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COMBINING ASPHALT-RUBBER (AR) AND FAST-PYROLYSIS BIO-OIL TO CREATE A BINDER FOR FLEXIBLE PAVEMENTS

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ABSTRACT
The bio-oil from fast pyrolysis is mainly a product of the recycling of waste materials. This is a viscoelastic material, and after a heat treatment it has a viscosity similar to many types of asphalt used in the paving industry. Although bio-oil showed very good high temperature performance, the same was not verified at low temperatures. Therefore, GTR from cryogenic milling was used to modify the bio-oil. Then, a blend was produced by adding 20% (w/w) of this bio-binder to a PG 64-22 asphalt. The resulting binder was aged, and the storage stability test (separation sensibility) was performed. The initial GTR, bio-oil, bio-binder, asphalt and resulting binder were tested by means of Fourier Transform Infrared Spectroscopy (FTIR) before and after aging. The Dynamic Shear Rheometer (DSR) was used to build the master curves of all the materials and the binders high temperature continuous performance grade was determined. Two mixes compacted for 4% and 7% air voids were studied in regard to water susceptibility, fatigue cracking, dynamic modulus, flow number and low temperature fracture resistance. The results showed that this binder can perform as well or better than conventional asphalts over a large range of temperatures.

Keywords: Bio-binder; asphalt pavements; fast-pyrolysis bio-oil; asphalt-rubber; grounded used tire rubber

INTRODUCTION
Bio-oil has been studied recently as an asphalt substitute in the production of asphalt mixes for construction of flexible pavements. Although it presents a rheological performance very similar to conventional asphalt at high and intermediate temperatures [1], at low temperatures this binder is too stiff and quite brittle. Therefore, some additives can be used to improve the bio-oil characteristics at low temperatures, such as, ground rubber from used tires (GTR). The addition of GTR to bio oil resulted in a new environmental friendly material, the bio-binder, which showed good performance at low temperatures and improved the bio-oil performance at high and intermediate temperatures [2, 3]. However, the good performance of the bio-binder needs to be confirmed and tested in the production of asphalt mixes, and that phase of development is presented in this paper. Several performance tests can be used to characterize asphalt mixes giving a broad view about the mixes performance. These tests are especially important when, as in this case, a material is being used for the very first time, and no previous research data can be found on the subject.
Moisture susceptibility is a problem that typically leads to the stripping of the asphalt binder from the aggregates, and this stripping makes an asphalt concrete mixture ravel and disintegrate [4]. Moisture can damage hot mix asphalt (HMA) in two ways: (i) loss of bond between asphalt cement

or mastic and fine and coarse aggregate and (ii) weakening of mastic due to the presence of moisture [5}. A loss of the adhesive bond between aggregate and asphalt can lead to stripping and raveling, while a loss of cohesion can lead to a weakened pavement that is susceptible to premature cracking and pore pressure damage (6, 7]. AASHTO T 283 – 2007 [8] is the most commonly used test method for determining moisture susceptibility of HMA [5].

The permanent deformation (rutting) of asphalt pavements has a major impact on the performance of a pavement throughout its life. Rutting not only reduces the useful service life of pavements, but it may also affect basic vehicle handling maneuvers, which can be hazardous to highway users. Rutting develops gradually as the number of load applications increases. It is caused by a combination of densification and shear deformation [9].

The dynamic modulus has gained attention recently, since it has become the main input in the MEPDG to determine the temperature and rate temperature dependent behavior of an asphalt concrete layer, as well as to predict rutting in asphalt mixtures [4]. Deformation or complex modulus (E*) corresponds to the ratio between the amplitude of the applied tension and the corresponding extension in certain conditions of temperature and frequency. Typically these properties are measured by repeated loading tests, in which a tension varying cyclically over time is applied. The dynamic modulus measured in these tests is "the absolute value of complex modulus |E*| that defines the elastic properties of a viscoelastic material subjected to a linear sinusoidal load" without rest periods ([10]. However, it is felt that an additional test should be employed in conjunction with dynamic modulus for evaluation of the rutting performance of mixtures [11]. The Triaxial Repeated Load Permanent Deformation (TRLPD) can be used to evaluate the rutting resistance of HMA materials. The Flow Number (Fn), which is the number of load cycles the pavement can tolerate before it flowsis also an output of the TRLPD test [4].

The Semi-Circular Bending (SCB) test provides measures of the stiffness, fracture toughness, and fracture energy of HMA, giving valuable information about the fracture mechanics and susceptibility to cracking at low temperatures [12]. This test employs the crack mouth opening displacement (CMOD) as the feedback signal. The load-line displacement (u) and the CMOD are measured by extensometers. The load, deflection and CMOD are recorded, and the load and load-line displacement (P-u) curve is also plotted when tests are performed at low temperatures [4].

Fatigue resistance is used in the analysis and design of pavements to predict the pavement life. The fatigue characteristics of asphalt mixtures are usually expressed as relationships between the initial stress or strain and the number of load repetitions to failure, determined by using repeated flexure, direct tension, or diametrical tests performed at several stress or strain levels [13]. Two constants (K_1 and K_2), obtained from a statistical analysis, are involved in this relationship. These constants correspond to the intercept and slope of the fatigue line in the log-log scale [14].

Presently, the installed capacity for production of bio-oil suitable for pavement construction is very low. Therefore, from a safe and reasonable perspective for the near term, a new binder comprising a blend of conventional asphalt and bio-binder was studied in this work. Then, mixtures were produced with this new binder, and their performance in lab was assessed in order to certify the suitability and applicability of the binder in the construction of flexible pavements.

EXPERIMENTAL PLAN

Materials

The materials used in this work for the production and study of the new binder were, bio-oil from the fast pyrolysis of red oak wood wastes supplied by Ensyn (bio-oil), ground tire rubber (GTR) from cryogenic milling supplied by Lehigh Technologies under the designation of MicroDyne™ MD 184 TR – 180 µm to 75 µm (80 - 200 mesh) (cryoGTR), as well as, a conventional asphalt binder (PG 64-22), supplied by Seneca Petroleum.

Several different aggregate fractions from Iowa quarries were used to obtain a final aggregate gradation with 9.5 mm nominal maximum aggregate size (NMAS). The aggregates used were:

- 12.5 mm crushed limestone with a NMAS of 9.5 mm;
- 9.5 mm crushed limestone with a NMAS of 4.75 mm;
- Natural Sand with a NMAS of 4.75 mm; and
- Aggregate Lime with a NMAS of 2.36 mm.

Experimental procedure

The experimental work starts with the production and testing of several binders. Later, the binder selected in this first phase of the work is used in the production of asphalt mixtures. These mixtures were finally tested in order to estimate their field performance in the pavement.

Production and testing of binders - The bio-oil was modified with GTR from cryogenic milling (cryoGTR). The bio-oil was placed in a Silverstone Shear Mill at 95 °C with an agitation of around 1000 rpm, until the bio-oil stops boiling (around 15 minutes). Then, 15 wt% of rubber was added and blended with the bio-oil, and the velocity was raised to 3000 rpm, while the temperature was steadily incremented until 120 °C. These conditions were maintained for some time in order to facilitate the interaction between bio-oil and rubber. Samples were collected after 60 minutes of interaction. The asphalt binder (PG64-22) was blended with 20 wt% of the modified bio-binder (cryoMBO), in a Silverstone Shear Mill, at a velocity of 3000 rpm, for 20 minutes and at 130 °C. Finally, the binder previously produced was aged with the rolling thin film oven test (RTFO), according to ASTM D2872-04 standard, although the aging temperature was changed to 140 °C.

To assess the interaction and differences between the materials used to produce the binders, they were tested by FTIR. Then a Dynamic Shear Rheometer (DSR) was used to obtain the rheometer master curves and high temperature performance grades of the binders. At last some properties required to do the formulation of the mixes were assessed, namely, the mixing and compaction temperatures using the Brookfield viscometer, the binders density and storage stability.

Production and testing of asphalt mixtures - The optimum binder content was determined as being 5.5% using the SuperPave mix design methodology [12]. The mixtures were designed for a traffic level between 3 and 30 millions of Equivalent Single Axle Loads (ESAL). The mixing temperature was 150 °C and the specimens produced with the mixture were compacted at 140 °C. With the mentioned materials, 4% and 7% air voids air void (AV) specimens were prepared for the performance tests.

The moisture sensibility of the mixtures was determined according to AASHTO T283-07 [8] standard. The fatigue cracking resistance of the mixtures was evaluated according to AASHTO T321-07 [15]. The dynamic modulus of the mixtures was accessed in accordance with the AASHTO TP79-10 standard [16]. The triaxial repeated loading test [16] was used to determine the flow number. The semicircular bending test [17] was used to evaluate the low temperature cracking performance of the mixture in the pavement at 0, - 12 and - 24 ºC,

RESULTS AND DISCUSSION

Binders testing

The results of the binder testing are presented sequentially, in the same order as they were described when the corresponding methods were presented.

The differences between the binder materials were analyzed by FTIR (Fig. 1).

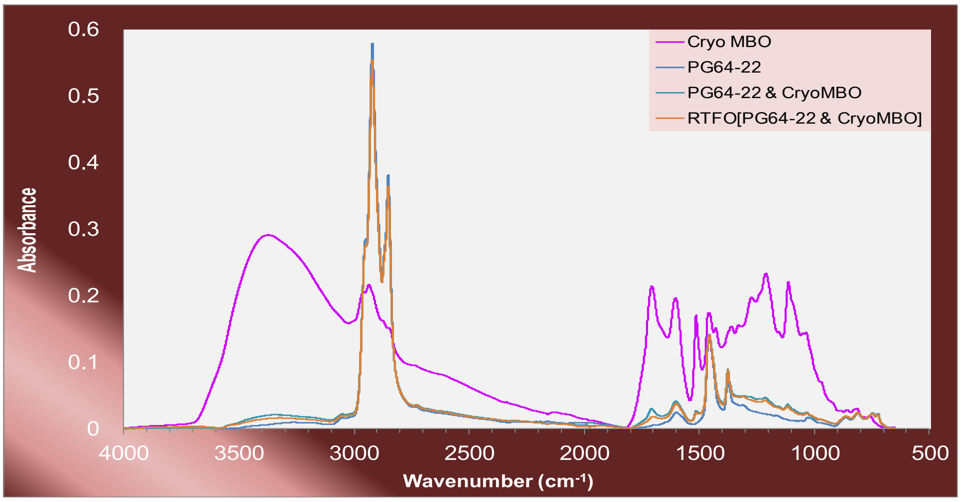

Fig. 1. FTIR spectra of the base materials, un-aged and RTFO aged combined binder

The comparison between the FTIR spectra is a preliminary approach to the chemical transformations that occur during the bio-binder production. The FTIR results are a very useful to show chemical interactions that are occurring and contributing to the binder materials performance.
Although the spectra of all the binder materials are similar (the results are mainly controlled by asphalt), there are some differences among them. Thus, the reduction of the band at around 3300 cm^{-1} is associated with a decrease of the water content in the RTFO aged materials.
The rheology of the binder materials was assessed with a Dynamic Shear Rheometer (DSR), by carrying out frequency sweeps at different temperatures [18]. The results were then used to build the |G*|/sinδ master curves of the several materials (Fig. 2), and to determine their high temperature continuous performance grade (Table 1) specified in the SuperPave methodology [19].

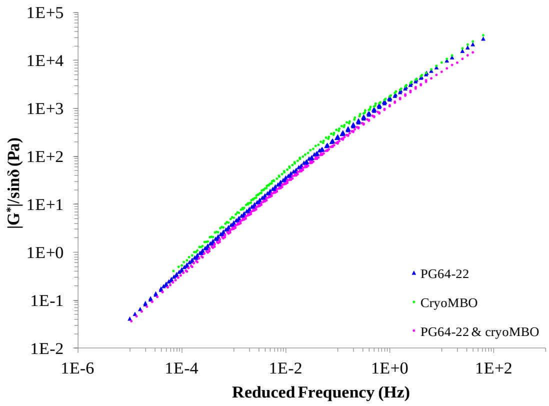

Fig. 2. Master curves of the base materials and the combined binder (T_{ref} = 20 ºC)

Table 1. High temperature continuous performance grade of the un-aged and RTFO aged binder materials

Materials	High Temperature Continuous Performance Grade (°C)	
	Un-Aged	RTFO Aged
PG64-22	66.6	N.A.
cryoMBO	61.3	N.A.
PG64-22 & cryoMBO	65.5	70.7

The master curves of all materials present very similar shapes, indicating that the bio-binder rheological behavior is comparable to the conventional asphalt PG 64-22, and therefore the same is observed in the combined binder.
The blending of conventional asphalt with 20% of bio-binder (cryoMBO) did not change the high temperature performance grade of the base asphalt, and the RTFO aging process caused minor differences in the performance grade of the blend PG64-22 with 20% cryoMBO. In summary, these results indicate that the new blended binders are suitable for flexible pavement construction.
The viscosity of the binder materials was assessed by using a Brookfield viscometer according to ASTM D4402-06 standard [20]. The mixing and compaction temperatures for HMA production were determined to be 160 and 150 ºC, respectively. The combined binder presented mixing and compaction temperatures higher than expected due to the particle effect of the GTR.
The density of the binder materials was measured according to ASTM D70-09 standard [21]. The bio-oil has a high density (1218) when compared to asphalt (1020) resulting in an overall density of the cryoMBO of 1246. The final density of the blend between the asphalt and the cryoMBO (1066) reflects the lower density of asphalt (80%) and the higher density of the bio-binder.
The different densities of the bio-oil and asphalt materials justifies the evaluation of their separation susceptibility [22] at 140 ºC for 24h. The top and bottom fractions high temperature performance grade of 66.2 and 72.7, respectively were determined and used to compute a difference between the performance grade of the top and bottom fractions of 8.8%. The results show that the blend of the PG64-22 asphalt with cryoMBO passed the separation criteria, but it is near the specification limit of 10%.

Mixtures testing

Mixtures designed and prepared according to the SuperPave mix design method (Asphalt Institute, 2001) were compacted to obtain specimens with 4% and 7% air voids. The specimens were tested to evaluate the suitability of these new mixtures to be used in pavements.
The tensile strength ratio (TSR) value, that is the ratio between the indirect tensile strength (IDT) of unconditioned and water conditioned specimens, was used to measure the moisture susceptibility of the studied asphalt mixes. A minimum TSR value of 0.70 is recommended (NCHRP 246), but

more conservative agencies specify a TSR value of 0.80 [23, 5]. The TSR of the studied mixture (0.84) is greater than 0.8 and, thus meeting the Iowa DOT specifications for this property.
The results of the four-point bending beam fatigue tests carried out to evaluate the fatigue cracking resistance of the studied mixtures are summarized in Fig. 3, namely the variation in the number of load cycles (N_f) before fatigue failure in function of the tensile micro strain (με) applied in the test, both for mixes with 4% and 7% air voids contents.

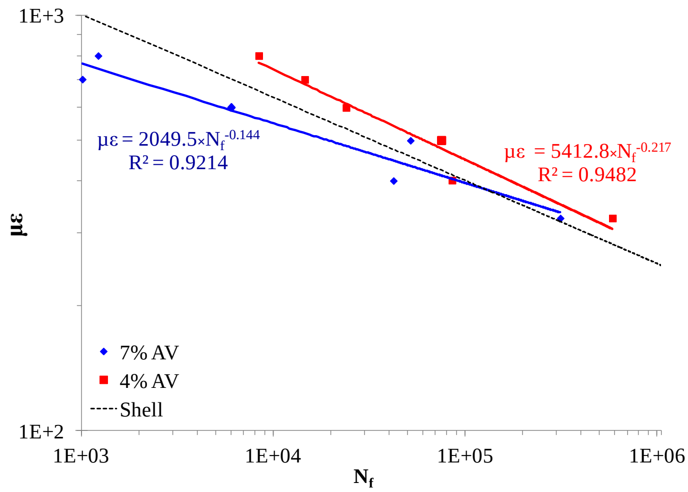

Fig. 3. Variation of the N_f with the με applied in mixtures with 4% and 7% air voids (AV)

The results of Fig. 3 show that the mixture with 7% air voids is less resistant to fatigue cracking than the mixture with 4% air voids, at least in the range of the tensile microstrain values used in the fatigue test.
Shell (1978) [25] proposed a model to predict the fatigue life of a pavement. This model is shown in Fig. 3 for a conventional asphalt mix with 4000 MPa and an asphalt volume content of 12%. The fatigue laws for the mixtures with 4% and 7% air voids contents indicate a fatigue life similar to or slightly higher than the conventional mixtures. The experimental fatigue coefficients K_1 and K_2 were determined to be 2.2E-10 and 4.3675 respectively for the mixture with 4% air voids. The mixture with 7% air voids had fatigue coefficients of K_1=1.2E-17 and K_2=6.4148. Both mixtures show a fatigue performance similar to that of conventional asphalt mixtures.
Fig. 4 shows the master curves of the dynamic modulus for the 4% and 7% air voids mixtures at 4, 21 and 37 ºC [26]. The response measured in the flow number test was the accumulated strain and is presented in Fig. 5.

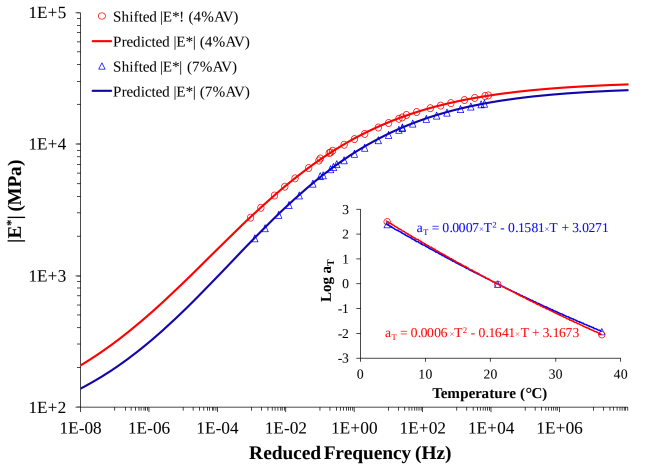

Fig. 4. Master curve of the shifted and predicted dynamic modulus for the 4% AV and 7% AV samples (T_{ref} = 21ºC), with an insert with the quadratic shifting

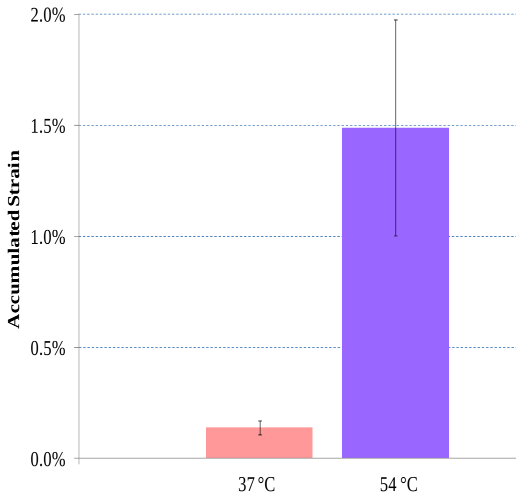

Fig. 5. Accumulated strain at 10,000 cycles for the 7% air voids mixture

The fitted prediction model shows that the asphalt mixture with 4% air voids is stiffer than that with 7% air voids. Thus, it can be concluded that the mixture with 4% air voids should perform better at high temperatures, having a high rutting resistance. Moreover, the values of the dynamic modulus at 37 °C for both mixtures with 4% and 7% air voids are higher than those of conventional asphalt mixtures [27, 28], and thus they should perform properly in rutting.
The flow number of an asphalt mixture corresponds to the number of cycles needed to accumulate 0.5 percent strain in the sample tested. The test ends at 10,000 load cycles even if the sample has not accumulated 0.5 percent strain. A sample that reaches 10,000 load cycles is considered to be essentially rut resistant at the testing temperature. All the samples tested reached 10,000 load cycles at 37 ºC and 54 ºC. The presented values are the average strain levels of three samples in each batch and at each temperature. In comparison with traditional mixtures, the accumulated strain of the mixtures developed in this work is exceptionally low at both temperatures.
The semi-circular bending test (SCB) was performed in order to evaluate the low temperature fracture mechanics of the studied mixtures, and the results obtained with that test for several parameters are plotted in Fig. 6.

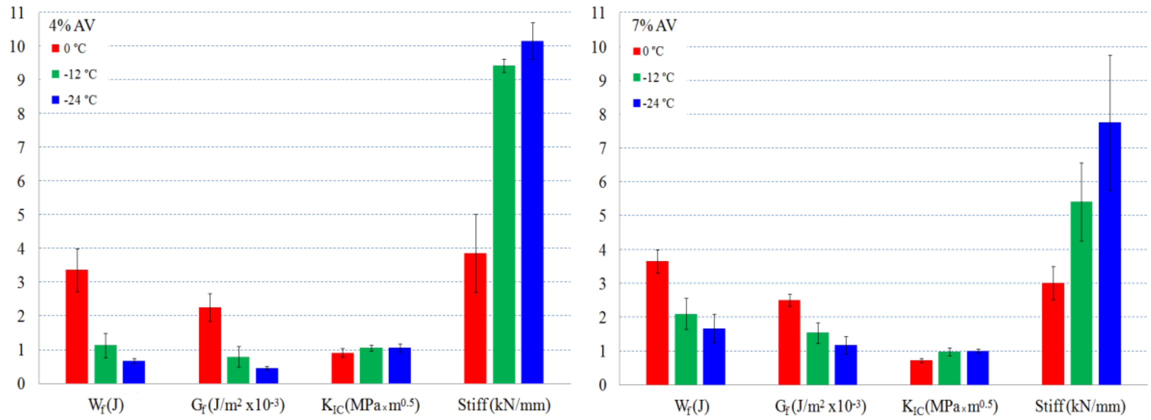

Fig. 6. Low temperature fracture mechanics of the studied mixtures in the SCB test

The results obtained for the newly developed mixes were compared with other results from the literature [27, 29, 30], and it was found that in all cases the mixes studied in this work have higher values for the SCB parameters, especially the stiffness. This indicates, contrary to what was expected, that the designed mixes can resist fracture cracking at low temperatures with their very high stiffness.

CONCLUSIONS

In this study the bio-oil was modified with cryogenic ground tire rubber. This bio-oil binder was then blended with a PG64-22 asphalt. The bio-binder (cryoMBO), the asphalt (PG64 22), and the final blend were then characterized. The tests performed included RTFO aging, FTIR, viscosity, rheology, density and separation susceptibility. The characterization results of these binder showed that the final blends have characteristics similar to those of conventional asphalts.
After the binder study, the blend of the PG64-22 asphalt with 20% cryoMBO was mixed with a 9.5 mm NMAS Iowa aggregates. Afterwards, flexural fatigue cracking, moisture susceptibility, dynamic modulus, rutting resistance and low temperature facture tests were performed to evaluate the performance of the mixtures developed in this work. The mixtures showed very good performance in all the tests carried out, and thus it is not expected to see any rutting or early fatigue cracking distresses, and these mixtures should not be sensitive to moisture nor to low temperature cracking. The very good performance of the new mixtures developed in this work should now be validated in a pavement trial, after asphalt plant production, in order to persuade the road administration to use higher quantities of bio-oil as an alternative binder material in pavement works.

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