



High-temperature, low-temperature and weathering aging performance of lignin modified asphalt binders



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ABSTRACT

The objective of this work is to analyze the physical and chemical properties of lignin modified asphalt binders aiming to apply lignin obtained as a subproduct of the pulp and paper industry in asphaltic pavements. Lignin increased the Brookfield viscosity of binders mainly for 135 °C. Lignin modified asphalt binders registered a lower carbonyl index and thus a higher weathering aging resistance than the conventional binder, except for the binder with 4 wt.% of lignin after 200 h of weathering test. A content of 4 wt.% of lignin increased thermal stability of binder after the Rolling Thin Film Oven Test (RTFOT). Dynamic Shear Rheometer analysis showed that as the lignin content in binder increases, the stiffening increases as well and the corresponding high-temperature performance grade for unaged and RTFOT aged samples also improves. Lignin significantly improves the asphalt's resistance to thermal cracking at temperatures up to –12 °C.

1. Introduction

Asphalt binder, commonly used in flexible pavements in Brazil, is subject to the action of photodegradation mainly in geographic regions where the incidence of solar radiation is more intense (Araujo et al., 2013a). In these geographic regions, high room temperatures are commonly associated with the ultraviolet radiation causing a severe aging of asphalt binders (Xiao et al., 2013; Wang et al., 2012; Petersen, 2009).

During the asphalt aging, the concentration of carbonyl and sulfide groups increase in the asphalt binder, as well as polar functional groups (Lins et al., 2008). The carbonyl groups in the asphalt binder include carboxylic acid, aldehyde, amide, anhydride, ester, and ketone (Petersen et al., 1975). During the oxidation of asphalt, the polar and aromatic groups in binder agglomerate, and mobility and reactivity of asphalt binder are reduced (Campbell and Wright, 1964; Lu and Isacson, 2002; Fernández-Gómez et al., 2013).

Literature usually reports asphalt aging studies that use the Rolling Thin Film Oven Test (RTFOT) to simulate the short-term aging and the Pressure Aging Vessel (PAV) to simulate the long-term aging process (Abbas et al., 2002; Lu and Isacson, 1998; Polacco et al., 2015).

However, research developed considering the weathering aging of asphalt as a long-term process is scarcer (Lins et al., 2008).

In recent years, modified asphalt materials have been widely studied and applied to improve pavement performance on anti-aging properties (Lu and Isacson, 2000; Xiao et al., 2009; Yao et al., 2012; Yao et al., 2013; Cortizo et al., 2004; Ahmedzade and Yilmaz, 2008). Polymers have been considered the most cost effective additives to enhance the durability of asphalt pavements (Polacco et al., 2015; Wang et al., 2015; Li et al., 2015; Al-Mansob et al., 2016). Regarding the prevention of bitumen aging, some methods concerning the thermal or photo-oxidative aging have been investigated (Ouyang et al., 2006; Apeageyi, 2011; Feng et al., 2011, 2012a, 2012b; Yu et al., 2009; Yamaguchi et al., 2005). Various modifiers, such as antioxidants (Ouyang et al., 2006; Apeageyi, 2011; Feng et al., 2011), UV absorbers (Feng et al., 2011; Feng et al., 2012a), organo-montmorillonite (Yu et al., 2009), carbon black (Yamaguchi et al., 2005) and other phosphorus compounds (Filippis et al., 1995) can improve the resistance of bitumen to thermal or photo-oxidative aging.

Currently, it is sought to find applications in the market for lignin (Gómez-Fernández et al., 2017; Qian et al., 2017; Kühnel et al., 2017; Li et al., 2017; Luo et al., 2017). Gellerstedt (2015) stressed the relevance

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of development of material systems based on softwood Kraft lignin for the future sustainable society and add value to a renewable resource. Lignin accounts for 15–25 wt.% of herbaceous biomass. Reports about the transformation of lignin into profitable products are scarce (Buranov and Mazza, 2008). Nevertheless, recently Xu et al. (2017) evaluated the potential use of wood lignin as partial substitute and performance modifier in asphalt binder. The utilization of Kraft lignin as a cationic emulsifier in the preparation of bitumen emulsions has been assessed in the work published by Yuliestyan et al. (2016). It has been estimated that more than 25 million tons of kraft lignin are produced from alkaline pulping of wood chips annually in the U.S (Stewart, 2008). The conversion of biomass (lignocellulosic material) into biofuel produces large amounts of solids containing lignin and lignin derivatives. Lignin is considered a waste material, and its use is predominantly limited to fuel (Stewart, 2008). Therefore, new applications for lignin as antioxidant would contribute to the growth of biofuel and paper industries and to the reduction of industrial waste.

The oxidation reaction typically follows three main steps: initiation, in which free radicals are produced; propagation, and finally termination, when two free radicals interact with each other to end the reaction. When acting as an antioxidant, lignin-derived units inhibit the oxidation propagation reaction through hydrogen donation, which occurs primarily due to the presence of phenolic hydroxyl groups (Pan et al., 2006). Lignin compounds that contain more phenolic hydroxyl groups and fewer aliphatic hydroxyl groups have low molecular weight and high antioxidant property (Wang and Derewecki, 2013).

The objective of this work is to evaluate the lignin modified asphalt (LMA) binders considering physical properties, thermal stability, rheology and the weathering aging resistance to make the application of lignin from pulp and paper mill in asphaltic pavements feasible.

2. Material and methods

The asphalt binder, CAP 50/70, was provided by a Brazilian oil and gas company. A commercial Kraft lignin (k-lignin) was supplied by a Brazilian pulp and paper mill. The physical properties of bitumen are shown in Table 1.

Table 2 shows the molecular weight, ash content, pH, and moisture of the lignin provided by a Brazilian paper and pulp mill.

The modified asphalt with anti-aging additive was prepared via melt blending using mechanical stirrer. At the start of the process, the bitumen was heated until it was absolutely flowing. Then, pre-determined percentages (1 wt.%, 4 wt.% and 6 wt.%) of lignin were added to each sample of bitumen and blended in an aluminum container at a fixed mixing speed of 5000 rpm. The modified bitumen was finally obtained after the mixtures were blended at 160 °C for 60 min.

The Brookfield viscosity measurement was performed at temperatures of 135 °C, 155 °C, and 177 °C according to the ASTM D 4402/02 Standard. For the penetration test, a

Table 1
Physical properties of conventional binder 50/70.

Property	Result	Unit
Penetration 25 °C, ASTM D 5 Standard	41.1	0.1 mm
Softening Point, ASTM D 36 Standard	50.5	°C
Thermal susceptibility index, X 018 standard	−1.1	
Solubility in trichloroethylene, ASTM D 2042 Standard	99.5	wt.%
Flash point, ASTM D 92 Standard	328	°C
Ductility, 25 °C, ASTM D 113 Standard	> 150	cm
RTFOT Mass variation, ASTM D2872 Standard	−0.182	%
RTFOT Ductility at 25 °C, ASTM D113 Standard	> 150	cm
RTFOT – Increase of softening point, ASTM D36	3.9	°C
RTFOT-Penetration retained, ASTM D5 Standard	57	%
Viscosity Brookfield 135 °C, ASTM D4402	337.5	cP
Viscosity Brookfield 155 °C, ASTM D4402	172	cP
Viscosity Brookfield 177 °C, ASTM D4402	63.7	cP

Table 2
Chemical characterization of Kraft lignin.

Parameters	Result	Unit
Number Average Molecular Weight Mn	2260 ± 32	
Weight Average Molecular Weight Mw	3378 ± 79	
Mw/Mn	1.49469	
Ash content at 800 °C, 12 h	3.3	%
pH	3.5	
Moisture content	3.8	%

100 g needle penetrated into a binder sample for 5 s at 25 °C, according to the ASTM D 5 Standard. The softening point was evaluated according to the ASTM D36 Standard.

Thermal analysis was performed using a SHIMADZU – DTG60 equipment that enables simultaneous recording of TG/DTG signals. The samples (mass of 30 mg) were heated in a standard aluminum oxide crucible. All experiments were carried out in a nitrogen and oxygen atmosphere with a flow rate of 100 mL min^{−1}, and a heating rate of 10 °C min^{−1} up until 700 °C.

The asphalt binder was submitted to the Rolling Thin Film Oven Test (RTFOT), according to the ASTM D 2872 Standard. The rheology of binders was analyzed by using a Dynamic Shear Rheometer (DSR), according to the AASHTO T315 Standard. The Standard Specification for Performance-Graded Asphalt Binder Using Multiple Stress Creep Recovery (MSCR) was used, according to the AASHTO M332 Standard. The Pressure Aging Vessel (PAV) was performed according to the ASTM D 6521 Standard. Subsequently, the sample degradation was evaluated using the dynamic shear rheometer (DSR), according to the AASHTO T315 Standard and the test bending beam rheometer (BBR), according to the AASHTO T313 Standard.

After the RFOT, the samples were also exposed in the weathering chamber with alternating cycles of xenon radiation and moisture at 60 °C. Films of 0.6 mm thickness of RTFOT-aged conventional binder and modified binders with 1 wt.%, 4 wt.% and 6 wt.% of lignin were deposited on granite plates of 150 mm in length and 100 mm. This procedure aims to simulate the thickness of the asphalt binder coating on the stone in the hot mix asphalt (HMA). Four granite plates coated with asphalt binders were exposed in a Q-Sun Xe 1 weathering chamber, according to the ASTM D-4798 (2008) Standard. The samples were exposed for 10, 20, 30, 40, 50, 100, 150, 200 and 250 h to simulate a long-term aging process. Cycles of 80 min were performed: 64 min of light, 16 min of light and water, the specimen temperature of 60 °C and irradiation at 340 nm = 0.35 W/m². Variable power from 3500 to 6500 W maintained the intensity of irradiation.

The aged samples were analyzed by using Fourier Transform Infrared Spectroscopy (FTIR) technique in the Bruker apparatus, Alpha model, by the ATR (Attenuated Total Reflectance) method under a nitrogen atmosphere. The spectrum was obtained in units of absorbance between 2000 and 950 cm^{−1} with 32 accumulations and a resolution of 4 cm^{−1}. The ORIGIN software was used to evaluate the areas of the peaks centered at 1375 cm^{−1} and 1465 cm^{−1}, corresponding to the δCH₃ and δCH₂ respectively, where δ is the bending vibration. The peak area centered at 1700 cm^{−1}, corresponding to the C=O peak (carbonyl), was also measured. The Carbonyl Index (CI) was calculated by using equation 1.

$$CI = (\text{Peak area of C=O centered at } 1700 \text{ cm}^{-1}) / (\text{Peak area of CH}_2 + \text{CH}_3 \text{ at } 1375 \text{ and } 1465 \text{ cm}^{-1}) \quad (1)$$

A flowchart (Fig. 1) synthesizes the experimental procedure.

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