. This is very favorable for the physical properties of the asphalt binder. This means that at low temperatures the BioMAG binder will be able to dissipate internal stresses more quickly through viscous flow, and at high temperatures the binder will resist deformation due to a higher elastic response. Therefore, it can be concluded that the modification of asphalt binder with PAEHOSO and SESO can improve the low temperature and high temperature performance of asphalt binder. The extracted binder, however, shifted the peak back to the left similar to the unmodified binder. This may be caused by a dilution of the polymer network by the RAP binder or impediment of the effect of PAEHOSO and SESO by the large increase in stiffness. Therefore, if these two additives are not added, the asphalt binder would be stiffer and there would be a high possibility for the asphalt mixture to fail prematurely.

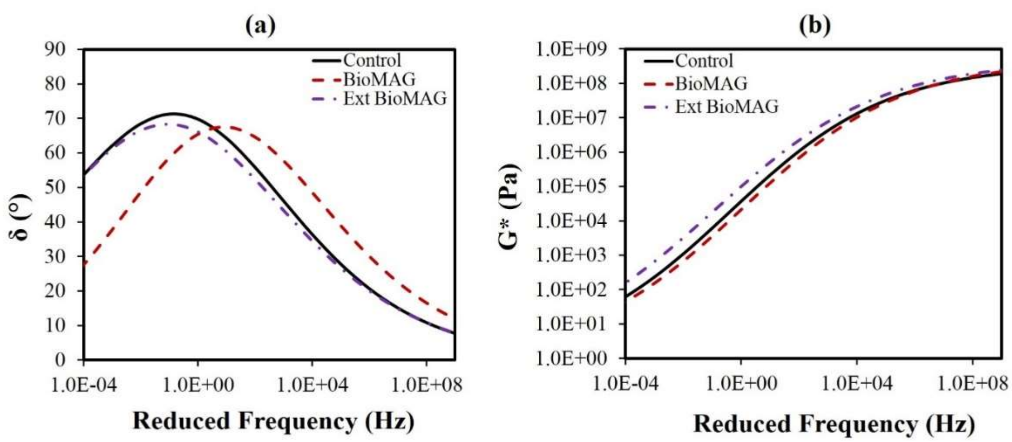

Fig. 3. Phase angle (a) and complex shear modulus (b) master curves constructed for asphalt binders at a reference temperature of 34°C.

3.4. Binders' Black space diagram data analysis

The continuity in the Black space diagrams (Fig. 4) is indicative of homogeneity of asphalt binders and occurrence of no phase transition due to asphalt binder modification [42]. These diagrams also support the previous prediction that the RAP binder disrupts the effect of the PAEHOSO and SESO or dilutes the polymer network.

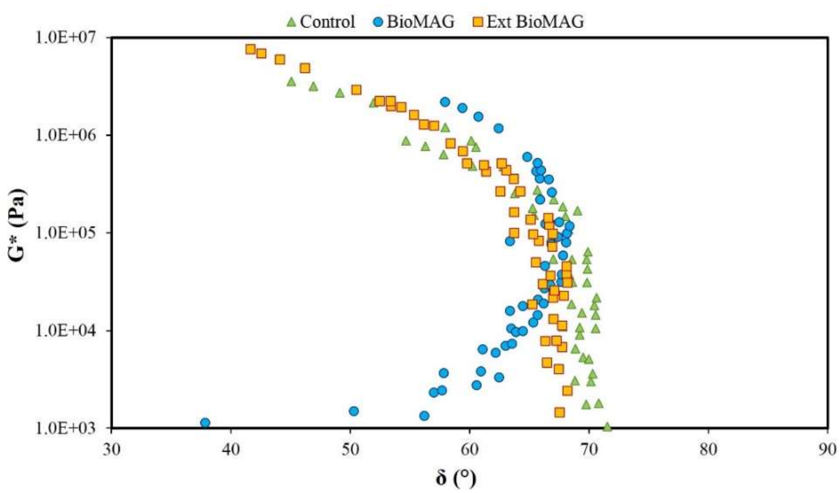

Fig. 4. Black space diagram of the control, BioMAG and extracted BioMAG asphalt binders.

In a Black space diagram, at a given complex shear modulus, a binder with a higher phase angle is more viscous than a binder with a lower phase angle [63]. According to Fig. 4, the modified binder, BioMAG, shows unique behavior. At high stiffness, or low temperatures, the binder is more elastic, then at intermediate temperatures the phase angle reaches a maximum around 70 degrees, then reduces again at low stiffness, or high temperatures. This can be attributed to the higher elasticity of this binder at lower temperatures [63], but as temperature increases, the binder becomes softer and more viscous. At some intermediate temperature the behavior starts to become dominated by the polymer network, causing the phase angle to decrease again. The extracted binder, Ext BioMAG, shows a similar trend to the unmodified binder with a slightly lower phase angle at lower stiffness values. This shows that the extracted binder had a more elastic behavior.

3.5. Mixtures' master curve data analysis

Fig. 5 shows the master curves constructed for the asphalt mixtures. The higher the phase angle, the higher the relaxation ability, and hence the better the resistance to cracking [45], [64]. Also, the higher the dynamic modulus, the higher the stiffness and hence the higher the resistance to deformation [45], [64]. Based on the time-temperature superposition principle, the behavior of asphalt materials at high and low frequencies is identical to their behavior at, respectively, low and high temperatures. Therefore, these variables (i.e., time and frequency) can be used interchangeably throughout the text. According to Fig. 5a, the phase angle of BioMAG mixture is smaller than that of control mixture that can be attributed to the presence of PAEHOSO. This polymer-based modifier, through formation of polymer networks made of large polymers, increases the resistance to rutting (or permanent deformation) at high temperatures. The SESO, also, could have increased the penetration of BioMAG asphalt binder into the aggregate water permeable voids, increased the bind strength at the interface of aggregate and asphalt binder, and hence decreased the deformation/phase angle values at high temperatures. Such improved performance, due to the presence of SESO, can be attributed to the ability of this reactive rejuvenator (or RRM) to de-aggregate asphaltenes and reduce their radii of gyration [32]. To verify such hypothesis, however, it would be necessary to conduct adhesions tests in the future studies.

According to Daniel et al., the transition from the "more" vicous to "more" elastic bhavior is the reason for the occurenece of inflection point (or peak) in phase angle masture curves [65]. It is known that the aggregates and binders are the reasons for, respectively, elastic and viscous behaviors in asphalt mixture [65]. Therefore, the reason for observing a shift in the phase angle master curve for the BioMAG mixture (Fig. 5a) can be attributed to the presence of SESO that has a softening effect on the asphalt binder.

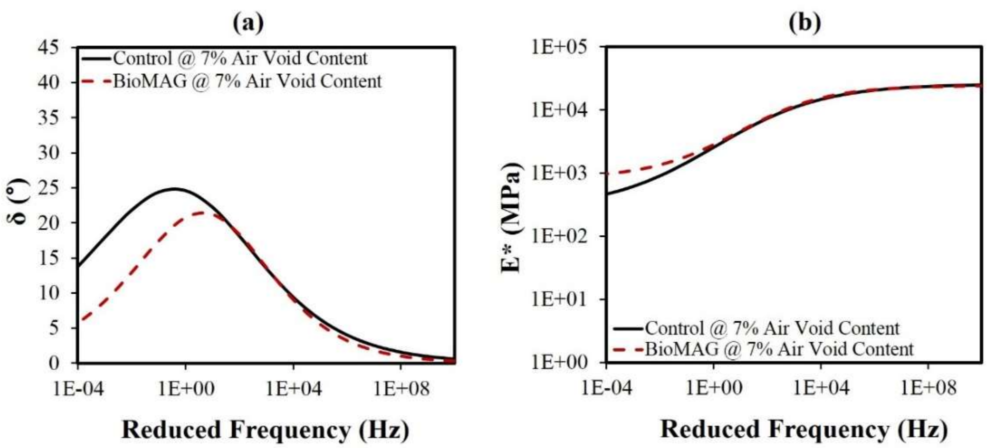

Fig. 5. Phase angle (a) and dynamic modulus (b) master curves constructed for asphalt mixtures at a reference temperature of 21°C.

As it can be seen in Fig. 5b, the presence of bio modifiers (i.e., SESO and PAEHOSO) results in increasing the dynamic modulus values at high temperatures proving the increase to rutting resistance. At low temperatures, however, these modifiers do not result in a significant change in dynamic modulus and phase angle values of the asphalt mixture (Fig. 5). As a result, it would be necessary to evaluate the change in low temperature cracking resistance when the SESO and PAEHOSO are used for binder modification.

3.6. Mixtures' thermal cracking resistance

The aged asphalt binder in RAP [66] decreases the fracture energy and hence increases the brittleness of RAP-incorporated asphalt mixtures [67]. The brittleness or ductility of asphalt mixtures can be evaluated using fracture mechanics-based numerical methods [68]. Dave et al. [69] were among the many researchers who proved that lower fracture energy (or higher brittleness) results in the increase of transverse cracking occurrence frequency. Fig. 6 provides an example of the difference between the behaviors of control and BioMAG mixtures tested using a DCT machine. Table 4 provides more detailed information on the data analyzed for the DCT test. As it can be interpreted from the table, the greater fracture energy value calculated for the bio modified mixture proves that SESO was able to successfully restore the BioMAG mixture's relaxation capability that can also be proved with the larger area under the curve drawn for this mixture (Fig. 6). Such behavior is in agreement with the finding of Santos et al. [70] who proved the capability of bio oil-based rejuvenators in transforming the chemical composition and physical properties of aged asphalt binder (or RAP) resulting in the reforming/repairing/rebalancing the colloidal structure and hence the improvement of properties of asphalt materials. The results presented in this current research are also in accordance with the findings of Huang et al.[71] who proved enhancement in thermal properties of RAP binders modified with a vegetable-oil based rejuvenator. The presence of PAEHOSO was another reason for increasing the thermal cracking resistance, as this biopolymer through reacting with SBS and producing a larger polymer network increases the elasticity and hence the thermal cracking resistance of the mixture; it is known that polymers increase the resistance to thermal cracking [56]. Fig. 6 and Table 4 also shw that the fast rate of loading used for conducting the DCT test [72] resulted in lack of a significant difference between the peak loads of the mixture groups evaluated in this research.

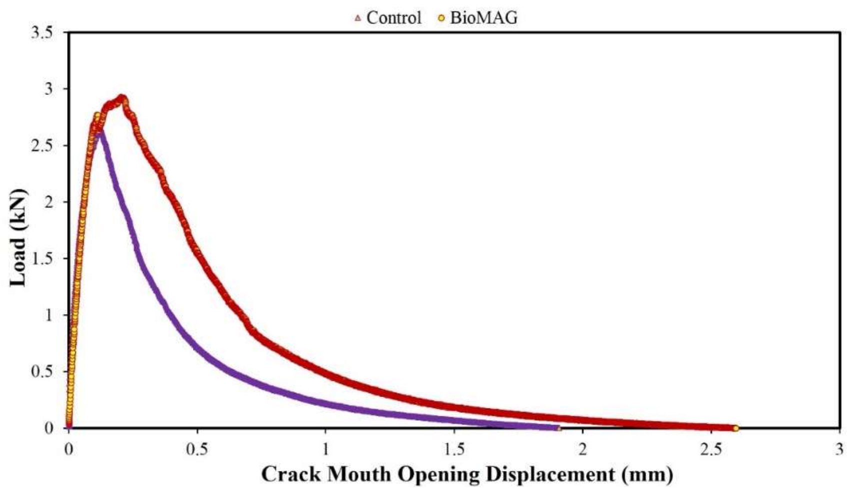

Fig. 6. Sample DCT test data plotted for two mixture groups.

In another study that was conducted by researchers from Iowa State University, it was found that the passing threshold/minimum for the acceptance of a mixture – in terms of low-temperature cracking resistance – is a fracture value of 400 J/m^2 [73], so the control mixture evaluated in this study would not satisfy the criterion and must be rejected based on this finding.

Table 4. DCT test results.

Specimen	Fracture Energy (J/m^2)		Maximum Load (kN)	
Type	Avg.	SE	Avg.	SE
Control	355	26	2.73	0.03
BioMAG	468	28	2.94	0.16

Note: Avg = Average and SE = Standard Error.

3.7. Mixtures' rutting and stripping resistance

The stripping and rutting behaviors of asphalt mixture can be interpreted from the slopes of the lines drawn on the linear regions of HWT test data (see Fig. 7). Steeper slopes indicate less resistance to rutting and moisture damage. Therefore, the control asphalt mixture is more prone to the occurrence of these distresses (see Fig. 7 and Table 5). The superior elastic recovery of asphalt binder used in BioMAG asphalt mixture can justify the enhanced resistance to rutting. As it was indicated earlier in the text, the PAEHOSO through the formation of larger polymers has a significant contribution to increasing the elastic recovery of asphalt binder. This improved behavior is also in agreement with the findings of other researchers who investigated the influence of other soybean-derived additives/modifiers on enhancing the rutting performance of asphalt mixtures [74].

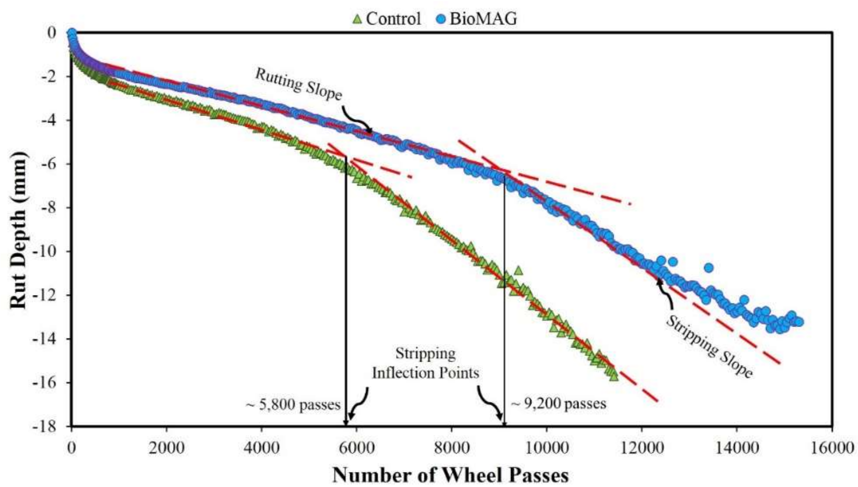

Fig. 7. Sample HWT test data plotted and analyzed for two mixture groups.

The asphalt binder cohesion and aggregate-binder adhesion are the important mechanisms responsible for holding the aggregate skeleton together. Infiltration of moisture into an asphalt mixture due to precipitation, rising ground water, etc. can disrupt these mechanisms [75]. The increase in stripping resistance observed in BioMAG mixture is due to the enhanced work of adhesion [76] in the aggregated-asphalt binder interface that can be attributed to the influence of PAEHOSO that is made of epoxidized soybean oil. The epoxidized soybean oil, being a water-repellent materials, has been used for increasing the hydrophobicity (or water-repellency) of bio-and polymer-based materials [77]. Such feature, therefore, results in a less tendency in modified asphalt binder to retain water at the aggregate-binder interface resulting in enhanced adhesion of binder to aggregate and hence an improved moisture damage resistance in asphalt mixture.

Table 5. HWT test results.

Specimen Type	**Rutting Slope**		**Stripping Slope**		**Stripping Inflection Point (No. of Passes)**	
	Avg.	**SE**	**Avg.**	**SE**	**Avg.**	**SE**
Control	- 0.56	0.03	-3.50	0.23	6,391	626
BioMAG	- 0.28	0.04	-2.17	0.16	10,010	919

Note: Avg = Average and SE = Standard Error.

The superior performance observed in the BioMAG asphalt mixture is a proof for the selection of dosage rates of PAEHOSO and SESO modifiers at appropriate/accurate contents, as overdosage of rejuvenators can increase the moisture damage potential [78].

4. Conclusions and Recommendations

In this research, the rheological properties of asphalt binder modified with poly(acrylated epoxidized high oleic soybean oil) (PAEHOSO) and sub-epoxidized soybean oil (SESO) were thoroughly investigated. This modified asphalt binder, referred to as BioMAG binder, was prepared at an asphalt binder terminal and was then transferred to an asphalt plant for producing a BioMAG asphalt mixture for a pavement demonstration project. Asphalt binder and loose asphalt mixture samples were procured from the terminal and the plant to perform quality control, investigate the addition influence of BioMAG on mitigating the undesired effects of reclaimed asphalt pavement (RAP), and predict the behavior of BioMAG mixture in the field. This research highlights the simultaneous addition influence of PAEHOSO and SESO on changing the properties of asphalt binder and asphalt mixture:

- The addition of SESO and PAEHOSO, through neutralizing the influence of RAP and improving the high-temperature properties, resulted in a continuous performance grade (PG) comparable to the base binder.
- The non-recoverable creep compliance (J_{nr}) of base binder increased due to the presence of SESO that proves the softening effect of this modifiers, and the elastic recovery (R) of the base binder increased due to the presence of PAEHOSO proving the effectiveness of this biopolymer for developing larger polymer networks.
- The bio-binder (or BioMAG binder) obtained through the modification of base binder with the SESO and PAEHOSO could result in improving the J_{nr} and R values, and the RAP binder - possibly previously modified with polymer (s) - had a positive influence on increasing the elasticity.
- Based on the frequency sweep test results obtained from the base and modified binders, the SESO and PAEHOSO decrease the stiffness and increase the elasticity of base binder at lower frequencies, and maintain the stiffness and decrease the elasticity of base binder at higher frequencies.
- Based on the dynamic modulus test data, the BioMAG binder's behavior was indicative of an improved resistance to rutting, as this bio-modified binder, through providing a larger polymer network and possibly providing an enhanced adhesion at the aggregate-binder interface, resulted in the increase of elasticity and stiffness at lower frequencies, or higher temperatures.
- The BioMAG binder resulted in a significant improvement in the low-temperature cracking and rutting resistance of RAP-incorporated asphalt mixtures, and such performance is a

proof for the ability of the BioMAG binder in reversing the undesired effects of RAP and improving the high-temperature properties of asphalt materials assessed in this research.

- The BioMAG binder improved the moisture damage resistance of the mixture, and such improvement can be attributed to the hydrophobic properties of PAEHOSO that can result in the repellence of water at the aggregate-asphalt binder interface [77].
- The Delta T_c (ΔT_c), being a low strain test, was not able to properly characterize the cracking resistance of biopolymer (or PAEHOSO)-modified binders; therefore, there is a need to identify/utilize suitable testing methods such as using asphalt binder cracking device (ABCD) to then measure Delta T_f (ΔT_f) [44] that can reflect the positive influence of polymer modification on increasing the resistance to cracking.
- In the future, to further investigate the effectiveness of the SESO used in this research, it would be necessary to perform chemical analysis tests such as Fourier transform infrared (FTIR) spectroscopy to track oxidation, modification, and chemical functional groups of bio-modified asphalt binders at different aging levels.
- In addition to the FTIR spectroscopy, conducting pyrolysis/gas chromatography–mass spectrometry [79] can help with characterizing the composition of the SESO before and after aging, and identifying the molecular weight distribution of the bio-modified binder used in this study.

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