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Recombination of Asphalt with Bio-Asphalt: Binder Formulation and Asphalt Mixes Application

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Abstract
The bio-oil from fast pyrolysis is mainly produced using organic waste materials. This is a viscoelastic material, and after a heat treatment it has a viscosity and high/intermediate thermal rheological behavior similar to many types of asphalt used in the paving industry. These two characteristics show that this material could be a good alternative to replace asphalt. In order to improve the performance of bio-oil, it was hypothesized that the addition of crumb rubber would change the rheology of the modified bio-oil, making it rheologically similar to the conventional paving asphalts. Therefore, two sources of ground rubber from used tires (GTR), from different manufacturing processes, were used to modify the bio-oil. Then, two blends were produced by adding 20% (w/w) of this bio-binder to two different asphalts, a PG58-28 and a PG64-22. The binders were aged, and then storage stability tests (separation sensibility) were performed. The rheology of the initial bio-oil, bio-binder, asphalts and resulting binder-blends were assessed by using a Dynamic Shear Rheometer (DSR), namely by performing frequency sweeps at different temperatures. The results were then used to build the master curves of the materials, and to determine their high temperature continuous performance grade. Additionally, the performance related behavior of mixtures produced with this new material was also assessed, in order to evaluate the advantages of its use in pavements. Therefore, two mixes were produced with the binder that showed better performance regarding thermal rheological behavior, aging susceptibility and separation tendency. These new mixes were finally studied using performance related tests that are able to estimate their future behavior *in situ* in different environmental and traffic conditions, in particular in regard to water susceptibility, fatigue cracking, dynamic modulus, flow number and low temperature fracture resistance. The results from this first set of experiments showed that this material can perform as well or better than conventional asphalts over a large range of temperatures.

Keywords
Bio-binder, Bio-oil, fast-pyrolysis, rubber, asphalt-rubber, aging

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Recombination of Asphalt with Bio-Asphalt: Binder Formulation and Asphalt Mixes Application

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The bio-oil from fast pyrolysis is mainly produced using organic waste materials. This is a viscoelastic material, and after a heat treatment it has a viscosity and high/intermediate thermal rheological behavior similar to many types of asphalt used in the paving industry. These two characteristics show that this material could be a good alternative to replace asphalt. In order to improve the performance of bio-oil, it was hypothesized that the addition of crumb rubber would change the rheology of the modified bio-oil, making it rheologically similar to the conventional paving asphalts. Therefore, two sources of ground rubber from used tires (GTR), from different manufacturing processes, were used to modify the bio-oil. Then, two blends were produced by adding 20% (w/w) of this bio-binder to two different asphalts, a PG58-28 and a PG64-22. The binders were aged, and then storage stability tests (separation sensibility) were performed. The rheology of the initial bio-oil, bio-binder, asphalts and resulting binder-blends were assessed by using a Dynamic Shear Rheometer (DSR), namely by performing frequency sweeps at different temperatures. The results were then used to build the master curves of the materials, and to determine their high temperature continuous performance grade. Additionally, the performance related behavior of mixtures produced with this new material was also assessed, in order to evaluate the advantages of its use in pavements. Therefore, two mixes were produced with the binder that showed better performance regarding thermal rheological behavior, aging susceptibility and separation tendency. These new mixes were finally studied using performance related tests that are able to estimate their future behavior in situ in different environmental and traffic conditions, in particular in regard to water susceptibility, fatigue cracking, dynamic modulus, flow number and low temperature fracture resistance. The results from this first set of experiments showed that this material can perform as well or better than conventional asphalts over a large range of temperatures.

Keywords: Bio-binder. Bio-oil, fast-pyrolysis, rubber, asphalt-rubber, aging

1. Introduction

Asphalt or bitumen, is “a black sticky substance that is used for making roads” (Macmillan, 2013). Although this material is usually associated with a residue from petroleum distillation, the bio-oil from fast pyrolysis of agriculture and forestry residues can also be included in the previous definition when used as a binder in flexible pavement construction (bio-binder or bioasphalt).

The substantial increase in oil prices over the past five years was reflected in asphalt prices and also caused a reduction in its supply due to the maximization of fuel production in refineries. In fact, many refineries have installed coking facilities to further increase the production yield of transportation fuels with the consequent reduction in the supply of asphalt. This has led to (i) the production of a distillation residue that cannot be used

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as binder in asphalt mixtures and (ii) the substantial product development of alternative sources of using biological resources such as microalgae (Chailleux *et al.*, 2012); swine manure (Fini *et al.*, 2012; Fini *et al.*, 2011a; Fini *et al.*, 2011b); cornstover (Raouf and Williams, 2010b); swishgrass (Raouf and Williams, 2010a); wood residues (Peralta *et al.*, 2013; Peralta *et al.*, 2012; Raouf, 2010; Raouf and Williams, 2010c; Yang *et al.*, 2014); urban waste (Hill and Jennings, 2011); coffee and tea residues (Chaiya, 2011; Uzun *et al.*, 2010); rapeseed and soybean (Onay and Koçkar, 2006; Şensöz and Kaynar, 2006), among others. Most of these products were developed to replace fuel as an energy source, but some of them have been used as asphalt modifiers, partial replacers or substitutes.

Recent studies suggested the use of bio-oil as an asphalt substitute to produce asphalt mixes for construction of flexible pavements. They showed that it has potential for application on asphalt as an additive, modifier or extender, especially in mixtures with polymers (Williams *et al.*, 2009; Yang *et al.*, 2014). In fact, the bio-oil may even replace the entire asphalt as it presents rheological properties similar to asphalt after polymer modification (Peralta *et al.*, 2012; Raouf, 2010). However, the high melting point of most of the polymers currently used as asphalt modifiers restricts their use with bio-oil, since this last should be handled at lower temperatures. The bio-oil polymer modification is required to improve the pavement performance in a large range of temperatures and loads. Nevertheless, some additives can be used to improve the bio-binders characteristics, such as ground rubber from used tires (GTR). The addition of GTR to bio-oil results in a new environmental friendly material, the bio-binder, which presents a good performance at low temperatures and improves the bio-oil performance at high and intermediate temperatures (Peralta *et al.*, 2013; Peralta *et al.*, 2012). Besides the antioxidant properties of the bio-oil (due to high lignin content), it was also found that it contains significant amounts of furfural, which is beneficial in promoting interactions between asphalt and rubber (Shatanawi *et al.*, 2012).

The benefits of combining waste materials (such as rubber from used tires and residues from agriculture and forestry activities) with asphalt, which is ultimately the residue of crude petroleum distillation, applying well known technologies (such as fast pyrolysis and asphalt-rubber production methods) can change the way binders for flexible pavements are envisioned. However, besides the need of a more profound knowledge of the chemistry and interaction between these different materials, it is necessary to know if this new binder can perform at least as well as conventional asphalt when applied in innovative mixtures for flexible pavements.

The first application of bio-oil in a pavement (6% asphalt replacement) occurred in 2010 in Des Moines (Iowa) (ISU, 2010) and no damage can be noticed presently. Recently, a new work with 5 and 10% replacement of asphalt binder by bio-oil also showed a very good performance in laboratorial tests (Yang *et al.*, 2014). Thus, it is time to move on to higher percentages of asphalt replacement by bio-oil, as this will constitute a technological upgrade with positive consequences, both economically and environmentally.

The main objective of this work is to understand the interaction behaviour of asphalt with bio-oil and ground tire rubber (GTR), to effectively replace part of the petroleum asphalt, by using bio-oil from the fast pyrolysis of agriculture and forestry residues and, as a consequence, optimize the performance of asphalt mixtures with asphalt-rubber (AR).

Thus, this work aims to develop asphalt rubber mixtures with an optimized binder, using petroleum asphalt, bio-oils from fast pyrolysis of biomass, or both combined. These mixtures should have an improved performance throughout the life of the pavement and the required stability during the production and construction stages. The results of this research, development and innovation work should support and promote the future use of this new product in the rehabilitation of the current roadway system, as well as in the construction of new roads and highways, in particular due to the substantial reduction in the environmental and economic costs.

In order to assess the quality of these new mixtures, their behaviour must be characterized in laboratory in order to estimate the future performance in situ. The performance tests used to characterize asphalt mixes cannot give a broad view about their performance when used individually, but when combined they can provide valuable information to preview and understand the field performance of laboratory designed asphalt mixes. In this case, these tests are especially important because these innovative mixtures are being used for the very first time, and no previous research data can be found on the subject. The next paragraphs present the main properties related with the field performance of asphalt mixtures (including those mixtures developed in this work), and the corresponding tests used to assess those properties in the lab.

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 (Brown *et al.*, 2009).

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 (Williams and Breakah, 2010). There are six parameters that have been associated with moisture damage in asphalt mixtures: detachment, displacement, spontaneous emulsification, pore-pressure–induced damage, hydraulic scour, and environmental effects (Little and Jones, 2003; Roberts *et al.*, 1996). None of the above factors necessarily works alone in damaging an asphalt concrete pavement, as they can work in combination to damage a mix. Therefore, it is necessary to examine the adhesive interface between aggregates and asphalt and the cohesive strength and durability of mastics (Cheng *et al.*, 2003; Graf, 1986; Little and Jones, 2003). 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 (Kandhal, 1994; Majidzadeh and Brovold, 1968). AASHTO T 283-07 (2007) is recommended as the final step of the superpave mix design guide (Asphalt Institute, 2001), and thus it is the most commonly used test method for determining moisture susceptibility of HMA mixtures (Airey *et al.*, 2008; Chiu and Lu, 2007; Hajj *et al.*, 2011; Williams and Breakah, 2010), including in the experimental part of this work.

Another property of asphalt pavements that has a major impact on the performance of a pavement throughout its life is the permanent deformation (rutting) resistance. 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. Rutting appears as longitudinal depressions in the wheel paths and small upheavals to the sides. It is caused by a combination of densification and shear deformation (Sousa *et al.*, 1991).

Over the past few years there has been significant interest in establishing a test method that could be universally employed to evaluate the material properties during the hot mix asphalt design, such that it would be indicative of field performance, mainly regarding permanent deformation. Although other tests can be used, like Hamburg wheel tracking test, the main interest has been centered around dynamic modulus test, with primary efforts on refining associated predictive equations. However, it is felt that an additional test should be employed in conjunction with dynamic modulus for evaluation of the rutting performance of mixtures (Kvasnak *et al.*, 2007). Thus, the dynamic modulus test can be complemented by the triaxial compression test, in which a repeated loading is conducted with constant stress confinement, where the vertical load has a sinusoidal variation and the horizontal load (current confinement) is constant throughout the test. One example of this test is the Triaxial Repeated Load Permanent Deformation (TRLPD), which was used in this work to evaluate the rutting resistance of HMA materials. The outputs of this test are the Flow Number (Fn), which is the number of load cycles the pavement can support before it flows; and the permanent strain growth model, used in the Mechanistic-Empirical Pavement Design Guide (MEPDG) (Cheng *et al.*, 2003).

The dynamic modulus of asphalt mixtures 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 (Cheng *et al.*, 2003). These are also the reasons why this test was applied in the experimental part of this work to characterize the new asphalt rubber mixtures with bio-oil. The complex modulus (E^*) corresponds to the ratio between the amplitude of the applied stress (σ_0) and the corresponding strain (ε_0) in certain conditions of temperature (T) and frequency (Freq). These properties are typically measured by using repeated loading tests, namely by applying a stress value varying cyclically over time with an angular velocity $\omega = 2.\pi.\text{Freq}$. 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 (ASTM D 3497-79, 1995). Under certain conditions of temperature and frequency, the value of the complex modulus is strongly influenced by the type of asphalt (or bio-binder), and those mixtures with stiffer asphalt binders have higher modulus (Fontes, 2009).

The low temperature performance of these mixtures with bio-oil must also be evaluated in order to understand if they can be used in pavements subjected to difficult winter conditions, namely by using the Semi-Circular Bending (SCB) test. This test provides a different method of measuring the stiffness, fracture toughness, and fracture energy of HMA mixtures. The semi circular slices used in this test, with 25 mm thickness, are cut from SuperPave Gyratory Compactor specimens (Asphalt Institute, 2001). This test employs the crack mouth opening displacement (CMOD) as the feedback signal, being conducted in a constant CMOD rate mode. The load-line displacement (*u*) and the CMOD are measured by extensometers, also used in the Indirect Tensile Strength test (IDT) setup. 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 (Brown *et al.*, 2009), since it can give valuable information about the fracture mechanics and susceptibility to cracking at low temperatures.

Fatigue resistance is used in the analysis and design of pavements to predict their fatigue life, and this is another performance related property of the asphalt rubber mixtures with bio-oil evaluated in this work. 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 bending, direct tension, or diametrical tests performed at several stress or strain levels (Tayebali *et al.*, 1994). The results of fatigue tests are expressed in terms of the number of cycles before fatigue failure for the tensile strain level applied. 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 (Pais *et al.*, 2009).

It was observed that, 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. These are the main phases of development of the work presented in this paper.

2. Experimental Section

The experimental work consisted in the production, study and selection of a new binder constituted by 80% of a conventional binder and 20% of a rubber modified bio-oil (85% bio-oil with 15% ground tire rubber – GTR), which was then applied in the production of two asphalt mixes. These mixtures were than compacted and characterized with performance related tests in order to verify if they would have adequate performance when applied in pavements.

2.1 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 (bio-oil), and two types of rubber: Ground tire rubber from cryogenic milling supplied by Lehigh Technologies under the designation of MicroDyne™ MD 184 TR – 180 µm to 75 µm (80 - 200 mesh) (cryoGTR); and ground tire rubber from ambient shredding supplied by Seneca Petroleum (30 mesh) (ambGTR). Two conventional asphalt binders supplied by Seneca Petroleum, a PG58-28 and a PG64-22, were also used in this work.

Several different aggregate fractions from Iowa quarries were used to obtain a final aggregate gradation with 9.5 mm nominal maximum aggregate size (NMAS) (Figure 1). 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.

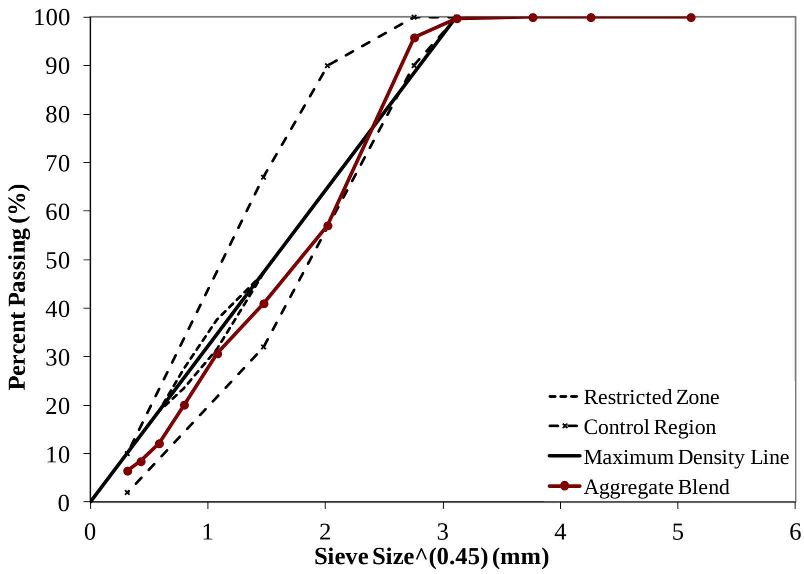

Figure 1. Final aggregate blend of the mixture, restricted zones and control points for an aggregate gradation with a NMAS of 9.5 mm in the 0.45 power chart

2.2 Methods

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 ultimately tested in order to estimate their field performance in the pavement.

2.2.1 Production and testing of binders

Initially, the changes in the viscosity of the bio-oil upon heating were monitored over time in order to evaluate the effect of using this material at high production temperatures. The changes in the bio-oil viscosity were assessed using a Brookfield viscometer. The samples were heated at 95 °C and placed in the testing chamber. The spindle used was the S21 and the velocity was 20 rpm. The sample was tested for 8.5 hours, and readings were recorded every 15 minutes.

The next phase of the work was the removal of free water from the bio oil, upgrading it in order to obtain an adequate viscosity for mixture production (Raouf, 2010; Raouf and Williams, 2009)), as indicated in the Superpave mix design guide (Asphalt Institute, 2001). The bio-oil was placed in a Silverstone Shear Mill at a velocity of 3000 rpm for about one hour. The initial temperature of 90 °C was steadily incremented until 110 °C, in order to maintain the bio-oil steaming (with low boiling). When the bio-oil reached 110 °C, this temperature was maintained until it stopped boiling, for a total time of treatment of 1 hour (Figure 2). The final product obtained at the end of this phase was called “Heat Treated Bio-Oil” (HTBO).

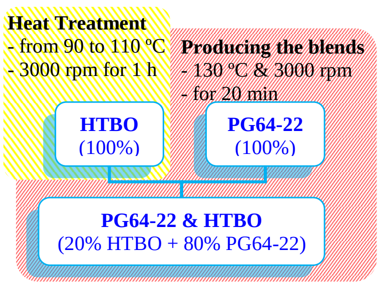

Figure 2. Production of the PG64-22 & HTBO

Another batch of bio-oil was modified with two ground tire rubbers (Figure 3)selected for this study, namely from cryogenic milling and ambient shredding (cryoGTR and ambGTR). The bio-oil was placed in a Silverstone Shear Mill at 95 °C with an agitation of around 1000 rpm, and gradually increased until 110 ºC, until the bio-oil stops boiling (around 15 minutes). During this stage most of the water is removed from the bio-oil, with a mass loss of around 15%. Then, 15wt% of rubber was added and blended with the bio-oil n this study, because that was the rubber content that produces a bio-binder with the better characteristics, as stated in a previous study (Peralta *et al.*, 2012). During this phase the velocity was raised to 3000 rpm, while the temperature was steadily incremented until 120 °C. These conditions were maintained for 1 hour in order to facilitate the interaction between the bio-oil and rubber. Samples were collected after 30 minutes and 1 hour of interaction. Only the material that interacted for 1 hour was subsequently used for testing, because the interaction for 30 minutes was insufficient to obtain a consistency similar to that of a conventional paving asphalt (PG58-28 or PG64-22) at room temperature. Therefore, the modified bio-oils (MBO) produced in this part of the work were the following:

- The material obtained after 1 hour of interaction of the cryogenic rubber (cryoGTR) with the bio-oil is further designated as "cryogenic modified bio-oil" (cryoMBO); and
- After 1 hour of interaction of the ambient rubber (ambGTR) with the bio-oil, the resulting material is further designated as "ambient modified bio-oil" (ambMBO).

The next phase of the work was the production of asphalt binders modified with the previously modified bio-oils (cryoMBO and ambMBO). Previous studies showed that a replacement of asphalt by 5 and 10% of bio-oil produce good results in laboratory pavement performance tests (Yang *et al.*, 2014), Moreover, at the present prices of asphalt and bio-oil, it is estimated that a higher rate of replacement of asphalt (20%) would represent an economical advantage for the paving industry. Thus, the two conventional asphalt binders (PG58-28 and PG64-22) were blended with 20 wt% of the modified bio-oil based materials formerly produced, cryoMBO, ambMBO (Figure 3) and with HTBO (Figure 2). This final process was carried out in a Silverstone Shear Mill, at a velocity of 3000 rpm, for 20 minutes and at 130 °C. This temperature was selected because at higher temperatures the bio-oil based materials will quickly change their properties. The four asphalt binders produced in this phase were:

- Asphalt PG58-28 blended with 20% cryoMBO;
- Asphalt PG64-22 blended with 20% HTBO;
- Asphalt PG64-22 blended with 20% cryoMBO; and
- Asphalt PG64-22 blended with 20% ambMBO.

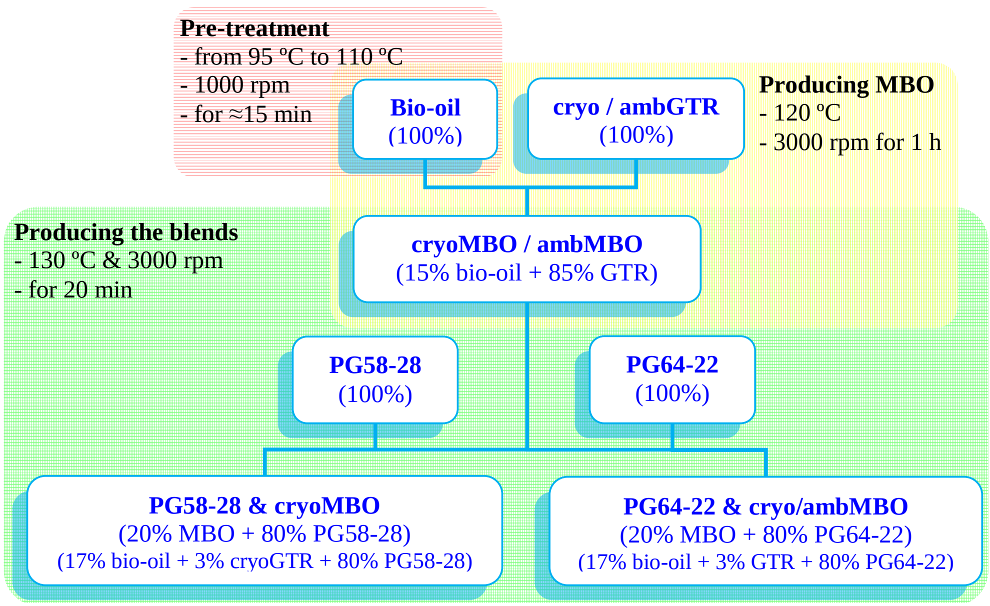

Figure 3. Production of the PG64-22 & cryoMBO, PG64-22 & ambMBO and PG58-28 & cryoMBO

All binders were then aged with the rolling thin film oven test (RTFO) apparatus, according to a modified ASTM D 2872-04 (2004) standard, where the aging temperature was changed to 140 °C. According to the ASTM D 2872-04 (2004) standard, the method can only be properly applied to conventional hot mix asphalt. Otherwise, the method must be changed and the viscosity and other rheological measurements should be taken into account. It is also stated that the method "yields a residue which approximates the asphalt condition as incorporated in the pavement", which implies the mixing, mixtures curing and compaction" (Asphalt Institute, 2003). Thus, the lower temperature used in this work, in comparison with that presented in the standard, was selected in order to better simulate the compaction temperature of the asphalt mixtures applied in the next phase of the work. This reduction of the RTFO test temperature has also been mentioned and justified by several authors (Arega *et al.*, 2011; Gandhi *et al.*, 2009, 2010; Kim *et al.*, 2012). Moreover, according to Hanz *et al.* (2011) the effect of reducing the RTFO test temperature to 120 ºC would cause only a 3 ºC reduction on the continuous PG temperature of the binder (and this difference would necessarily be lower at 140 ºC). Despite the small impact of the reduction of the RTFO test temperature in the binder grade, using a higher temperature would be less representative of the mixing and compaction conditions, because it would cause a binder degradation (Meier *et al.*, 2013; Raouf, 2010) that is being avoided in the actual production of the mixtures.

The four aged binders obtained after the modified RTFO tests were labelled as follows:

- RTFO (Asphalt PG58-28 blended with 20% cryoMBO);
- RTFO (Asphalt PG64-22 blended with 20% HTBO);
- RTFO (Asphalt PG64-22 blended with 20% cryoMBO); and
- RTFO (Asphalt PG64-22 blended with 20% ambMBO).

Additionally, another aging process was applied to assess the mass loss with the reduced amount of oxidation of the binders. The method consisted in the complete filling of eight ounce metal cans with the binders, loosely cover the cans and place them in an oven at 140 °C for 24 hours (the resulting material will be further referred as "oven aged").

Additionally, the evolution of the viscosity of the bio-oil was monitored using a Brookfield viscometer at 90 °C for 8 hours and 30 min.

To assess the interaction and differences between the materials used to produce the binders, they were tested by Fourier Transform Infrared Spectroscopy (FTIR) using a Thermo Scientific Nicolet iS10 (Thermo Scientific®, Hanover Park, IL) equipped with a Smart iTR accessory, utilizing OMNIC Software (Thermo Scientific®, Hanover Park, IL) operating system. The background was collected before every sample and attenuated total reflectance (ATR) correction was used with 4 wavenumber resolution. Spectra were obtained by averaging 32 scans in the range of 4000-650 cm^{-1}. The solid samples were placed on the diamond crystal plate and prepared for analysis with an equipped constant pressure press.

Subsequently, a Dynamic Shear Rheometer (DSR) was used (ASTM D 7175-08, 2008) to obtain the rheometer master curves and high temperature performance grades of the binders.

At last, some properties of the binders required to design the mixes were assessed, namely, the mixing and compaction temperatures using the Brookfield viscometer (ASTM D 4402-06, 2006) the binder's density (ASTM D 70-09, 2009) and the storage stability (ASTM D 7173-05, 2005)..

2.2.2 Production and testing of asphalt mixtures

After analyzing the results of the binders testing, the blend of PG64-22 asphalt with 20% cryoMBO was selected to produce the mixtures in this stage of the work. The optimum binder content was determined as being 5.5% using the SuperPave mix design methodology (Asphalt Institute, 2001).The mixtures were designed for a traffic level between 3 and 30 millions of Equivalent Single Axle Loads (ESALs).

The method used to produce the mixtures started by letting the aggregates in the oven (dry and heat) for 12 hours at 150 ºC. These aggregates were mixed with the just completed binder blend (produced at 130 ºC, as explained previously) for a short period of time (around 5 min) to completely coat the aggregates. The mixtures were then evenly spread in a flat shallow pan and cured in an oven at 140 ºC for two hours, and compacted at the same temperature. The mentioned materials were used to produce specimens for testing with two air voids (AV) contents, namely 4% and 7%, using the gyratory shear compactor and/or the roller compactor. The compacted specimens were tested in order to evaluate some properties related with their field performance in the pavement, according to the procedures presented in the following paragraphs.

The moisture sensibility of the mixtures was determined according to AASHTO T 283-07 (2007) standard test. The main result obtained in this test (Tensile Strength Ratio, or TSR) must be in compliance with the criteria accepted by the road administrations, otherwise the mixture studied in this work could not be considered suitable for paving works, and thus no further tests would be needed.

The fatigue cracking resistance of the mixtures was evaluated according to AASHTO T 321-07 (2007) standard test. This test simulates the repetitive load application of the traffic in the pavement, and the results of this test can show the level of traffic that the mixture will bear in the pavement without cracking at a different tensile strain levels (fatigue law).

The dynamic modulus of the mixtures was accessed in accordance with the ASTM D 3497-79 (1995) and/or AASHTO T 342-11 (2011) standard test procedures. This non destructive test is performed to determine the temperature and rate temperature dependent behavior of asphalt mixtures applied in pavement layers. The results of this test are the main input in the MEPDG pavement design method, thus justifying the assessment of this property for the innovative mixtures developed in this work. This test can also be used to evaluate the pavement ability to resist permanent deformation.

The triaxial repeated load permanent deformation (Witczak, 2005). This non destructive test is performed to determine the temperature and rate temperature dependent behavior of asphalt mixtures applied in pavement layers. The results of this test are the main input in the MEPDG pavement design method, thus justifying the assessment of this property for the innovative mixtures developed in this work. This test can also be used to evaluate the pavement ability to resist permanent deformation (Witczak, 2005).

Finally, the semicircular bending test (AASHTO TP 105-13, 2013) was used to evaluate the low temperature cracking performance of the mixture in the pavement, after confirming that all test results obtained at intermediate temperatures are acceptable according to road administration specifications. This test evaluated the cracking resistance of the studied mixtures at temperatures of 0, 12 and 24 ºC, because the commercial asphalt binder used has a low temperature PG of -22. Moreover, in this work the choice of the low reference temperature for the SCB testing was based on the minimum acceptable low temperature PG of binders typically applied in most of the United States. The result of this test indicates if the bio-oil will reduce the expectable performance of asphalt at this temperature. Ultimately, since the low PG temperature of the binder blend was not determined and after observing that mixtures were so stiff at room temperature, the results of this test are fundamental to clarify the new binder and mixture low temperature performance.

3. Results and Discussion

3.1 Results of the binders testing

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

3.1.1 Changes in the bio-oil viscosity upon heating over time

The initial viscosity of the bio-oil was 42.5 cP and increased following an exponential trend to a final viscosity of 207.5 cP after 8 hours and 30 minutes in the Brookfield viscometer at 95 °C (Figure 4).

The changes in the viscosity of the bio-oil were expected and are mainly caused by polymerization of the phenols combined with the furfural within the bio-oil (Williams *et al.*, 2009), and the volatilization of some free water.mbined with the furfural within the bio-oil (Williams *et al.*, 2009), and to the volatilization of some free water.

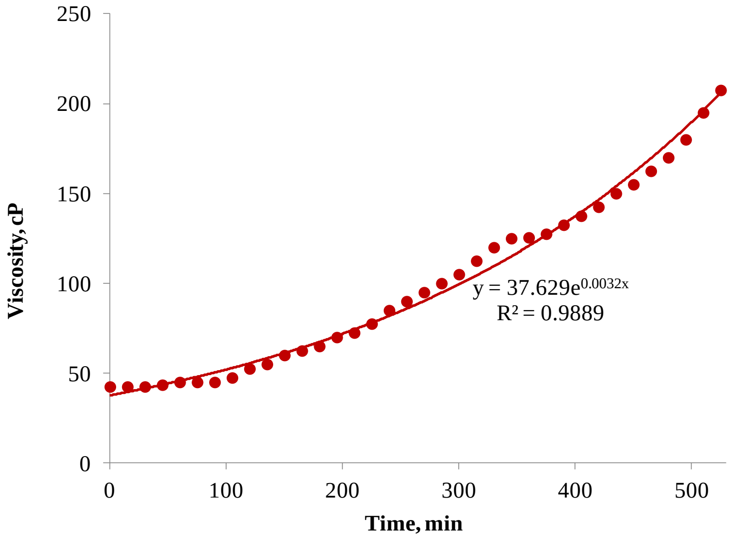

Figure 4. Changes in the viscosity of the bio-oil over time at 95 ºC

3.1.2 FTIR analysis of the binder materials

The binder materials were analyzed by FTIR and the comparison between spectra show the results of a preliminary approach to the chemical changes that occur during the bio-binder production, and a more in-depth study should be carried out in the future to confirm these results. However, the results are very useful to show chemical interactions that are occurring and contributing to change the materials' consistency and therefore the performance of the binder materials.

In Figure 5 are compared the spectra of the base materials (bio-oil, asphalt, cryoGTR), the bio-binder's (cryoMBO), and the bio-blend's (PG64-22&cryoMBO before and after RTFO aging. The interactions observed are very similar to the ones observed in the production and aging of the other bio-binders studied.

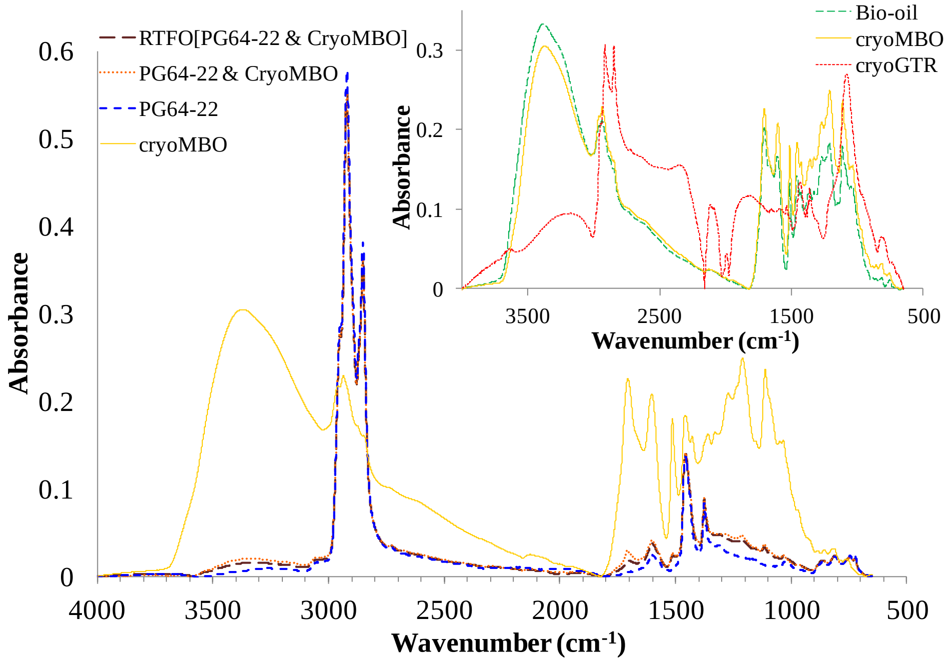

Figure 5. FTIR spectra of different binder constituents

The transferences of parts of rubber to the bio-oil and from the bio-oil to the rubber are visible from the analysis of the spectra presented in Figure 5. Between wavenumbers 800 and 1150 cm^{-1} some molecules decreased from the base bio-oil to the cryoMBO. This region of the spectra can be correlated to the aliphatic hydrocarbon region from the rubber, but it can also be related to primary aliphatic alcohols from the bio-oil. These are most likely the molecules that facilitate the swelling process in the rubber (Peralta *et al.*, 2012). These observations suggest that these components are compatible with the linear polymeric skeleton of the rubber, and this is consistent with the results previously reported (Gawel *et al.*, 2006), that mention a preferential absorption of the compounds with linear aliphatic chains into the rubber.

The bio-oil contains between 15 and 30 wt% of moisture (Bridgwater, 2012; Meier *et al.*, 2013; Yang *et al.*, 2014) that can be seen in the band around 3300 cm^{-1}, and this moisture content is smaller in the bio-binder (cryoMBO). The moisture can be present in the bio-oil in two different forms, dissolved or as emulsion, and some of this free moisture can evaporate during the production of the bio-binder (Oasmaa and Peacocke, 2010). However, some more moisture is added with the rubber, thus justifying the final higher content of moisture in the cryoMBO.

Although the spectrum of the binder blend (PG64-22&cryoMBO) is similar to that of asphalt, the integration of 20% of cryoMBO caused some changes. In the band at around 3300 cm^{-1} there is an increase of moisture from the asphalt to the binder blend that decreases after the RTFO aging of the material, which is in agreement with the mass loss of 3.6 wt% (Table 5). Around 1300 cm^{-1} there is a high amount of aliphatic compounds in the bio binder than in asphalt, which came from the bio-oil and rubber. These elements present very small changes with the RTFO process, indicating that they are stable in the final binder blend. Additionally, new peaks in the spectra of the bio-binder are due the incorporation of the cryoMBO in the asphalt.

3.1.3 Rheology of the binder materials

The rheology of the binder materials was assessed with a Dynamic Shear Rheometer (DSR), by carrying out frequency sweeps at different temperatures (ASTM D 7175-08, 2008). The results were then used to build the $|G^*|/\sin\delta$ master curves of the several materials (Figure 6), and to determine their high temperature continuous performance grade (Table 1) specified in the SuperPave methodology (Asphalt Institute, 2003).

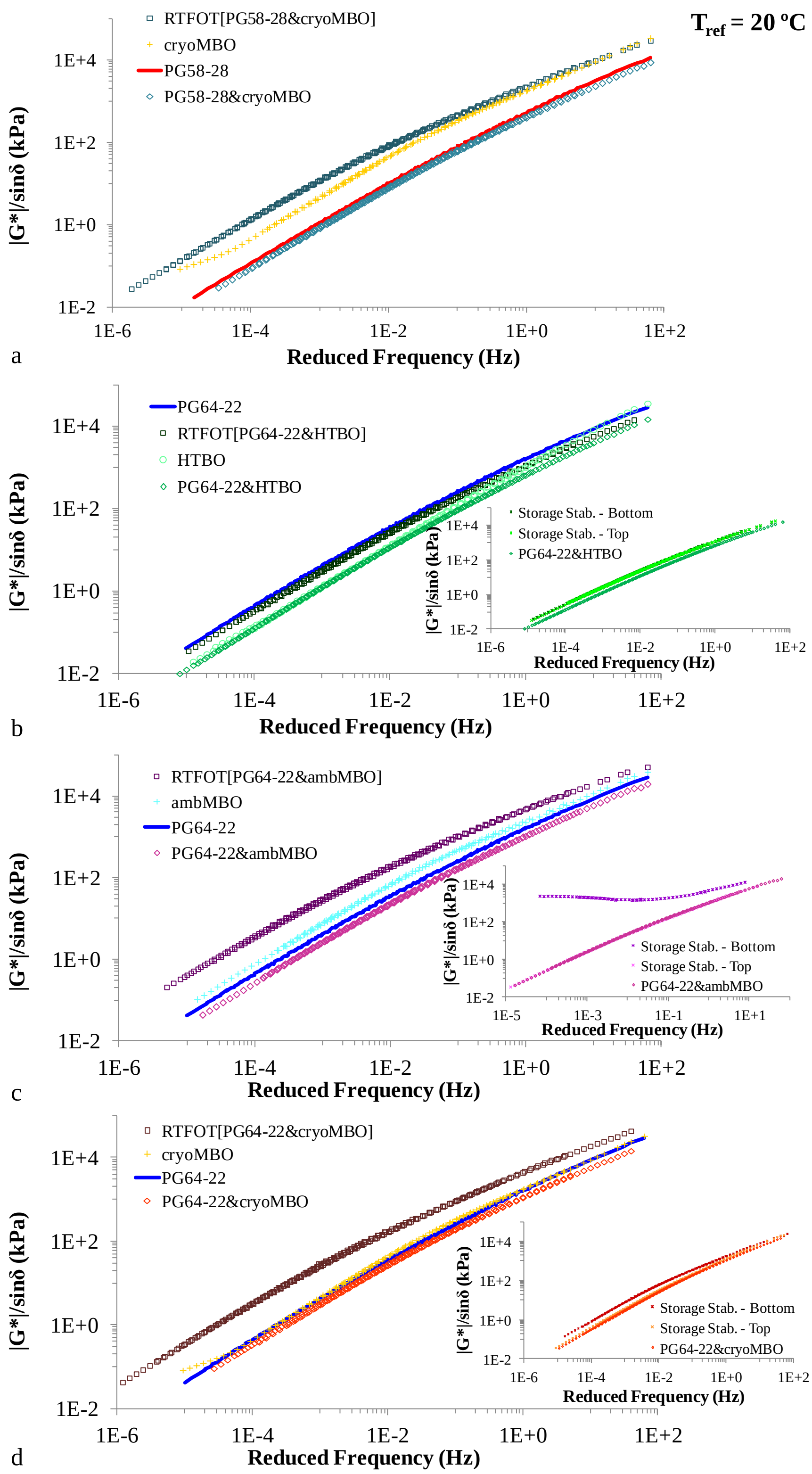

Figure 6. Master curves of the different binder materials (the legends are sorted by the order the curves appear at the reduced frequency around 0.01 Hz)

A preliminary approach to the master curve of the bio-oil shows a material more sensitive to temperature and frequency than conventional asphalt. This scenario completely changes when the cryoMBO master curve is analyzed, as it presents a shape and consistency very similar to that of asphalt in a large range of temperatures and frequencies.

The master curves of all materials present very similar shapes, and the master curves of the asphalts blended with 20% bio-oil based binders fit between the master curves of the conventional asphalts (PG58-28 and PG64 22). The exception was the binder with PG58-28 asphalt blended with 20% cryoMBO, with a master curve below that of the conventional asphalt PG58-28.

The master curves of the bio-binders (ambMBO and cryoMBO) are located above those of the other unaged binder materials. Nevertheless, it should be noticed that the higher content of rubber particles in the MBO (15 wt%) can interfere with the DSR parallel plates, possibly affecting the rheological measurement of the MBO binders more than with the final binder blends containing only 3 wt% rubber particles.

After production, it is common to store the binder for some time before mixing, and during this period some changes can occur in the binder rheology. By observing the inserts displaying the master-curves of the top and bottom materials that result from the separation tendency test of the binder blends, it is evident that there was a small amount of hardening. By computing the percent differences of the values of the temperatures at which $G^*/\sin\delta$ equals 1.0 kPa, of the just blended and stored material, a difference of 7% was found for the PG64-22&HTBO (insert of Figure 6b), being this difference of 6% for the PG64-22&cryoMBO (insert of Figure 6d). The visible phase separation that occurred in the PG64-22&ambGTR (insert of Figure 6c) indicates that this material cannot be stored, and so it needs to be used immediately after production. Such process will imply using specific equipment at the mixing plant to produce the blend, and/or the technology must evolve to solve this issue.

The binder ages during mixing and compaction in the pavement, and aging in asphalt is usually evaluated through the material hardening. Thus, the same calculations of the continuous PG for the RTFO aged and just blended or un aged binders (Table 1) showed a difference around 22.0% for the PG58 28&cryoMBO and the PG64-22&HTBO, and smaller differences of nearly 16.5% for the PG64-22&cryo/ambMBOs. The hardening during the RTFO treatment is mainly due to water and volatiles loss, rubber swelling and possible polymerization reactions.

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
PG58-28 & cryoMBO	59.3	66.4
PG64-22 & HTBO	60.1	67.4
PG64-22 & cryoMBO	65.5	70.7
PG64-22 & ambMBO	65.2	72.1

**The indicated continuous grade for the RTFO residues is not the same as the standardized in AASHTO M 320, because the RTFO aging was modified, namely concerning the aging temperature used*

The blending of conventional asphalts with 20% of bio-binders (ambMBO and cryoMBO) did not change the high temperature performance grade of the base asphalt. There are no clear differences in the performance grade when blending the asphalt PG64-22 with the ambMBO or cryoMBO, although the RTFO aging process caused minor differences in the performance grade of the blend PG64-22 with 20% cryoMBO (justifying the selection of this binder for production of mixtures).

In summary, these results indicate that the new blended binders are suitable for flexible pavement construction, with a few advantages justifying the selection of the blend PG64-22 with 20% cryoMBO to continue the study.

3.1.4 Viscosity of the binder materials

The viscosity of the binder materials was assessed by using a Brookfield viscometer according to ASTM D 4402-06 (2006) standard, and as specified in the SuperPave1 asphalt binder testing methodology (Asphalt Institute, 2003). The obtained results (Table 2) are indicative of the mixing and compaction temperatures for HMA production.

Table 2. Mixing and compaction temperatures for the blended asphalt binders

Materials	Mixing Temperature (°C)	Compaction Temperature (°C)
PG58-28 & cryoMBO	160	140
PG64-22 & HTBO	150	140
PG64-22 & cryoMBO	160	150
PG64-22 & ambMBO	170	*

* *It could not be measured*

The blends of asphalt with 20% cryoMBO and ambMBO presented mixing and compaction temperatures higher than expected due to the particle effect of the GTR. In fact, the mixing and compaction temperatures of these blends could be slightly lower, as confirmed during the production of mixtures later in this work. Apparently the mixtures should be produced at 150 ºC and compacted at 160 ºC. However, it is known that the viscosity of a fluid with particles in suspension is higher than the fluid viscosity (Lefebvre and Maury, 2005), which is the parameter that is required for the definition of the mixing and compaction temperatures, because the binder is that liquid part, the rubber particles are solely a different kind of aggregate (Peralta *et al.*, 2013). Thus it was found that for this volume fraction of particles the suitable viscosity of the liquid for mixing and compaction temperatures would be achieved at 150 and 140 ºC respectively.

3.1.5 Density of the binder materials

The density of the binders and their components is a very important characteristic of the materials, because it impacts the binder blends production and the volumetric formulation of the mixtures, as it is a volumetric design. The density of the binder materials was measured according to ASTM D70-09 standard (ASTM D 70-09, 2009)(Table 3).

Table 3. Density of the different bio-oil materials and asphalt binders

Materials	Density (g/dm^3)
Bio-oil	1218
HTBO	1281
cryoMBO	1246
ambMBO	1247
PG58-28 & cryoMBO	1063
PG64-22 & HTBO	1070
PG64-22 & cryoMBO	1066
PG64-22 & ambMBO	1067

The bio-oil has a high density when compared to asphalt, and the heat treatment increases the bio-oil density. Since rubber has a density around 1140 g/dm^3, when added to the bio-oil it slightly reduces the overall density of the cryoMBO or ambMBO. The final density of the blend between the asphalt and the HTBO or MBO materials reflect the lower density of asphalt (80%) and the different densities of the bio-oil based materials. The higher density of the binder blend (when compared to asphalt) implies that it is necessary to add an higher weight percentage of the binder blend (5.5 wt%) than what would be necessary if asphalt was used (5.0 wt%), in order to obtain the same volume of binder in the mixture.

3.1.6 Storage stability or separation susceptibility of the binder materials

The different densities of the bio-oil and asphalt materials justifies the evaluation of their separation susceptibility (ASTM D 7173-05, 2005) after being blended in three chosen binders. After vertical column storage at high temperatures, the rheology of the top and bottom fractions of the tested materials was assessed by using a DSR and frequency sweep tests at different temperatures. These results were used to assess their high temperature performance grade, which was then used to compute the differences between the performance grade of the top and bottom fractions of each material (Table 4).

Table 4. Difference between the top and bottom binders fractions in the separation test

Materials	High Temperature Continuous Performance Grade (°C)		Difference (%)
	Top	Bottom	
PG64-22 & HTBO	64.6	64.3	0.6
PG64-22 & cryoMBO	66.2	72.7	8.8
PG64-22 & ambMBO	66.3	*	*

* *The value was too high and could not be measured; thus, the resulting difference was higher than 10%*

The results show that the blends of PG64-22 asphalt with cryo and ambMBO were susceptible to separation. Nevertheless the blend of PG64-22 with cryoMBO was inside the specification criteria, but it *was* near the specification limit of 10%. The higher separation tendency of the blend of PG64-22 with ambMBO might be caused by ambGTR bigger particle size when compared with cryoGTR's smaller particle size. Finally, although the HTBO presented the highest density, its blend with PG64-22 asphalt seems to be a more compatible system. Thus, the GTR is the main contributor for binder phase separation.

3.1.7 Mass loss of the binder materials on the RTFO test

The mass loss of the several binders using two aging tests procedures (modified RTFO and oven aged) was measured and the values are presented in Table 5.

Table 5. Mass loss due to asphalt binder aging with different methods

Materials	RTFO (% mass loss)	Oven aged (% mass loss)
PG58-28 & cryoMBO	3.9	3.3
PG64-22 & HTBO	3.7	3.0
PG64-22 & cryoMBO	3.6	3.1
PG64-22 & ambMBO	3.3	3.1

The results show higher percent mass loss then what would be expected for a conventional asphalt binder. However, most of this mass loss is caused by water loss (Meier *et al.*, 2013). This is confirmed by the similarity between the modified RTFO and oven aged mass loss. Thus, the performance of these binders would not be as harmed by oxidative aging as it could be concluded from these results.

Summing up the global evaluation of the binders, the material with best overall results in the binder characterization tests was the blend of PG64-22 asphalt with 20% cryoMBO, as this was thw blend with the closest rheological behavior to the commercial asphalt PG64-22, lower mixing and compaction temperatures, and less tendency to separation than the PG64-22 & ambMBo blend. Therefore, this binder was selected to proceed with the production and characterization of asphalt mixtures in the remaining part of this work.

3.2 Results of the mixtures testing

Mixtures designed and prepared according to the Superpave mix design method (Asphalt Institute, 2001) were compacted using the Superpave Gyratory Compactor (SCG) and the roller compactor, in order 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.

3.2.1 Moisture susceptibility of asphalt mixtures

The tensile strength ratio (TSR) value, which 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 (Table 6). An adequate TSR result is fundamental to accept the new mixtures and to proceed with their evaluation, or else the mix design will fail and new binders or mixes should be prepared.

Table 6. Tensile Strength Ratio (TSR) of asphalt mixes with an optimum asphalt content of 5.5% and a mean air voids content of 6.7%

	Unconditioned Strength (kPa)	Conditioned Strength (kPa)	TSR
Average	1756.4	1461.7	0.84
Standard Deviation	187.1	53.5	0.06
Coefficient of Variation	0.11	0.04	0.07

A minimum TSR value of 0.70 is recommended (Lottman, 1982), but many agencies specify a TSR value of 0.80 (Asphalt Institute, 2001; Brown *et al.*, 2009; Roberts *et al.*, 1996). The tensile strength ratio of the studied mixture (0.84) is greater than 0.8 and, thus, it can be considered that the mixes meet the Iowa DOT specifications for this property.

Harvey (2010) reported the relationship between the vales of the IDT of a mixture and its expected fatigue life. According to the criteria mentioned by Harvey (2010) and based on the presented IDT values, the studied mixtures will meet the design criteria (between 3 and 30 million ESALs), either conditioned or not, without cracking due to fatigue. Additionally, derived from other criteria mentioned in the same work, it could be inferred that the studied mixes should not present rutting problems.

The following tests will provide additional and more accurate information about the performance of the studied mixtures related to the main pavement distresses that lead to premature failure, such as fatigue cracking, rutting and low temperature cracking, in order to validate the use of these new mixtures in pavements.

3.2.2 Fatigue cracking resistance of asphalt mixtures

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 Figure 7, 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.

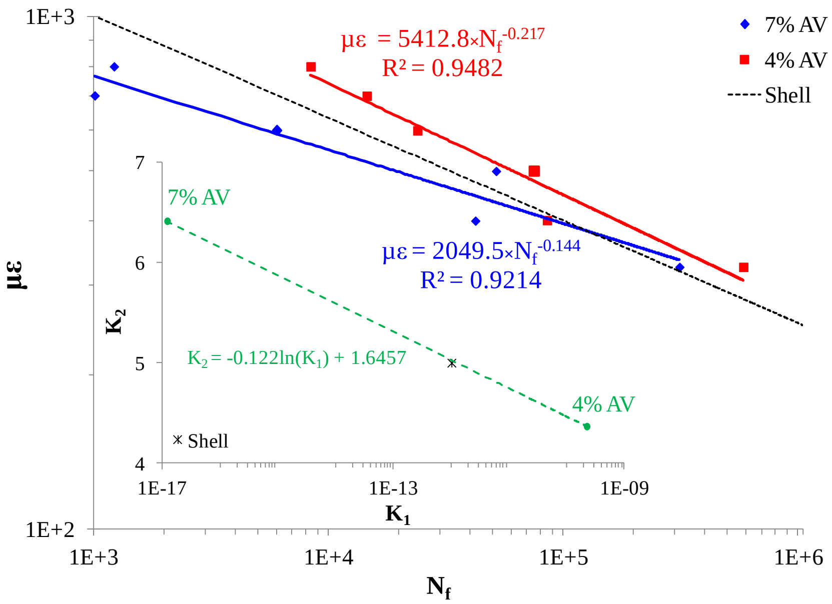

Figure 7. Variation of the fatigue life (N_f) with the tensile microstrain (με) applied in mixtures with 5.5% asphalt content compacted to 4% and 7% air voids (AV)

The results of Figure 7 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. This happens because the reduction of air voids content corresponds to an increase of the asphalt and aggregates, resulting in a reduction of tensile stress on these samples, since air does not transfer stress. Moreover, smaller air voids content creates a more homogeneous structure with less stress concentration in critical solid-air interfaces in which the microcracks that begin to develop, under repetitive loading, grow slower and take longer to interconnect because of the reduced number and smaller size of air voids (Harvey *et al.*, 1995).

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. It is expected that these mixtures exceed the design number of ESALs (30 million) for a tensile strain level of 129 με and 172 με, respectively for the mixtures with 4% and 7% air voids. Moreover, considering that each ESAL would induce a tensile strain level of 100 με when the studied mixtures are placed in the pavement, they will not show fatigue cracking distress before 65 million ESALs for the mixture with 4% air voids, and before 540 million ESALs for the mixture with 7% air voids.

Shell (1978) suggested a model to predict the fatigue life of a pavement, which related the tensile strain in the bottom of the asphalt concrete layers with the number of load cycles or ESALs. This model [1] was used in this work to compare the fatigue behavior of conventional mixtures (represented in a simple way by this model) with that of the new mixes produced in this work. This model is shown in Figure 7 for a conventional asphalt mix with 4000 MPa and an asphalt volume content of 12%.

$$\varepsilon_t = (0.856 \times Va + 1.08) \times \left|E^*\right|^{0.36} \times N_f^{-0.2} \qquad \text{(Eq. 1)}$$

where:
ε_t – tensile strain;
N_f – number of loading cycles (ESALs);

V_a – volume percentage of asphalt in the total volume of the mixture; and
$|E^*|$ – deformability or dynamic modulus of the asphalt mixture (Pa).

The Shell model, which is representing a conventional mixture, is able to support 98 million ESALs for a tensile strain of 100 με, and it can support the design number of 30 million ESALs for a tensile strain level of 126.7 με. These values show that the new mixtures developed in this work have an adequate fatigue cracking behavior.

The experimental fatigue coefficients K1 and K2 were determined to be respectively 2.2E-10 and 4.3675 for the mixture with 4% air voids. The mixture with 7% air voids had fatigue coefficients of K1=1.2E-17 and K2=6.4148. Thus, both mixtures show a fatigue performance similar to that of conventional asphalt mixtures, as reported in previous works:

- $2.907 < K_2 < 6.781$ and $2.497E-17 < K_1 < 1.398E-7$ by Cascione *et al.* (2011);
- $3.37 < K_2 < 6.43$ and $3.98E-15 < K_1 < 1.25E-7$ by Ghuzlan and Carpenter (2003); and
- $3.31 < K_2 < 6.45$ and $3.04E-12 < K_1 < 3.81E-7$ by Pais *et al.* (2009).

The K_1 coefficient characterizes the flexural modulus, and the K_2 coefficient indicates the rate of damage accumulation in a sample. When using this relationship as failure criterion for pavement design, a lower K_2 value is more conservative as it assumes faster accumulation of fatigue damage. The values suggested for K_2 are 4.477 by The Asphalt Institute, 4.0 by Shell, and 3.571 by the University of Nottingham (Huang, 2004). Carpenter (2006) recommended the Illinois DOT to use a K_2 value in the range of 3.5 to 4.5 (Williams *et al.*, 2011), as being that obtained in the mix with 4% air voids.

The experimental K_1 *vs.* K_2 values were plotted (insert of Figure 7) defining a line were the fatigue coefficients of the Shell model fit, which corroborates the suitability of the mixtures in terms of fatigue life..

Subsequently, Figure 8 shows the cumulative dissipated energy (computed according to AASHTO T 321-07 (2007) standard in the fatigue test in function of the number of load cycles for each studied mixture. With higher air voids content, the mixture with 7% air voids has a lower area under its curve, which shows a lower cumulative dissipated energy than the mixture with 4% air voids (in the range of the tensile strain values used in the fatigue test). These results are in line with those currently obtained with traditional asphalt mixtures. The area below the stress/strain curve indicates the toughness of the asphalt mixtures with 7% and 4% air voids, which were 2.33 MPa and 2.14 MPa respectively.

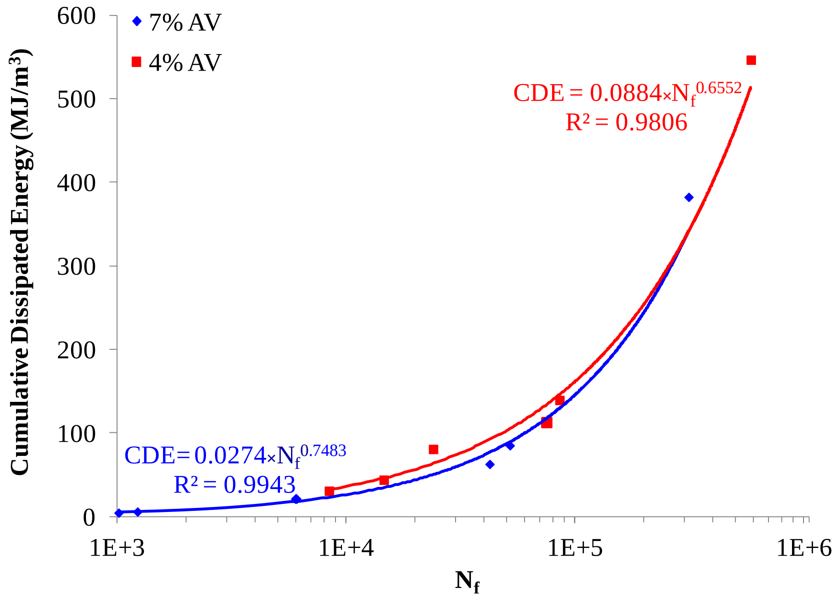

Figure 8. Variation of Cumulative Dissipated Energy (CDE) with the number of load cycles (N_f) for mixes with 5.5% asphalt content compacted to 4% and 7% air voids (AV)

The previous results obtained in the fatigue cracking test indicate that a pavement build with this mixture (with a range of 4% to 7% air voids content) should not present fatigue distresses within the design level of 30 million ESALs.

3.2.3 Dynamic modulus of asphalt mixtures

Figure 9 shows the master curves of the dynamic modulus for the mixtures designed with 4% and 7% air voids, which were built by shifting the frequency sweeps (six frequencies) at three testing temperatures (4, 21 and 37 ºC) using the time-temperature superposition principle and the sigmoidal or Witczak prediction model (Garcia and Thompson, 2007). The experimental data of the dynamic modulus at each temperature was shifted using a reference temperature of 21 ºC. The experimental master curve was then used to fit the prediction model.

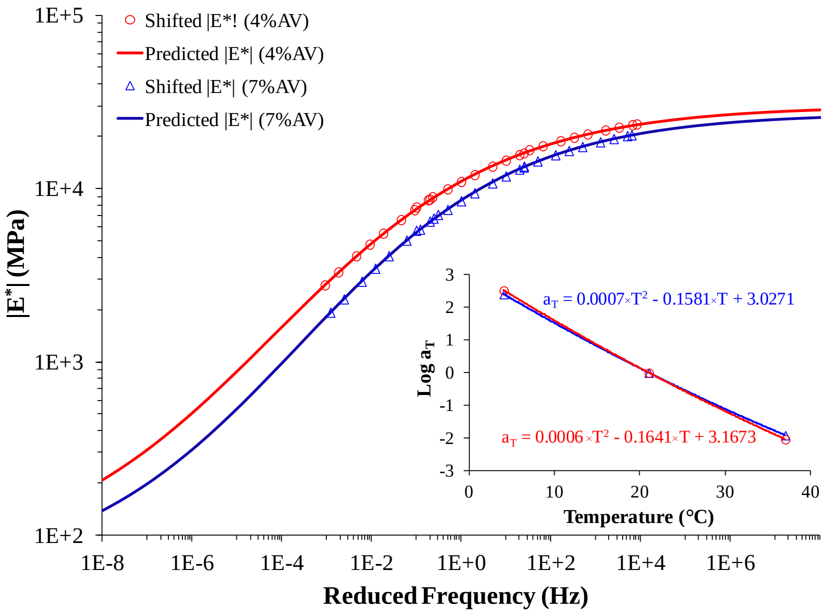

Figure 9. Master curve of the shifted and predicted dynamic modulus for the 4% AV and 7% AV samples (T_{ref} = 21ºC), with an insert with the quadratic shifting

The fitted prediction model shows that the asphalt mixture with 4% air voids is stiffer than that with 7% air voids, as it was expected due to the higher compactness of the mixture. Thus, it can be concluded that the mixture with 4% air voids should perform better at high temperatures, having a high rutting resistance (as a consequence of the high stiffness modulus at high temperatures). 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 (West *et al.*, 2013; Williams *et al.*, 2011). The rheological behavior of the mixtures at both levels of compaction (4 and 7% AV) converges at higher frequencies (or lower temperatures) as it approaches the glassy plateau, and diverges with the temperature increase (or reduction in frequency).

These results show that these new mixtures are very stiff materials after compaction, either for 4% or 7% AV contents, which are not prone to develop rutting. This result seems to be out-of-phase with the rheological evaluation of the binders, which indicated that the new binder blend is slightly softer than the conventional asphalt.

3.2.4 Rutting resistance of asphalt mixtures

The triaxial repeated load permanent deformation test can be used to evaluate the rutting resistance of HMA specimens, with the permanent deformation of the specimen recorded as a function of the number of load cycles. Specimen loading is carried out for 0.1 sec followed by a 0.9 sec dwell (or unloading). There are three phases of flow that occur during this type of test: primary, secondary, and tertiary. Under primary flow, there is a decrease in the strain rate over the initial loading phase. With continuous repeated load application, the next phase of material response is referred to as secondary flow, which is characterized by a relatively constant strain rate. Finally, the material enters tertiary flow, where the strain rate begins to increase as the test progresses. Tertiary flow means that a specimen is beginning to deform significantly and individual aggregates composing the skeleton of the mix are moving past each other. The flow number is based upon the initiation of tertiary flow, or the minimum strain rate recorded during the course of the test (Kvasnak *et al.*, 2007).

The flow number of an asphalt mixture corresponds to the number of cycles needed to accumulate 5.5 percent strain in the sample tested. A higher flow number indicates a higher resistance to rutting. The test ends at 10,000 load cycles even if the sample has not accumulated 5.5 percent strain. A sample that reaches 10,000 load cycles is considered to be essentially rut resistant at the testing temperature. Since all the samples tested reached 10,000 load cycles at 37 ºC, the test was repeated at 54 ºC. The response measured in this test was the accumulated strain and is presented in Figure 10.

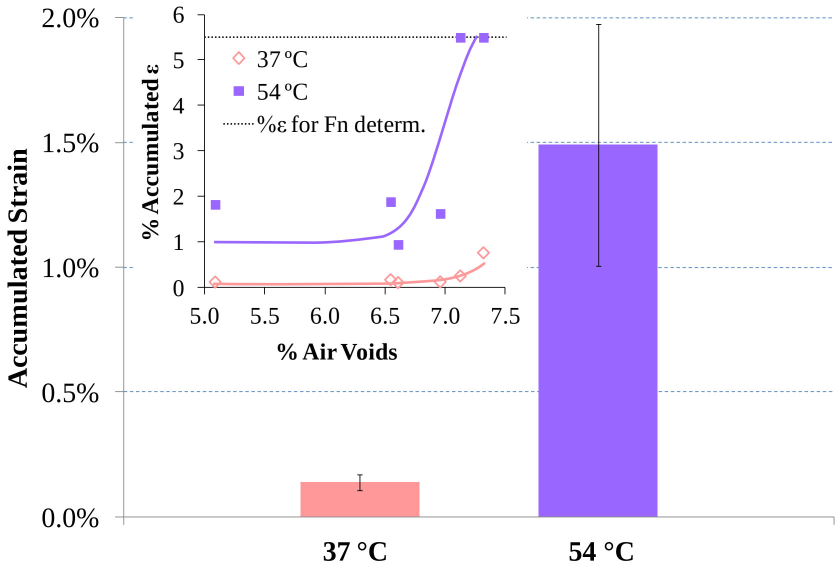

Figure 10. Accumulated strain at 10,000 cycles for the 7% air voids mixture

The presented values are the average strain levels of three samples in each batch and at each temperature. A lower accumulated strain level at the end of the test was interpreted as a mixture with higher resistance to rutting, which was necessarily obtained for tests carried out at 37 ºC. The samples with air voids contents lower than 6.5 presented similar values of deformation at the end of the test, both for tests at 37 or 54 ºC. However, the accumulated deformation at the end of the test started to increase for samples with higher air voids content, especially for samples tested at 54 ºC.

Primarily, it was observed that the accumulated strain of the mixtures developed in this work is exceptionally low at both test temperatures in comparison with the traditional mixtures. Thus, the rutting performance of these new mixtures in the field is expected to be very good even at high temperatures.

3.2.5 Low temperature fracture mechanics of asphalt mixtures

The previous results showed that the mixes produced with the blend of PG64-22 asphalt with 20% cryoMBO are quite stiff. Thus, although the fatigue life is sufficient for the designed level of ESALs and the compacted mixes were not susceptible to moisture damage, it could be expected that the performance at low temperatures may not be adequate. Therefore, the semi-circular bending test (SCB) was performed in order to evaluate the low temperature fracture mechanics of the studied mixtures. The results obtained with that test for several parameters are plotted in Figure 11.

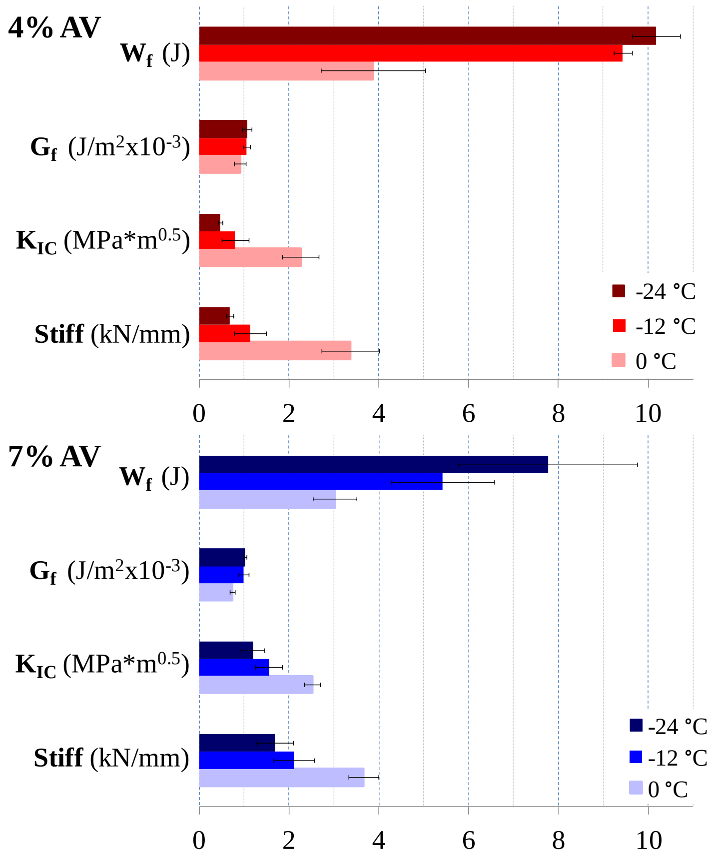

Figure 11. Low temperature fracture mechanics of the studied mixtures in the SCB test

The results obtained for the newly developed mixes were compared with other results from the literature (Li and Marasteanu, 2010; Velasquez *et al.*, 2009; West *et al.*, 2013), 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, contrarily to what was expected, that the designed mixes have a good resistance to fracture cracking at low temperatures, despite having presented a very high stiffness in the dynamic modulus test.

4. Conclusions

In this study the bio-oil was heat treated, and further modified with cryogenic and ambient ground tire rubber. These three bio-oil binders were then blended with a PG58-28 and a PG64-22 asphalt. The bio-oil based materials (bio-oil, HTBO, cryoMBO, ambMBO), the asphalts (PG58-28 and PG64-22), and the final blends were then characterized. The tests performed included RTFO aging, FTIR, viscosity, rheology, density and separation susceptibility. The results obtained in the characterization of these binders showed that the final blends have properties similar to those of conventional asphalts, except for the blend of the PG64-22 asphalt with ambMBO (which showed a higher susceptibility to separation during storage).

After the binder study, the blend of the PG64-22 asphalt with 20% cryoMBO was selected to produce asphalt mixes with a 9.5 mm NMAS, as being the binder with best global performance in the previous phase of the work. Afterwards, flexural fatigue cracking, moisture susceptibility, dynamic modulus, rutting resistance and low temperature facture tests were performed to evaluate the global performance of the mixtures developed in this work. The mixtures with the chosen asphalt blend showed very good performance in all the tests carried out, and thus it is not expected that rutting or early fatigue cracking distresses can occur, 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 for pavement works, which can present technological, environmental and economic advantages.

Acknowledgments

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