Characterization of 30/45 asphalt binder modified with kraft lignin in relation to short-term aging

Caracterização do ligante asfáltico 30/45 modificado com lignina kraft frente ao envelhecimento a curto prazo

Caracterización del ligante asfáltico 30/45 modificado con lignina kraft frente al envejecimiento a corto plazo

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ABSTRACT
In this study, the addition of lignin granules to the asphalt binder PAC 30/45 and its effects on short-term aging were investigated. Penetration, softening point, and ductility tests were conducted before and after RTFOT (Rolling Thin Film Oven Test). Laboratory results revealed that incorporating lignin into the asphalt binder significantly improved aging resistance compared to the pure binder. This was evidenced by reduced penetration, increased consistency, higher softening point, and satisfactory ductility after aging. Lignin also decreased the binder's sensitivity to temperature variation, indicated by a Thermal Susceptibility Index (TSI) closer to zero. While improvements were not as pronounced as in previous studies with PAC 50/70, adding 3% lignin showed benefits compared to the unmodified binder, whereas 6% lignin made PAC 30/45 more susceptible to oxidation. It is concluded that lignin can enhance the rheological properties of PAC 30/45, increasing its stiffness, durability, and aging resistance. Further exploration of lignin concentrations below 3% and above 6% is recommended to better understand oxidative effects on the 30/45 binder and confirm the benefits of minor additions.

Keywords: lignin, antioxidant, asphalt concrete, PAC 30/45, polymers, road engineering.

RESUMO
Neste estudo, investigou-se a adição de grânulos de lignina ao ligante asfáltico CAP 30/45 e seus efeitos no envelhecimento a curto prazo. Foram realizados ensaios de penetração, ponto de amolecimento e ductilidade, antes e após o RTFOT (Rolling Thin Film Oven Test). Os resultados laboratoriais revelaram que a inclusão de lignina no ligante asfáltico melhorou significativamente a resistência ao envelhecimento em comparação ao ligante puro, evidenciado pela redução na penetração, aumento da consistência e incremento no ponto de amolecimento, com ductilidade satisfatória. A lignina também reduziu a sensibilidade do ligante à variação de temperatura, conforme indicado pelo Índice de Suscetibilidade ao Trabalho (IST) mais próximo de zero. Embora as melhorias não tenham sido tão significativas quanto em estudos anteriores com CAP 50/70, a adição de 3% de lignina mostrou benefícios comparados ao ligante não modificado, enquanto 6% de lignina tornou o CAP 30/45 mais suscetível à oxidação. Conclui-se que a lignina pode aprimorar as propriedades reológicas do CAP 30/45, aumentando sua rigidez, durabilidade e resistência ao envelhecimento. Recomenda-se explorar outras concentrações de lignina, tanto abaixo de 3% quanto acima de 6%, para entender melhor os efeitos oxidativos no ligante 30/45 e confirmar os benefícios de pequenas adições.
Resumen
En este estudio, se investigó la adición de gránulos de lignina al ligante asfáltico CAP 30/45 y sus efectos sobre el envejecimiento a corto plazo. Se realizaron ensayos de penetración, punto de ablandamiento y ductilidad antes y después del RTFOT (Rolling Thin Film Oven Test). Los resultados de laboratorio revelaron que la incorporación de lignina en el ligante asfáltico mejoró significativamente la resistencia al envejecimiento en comparación con el ligante puro. Esto se evidenció por la reducción en la penetración, aumento en la consistencia, aumento en el punto de ablandamiento y ductilidad satisfactoria después del envejecimiento. La lignina también disminuyó la sensibilidad del ligante a la variación de temperatura, indicado por un Índice de Susceptibilidad al Trabajo (IST) más cercano a cero. Aunque las mejoras no fueron tan pronunciadas como en estudios anteriores con CAP 50/70, la adición del 3% de lignina mostró beneficios en comparación con el ligante no modificado, mientras que el 6% de lignina hizo que el CAP 30/45 fuera más susceptible a la oxidación. Se concluye que la lignina puede mejorar las propiedades reológicas del CAP 30/45, aumentando su rigidez, durabilidad y resistencia al envejecimiento. Se recomienda explorar concentraciones de lignina por debajo del 3% y por encima del 6% para entender mejor los efectos oxidativos en el ligante 30/45 y confirmar los beneficios de las adiciones menores.

Palabras clave: lignina, antioxidante, hormigón asfáltico, CAP 30/45, polímeros, ingeniería vial.

1 Introduction

Brazilian highways are the main means of transporting cargo and people in the country (Santos et al., 2024; Vilarinho et al., 2024). Road transport is the most widely used alternative in Brazil and accounts for approximately 65% of all Brazilian transportation infrastructure (Moretto; Júnior, 2024). This predominance reflects the extent of Brazil's road network: every year, thousands of kilometers of roads are paved to facilitate the transport of goods and passengers throughout the country (Isler et al., 2024; Wolff; Caldas, 2018). Thus, Brazilian land transportation has been heavily dependent on its road network, which in 2017 reached a length of 331,807 km including federal, state and municipal highways (Wang et al., 2024).

Although highways are the dominant mode in Brazil's transportation matrix, they face considerable challenges in terms of infrastructure quality, including problems such as inadequate paving, the presence of potholes and structural issues. A study carried out by the National Transport Confederation (CNT) in 2023 found serious deficiencies in the state of repair of highways. Of the 111,502 kilometers analyzed, only 32.5% are in excellent (7.9%) or good
Regular road maintenance is necessary to keep the road network in a satisfactory condition. The durability of paved roads is influenced by various factors, which can be categorized as external and internal. External factors that affect its performance include traffic load and climatic factors such as sunlight, heat and humidity. Internal factors relate to the composition of the sidewalk itself, such as rock aggregates (gravel or crushed stone), binder (asphalt binder) and voids. In addition, road conditions are linked to the age of the sidewalk, its degradation due to the high volume of traffic and the quality of the materials. Surface deformation, cracks and potholes are the biggest causes of accidents, due to the reduction in friction between the vehicle's wheels and the sidewalk, causing the vehicle to accelerate unexpectedly (Kamal; Bas, 2021).

To reduce these problems, additives and modifiers are being developed, both synthetic and of biological origin, with the aim of improving or restoring the properties of bitumen, such as adhesion, elasticity, fluidity, oxidation features and skid resistance. These additives can include fibers, powders, waxes, emulsifiers and recycling agents. Recycling agents are used in particular to recover the chemical, physical and rheological properties of aged asphalt binders (Ncirí; Kim, 2022; da Silveira et al., 2024).

Various polymers such as polyethylene (PE), ethy-ene vinyl acetate (EVA), ethylene butyl acrylate (EBA), and thermoplastic elastomers such as styrene–butadiene–styrene (SBS) and styrene–ethylene–butylene–styrene (SEBS) have been adopted as modifiers to improve the physical and rheological properties of the asphalt binder. Bitumen or asphalt binder with an appropriate polymer improves wear resistance at high temperatures without becoming excessively viscous for mixing or extremely rigid at room temperature, but this polymer must be compatible to avoid phase separation (Viscione et al., 2021).

One polymer with interesting properties is lignin. After cellulose, lignin is the second most abundant natural polymer in the world. It is present in the cell walls of plants and trees and is a source of renewable resources. An important aspect is that lignin has the ability to act as an antioxidant, due to the presence of phenolic structures that can neutralize free oxygen radicals. Free radicals break down molecules, destroying their chemical structure. The phenolic group can neutralize free radicals by donating a proton or an electron (Ben-Iwo; Manovic; Longhurst, 2016;
In addition, lignin is a widely available and economical organic and polyaromatic biopolymer, obtained as a by-product of waste from the wood industry. It has been explored as a total or partial substitute in bio-based and environmentally friendly binding materials for paving. It is also used in the development of high-performance bitumen with durability characteristics. Its use is advantageous for reducing CO₂ emissions, as it is a carbon-neutral biomaterial (Negi; Singh, 2023; Patel et al., 2023). Some other uses are as a surface treatment agent for natural fiber composites with petroleum-based resins (Kumar et al., 2021; Negi; Singh, 2023; Thielemans et al., 2002) and biosorbent for the removal of toxic metal ions from wastewater (Mohan; Pittman; Steele, 2006).

A widely used biopolymer is alkali lignin, a complex, three-dimensional polymer, also known as kraft lignin, which has undergone hydrolytic degradation. It is one of the main components of lignocellulosic materials and is considered an impurity in the separation of cellulose from wood (Braun; Holtman; Kadla, 2005; Jiang; Nowakowski; Bridgwater, 2010; Xu et al., 2020).

With a view to improving asphalt binders and the recurring damage to asphalt paving, this work prioritized investigating, by means of penetration, softening point, ductility and RTFOT aging tests, the effects and potential use of lignin added to 30/45 asphalt binder under normal and aged conditions.

2 MATERIALS AND METHODS

2.1 RAW MATERIALS

The lignin used in this study is of the alkali type, purchased from Sigma-Aldrich (catalog number 8068-05-1). The binder used is characterized as Petroleum asphalt cement (PAC) 30/45, which comes from the Refinaria Gabriel Passos (REGAP), belonging to Petrobrás S.A., located in the city of Betim, state of Minas Gerais, Brazil.

2.2 SYNTHESIS OF ASPHALT BINDER WITH LIGNIN

The PAC 30/45 samples modified with lignin were mixed manually with a glass rod and
homogenized at a temperature of 155 °C for 30 minutes. Lignin was added to PAC 30/45 at levels of 3% and 6% by weight. Conventional binder characterization tests were carried out before and after short-term aging to characterize both the pure PAC and the PAC modified with lignin at the predetermined levels. Figure 1 shows the synthesis of the pure binder with lignin and the respective characterization tests.

Figure 1. Synthesis of the 30/45 lignant with lignin and experimental program.

Source: Prepared by the authors.

2.3 MINERALOGICAL CHARACTERIZATION

2.3.1 Scanning Electron Microscopy (SEM) and Energy-Dispersive X-Ray Spectroscopy (EDS)

The lignin sample were morphologically analyzed by SEM using the QUANTA FEG 250 microscope, manufactured by FEI. The samples were coated with gold utilizing a Leica ACE600 high-vacuum coating chamber. SEM analysis was carried out under the following parameters: electron beam power of 20 kV, working distance ranging between 10.5 and 13 mm, spot size of
5, and image magnification at 50 and 200 x, utilizing the secondary electron detector. For EDS analysis, a detector from the manufacturer Bruker was employed, coupled to the microscope column.

2.4 PHYSICAL CHARACTERIZATION

2.4.1 Penetration test

Following the procedures described in the Brazilian standard DNIT-ME 155 (National Department Of Transportation Infrastructure (DNIT), 2010a), this test determines the consistency of the asphalt binder by measuring the depth, in tenths of a millimeter, that a needle of standard mass (100 g) penetrates vertically into a sample of binder for 5 seconds, at a temperature of 25°C.

2.4.2 Softening Point Test

Also known as the ring and ball test, the procedures described in the Brazilian standard DNIT-ME 131 (Departamento Nacional de Infraestrutura De Transportes (DNIT), 2010b). This test determines the temperature at which the asphalt binder reaches a specific consistency.

2.4.3 Ductility test

Carried out in accordance with the Brazilian standard DNER-ME 163 (National Department of Highways (DNER), 1998), This test measures the distance at which the asphalt binder breaks when subjected to constant elongation, under controlled conditions of temperature (25 ± 0.5°C) and speed (50 ± 2.5 mm/min). This test assesses the binder's ability to deform without breaking.
2.4.4 Short-term aging test

The Rolling Thin Film Oven Test (RTFOT) was conducted according to the American standard ASTM D2872 (American Society for Testing And Materials (ASTM), 2012). The test used a thin film of asphalt (35 g), which was continuously rotated in a glass container at 163 ºC for 85 min.

3 RESULTS AND DISCUSSION

3.1 SEM AND EDS RESULTS

The Figure 2 illustrates the SEM image of the sample fraction, with a magnification degree of 50 and 200 x, respectively. It can be seen in Figure 2 that the surface of the lignin shows a series of cracks, which were probably acquired in the material due to dehydration during the synthesis process carried out by the material manufacturer.

![Figure 2. Scanning electron microscopy (SEM) images of lignin at different magnifications: (left image) 50 x, showing the overall particle distribution; (right image) 120 x, highlighting the detailed morphology of the particles. Source: Prepared by the authors.](image)

Figure 3 shows the compositional analysis of the lignin by EDS, using the same point as the 200 x SEM image shown in Figure 2. The EDS analysis shows the presence of 3 elements: carbon (C) at 92.85 wt.%, oxygen (O) at 5.32 wt.% and nitrogen (N) at 1.83 wt.%. The large
predominance of carbon in the EDS is justifiable, since lignin is an organic element, where practically all of its chemical composition is made up of carbon (C₉H₁₀O₅). Hydrogen was not recorded because its atomic number is very low, preventing the detector from recording it during analysis. Nitrogen appeared in low quantities in the sample, which may have been the result of some residue during the synthesis of the material. Nitrogen is shown in green, in which it is spread out in a distributed manner around the entire region analyzed, in the same way that oxygen, represented by blue, is spread out.

Figure 3. EDS analysis of lignin.

Source: Developed by the authors.

3.2 MASS LOSS FROM RTFOT

Short-term aging simulates the oxidative process of the asphalt binder caused by the machining, application and compaction of the asphalt mixture. As a result, this procedure provides us with the variation in the mass loss of the samples submitted, as well as being a prerequisite for the other tests that need to predict the behavior of the materials under this aging condition.

According to Cravo & Bernucci (2018) the loss of mass of the binder samples indicates how much the material has suffered due to the action of the heat and air applied in the process,
causing degradation and reduction of part of the matter present. This implies changes in the composition and rheological properties of the material, such as an increase in its viscosity and rigidity. Figure 4 illustrates the mass loss results (%) of the pure binders and those modified by the addition of lignin after ageing, compared to the limit established by the National Petroleum, Natural Gas and Biofuels Agency (Agência Nacional do Petróleo, 2005).

![Figure 4. Loss of mass (%) of pure and lignin-modified binder after aging.](image)

Source: Prepared by the authors.

According to the SUPERPAVE specifications, the loss of mass cannot exceed 1 %, while in ANP Resolution n. 19 (Agência Nacional do Petróleo, 2005) this loss for pure binders must be a maximum of 0.5%. The results in figure 5 show that all the binders analyzed met the criteria established by the aforementioned standards.

3.3 PENETRATION TEST

Figure 5 illustrates the penetration results of the samples before and after aging, determined from the average of three readings on each sample. The penetration test determines the consistency of the binder at a temperature of 25°C and the penetration parameter is directly related to the stiffness property of the sidewalk.
Analysis of Figure 5 shows that the addition of lignin significantly reduced the penetration of the binder under normal and aged conditions, thus generating an increase in consistency, which could mean a tendency to increase stiffness and consequently an increase in resistance to deformation.

The limit established by ANP Resolution n. 19 (Agência Nacional Do Petróleo, 2005) for this parameter is the range of 30 to 45 tenths of a millimeter, whose values classify the very base binder used in this research. The results of the binder without added lignin and with 3% lignin met the criteria of the ANP specification.

However, the binder modified with 6% lignin did not fall within the range, as it showed a reduction in penetration, albeit minimal. Santos (Santos; Rodrigues; Mendonça, 2018) checked the properties of the 50/70 binder with additions of 3%, 6% and 9% lignin and also obtained penetration values below the specification, for example, at 9%, its value was reported at 38.4 tenths of a millimeter.

Cravo & Bernucci (2018) in their research, using lignin from the black liquor used in the production of second-generation ethanol (2G) and another highly pure commercial lignin and combining them with PAC 50/70 at levels of 1.5% and 15% of its weight, respectively, also confirms that the addition of the two types of lignin to the binder increased its consistency, reducing the penetration of the binder/additive mixture. Botaro et al. (2006) explain that the incorporation of many aromatic rings present in the structure of lignin contributes to increasing
the rigidity of the binder. The author studied the addition of 1% to 6% lignin extracted from sugar cane bagasse using the organosolv ethanol/water process to obtain CAP20 blends and the results showed a tendency for penetration to decrease as the lignin concentration increased.

Williams and McCready (2008) also contributes to these observations by stating in his work that the lignin hardened the binder at all stages of ageing. The result of the hardening improved the properties of the binder at high temperatures and was detrimental to the properties at low temperatures.

3.4 RETAINED PENETRATION

Figure 6 shows the results of retained penetration, and the results of the binders modified by the addition of lignin outperformed PAC 30/45, but did not follow a trend whereby the higher the lignin content, the higher the percentage of retained penetration (PPR). ANP Resolution 19/2005 (Agência Nacional do Petróleo, 2005) defines a minimum of 55% for this parameter. Thus, higher values for the percentage of penetration retained (PPR) indicate less sensitivity to ageing and vice versa. Thus, the addition of 3% lignin increased the resistance of the 30/45 binder to short-term ageing.

Figure 6. Percentage of penetration retained (PPR).

Source: Prepared by the authors.
3.5 DETERMINING THE SOFTENING POINT

Figure 7 shows the results of the softening point determination. This determination is another empirical measure of the binder's consistency and consists of defining a reference temperature at which the binder reaches a certain flow condition. This parameter is related to maintaining the properties of the binder at high temperatures and increasing resistance to permanent deformation.

The results were obtained from the average of two temperatures and it was found that there was an upward trend with the addition of lignin to the binder. This is advantageous, as the higher the softening point, the less deformation occurs and the less sensitive the binder becomes to temperature, maintaining its properties up to higher temperatures, a fact that can be observed with the notable increase of 28 °C in the binder with 6% lignin compared to the pure binder and 2.2 °C in the binder with 3 % lignin. It is due to these antioxidant characteristics that lignin has been studied as an additive to asphalt binders, with promising results in terms of increasing the resistance to plastic deformation of asphalt mixtures (Arafat et al., 2019; Azadfar et al., 2015; Batista et al., 2018; Wang; Derewecki, 2013; Xu; Wang; Zhu, 2017; Yuliestyan et al., 2017).
In the studies by Asukar, Behl and Gundaliya, (2016) regarding the association of two types of lignin at levels of 5% and 7% in two different binders, the authors also observed, in addition to a reduction in penetration, an increase in the softening point of the samples.

ANP Resolution N. 19 (Agência Nacional Do Petróleo, 2005) establishes a minimum temperature of 46°C for the softening point under normal conditions, and all the binders showed results that met the established limits. After aging, this parameter is limited to a maximum variation of 8 °C compared to the binder without aging. The results shown in Figure 8 prove that they comply with the specification.

In the studies made by Santos, Rodrigues and Mendonça (2018), the authors observed a smaller variation in softening points in binders modified by the addition of lignin in relation to the pure binder, reflecting its influence on resistance to ageing. However, these were evaluations on 50/70 binders. Van Vliet et al. (2016) in their research identified that the effect of adding lignin depends not only on the amount added, but also on the type of binder used.
3.6 THERMAL SUSCEPTIBILITY INDEX (TSI)

The CAP specification standard, DNIT 095 (Departamento Nacional De Infraestrutura De Transportes, 2006), establishes that the TSI, which indicates the binder's sensitivity to temperature variation, should be calculated according to Equation:

\[
TSI = \frac{500 \log(PEN) + 20(T\degree C) - 1951}{120 - 50 \log(PEN) + (T\degree C)}
\]  

(1)

Where:

\[T\degree C = \text{Softening point; PEN = penetration at 25} \degree \text{C, 100 g, 5 seg.}\]

Table 1 shows the TSI results for the pure binder and those modified by the addition of lignin. It was observed that the 30/45 and 3% lignin binders met the ANP specification, which defines the range for PAC 30/45 varying from (-1.5) to (0.7). The binder with 6% lignin obtained an IST of 2.7. According to Bernucci et al. (2008), results higher than (1.0) indicate oxidized asphalts and lower than (-2.0) asphalts that are very sensitive to temperature variations.

<table>
<thead>
<tr>
<th>Sample</th>
<th>TSI</th>
</tr>
</thead>
<tbody>
<tr>
<td>PAC 35/40</td>
<td>-0.8</td>
</tr>
<tr>
<td>Lignin 3</td>
<td>-0.6</td>
</tr>
<tr>
<td>Lignin 6</td>
<td>2.7</td>
</tr>
</tbody>
</table>

Source: Prepared by the authors.

According to the results shown in Table 1, the binder modified with 3% lignin kept the binder in a range closer to zero, which indicates that the binder is less susceptible to the effect of temperature variation, which is essential to ensure the sidewalk's performance in the field (Kalantar; Karim; Mahrez, 2012). In the sample with 6% addition, there was oxidation of the binder, as it showed values well above the index limit highlighted by Bernucci et al. (2008), compromising the rheological behavior of the binder and making it age, which could lead to possible damage to its performance in asphalt mixtures.

The research conducted by Robertson, Bishara and Mahoney (2006) observed in their studies that, at low concentrations of lignin, the binders showed limited improvement in
resistance to ageing and higher concentrations negatively affected the binders. However, in the study carried out by Hobson (2017) the action of lignin as an antioxidant was not supported.

The contradictory findings may be due to the different preparations and sources of lignin used. The physical and chemical behavior of a given lignin will differ depending on the original source and the extraction method used (Boeriu et al., 2014; Watkins et al., 2015).

For example, softwood is generally used in the pulp and paper industry, so the lignin extracted from black liquor will be predominantly guaiacyl. As rice is a grass-type plant, its lignin contains a mixture of all three types. The lignin normally extracted from pulp and paper mills by the Kraft process requires caustics and sulphide, while other processes require other sulphur-containing chemicals, acids or high pressure (Arafat et al., 2019).

3.7 DUCTILITY

For the ductility test, which indirectly assesses the cohesion of asphalt binders, the results showed that the samples modified with lignin had results similar to PAC 30/45 and in line with the limit parameter assessed by the standard (Departamento Nacional De Infraestrutura De Transportes, 2006), > 120 cm, both before and after RTFOT.

4 CONCLUSIONS

In this study, lignin granules were added to 30/45 asphalt binder to investigate its effect on short-term aging, by means of penetration, softening point and ductility tests, before and after RTFOT. Based on the results of the laboratory tests, the main conclusions are as follows:

After analyzing the empirical tests, it was observed that the inclusion of lignin in the asphalt binder contributed significantly to improving its resistance to aging compared to the pure binder. This improvement was evidenced by a reduction in penetration and consequent increase in consistency, as well as an increase in the softening point, supported by satisfactory ductility before and after the RTFOT (Rolling Thin Film Oven Test).

The lignin gave the binder less sensitivity to temperature variation, as indicated by a Thermal Susceptibility Index (TSI) closer to zero.
The use of lignin in PAC 30/45 did not result in improvements as significant as those observed in previous studies with PAC 50/70. However, the results indicated a tendency for the binder to improve with the addition of 3% lignin compared to the unmodified binder. On the other hand, the incorporation of 6% lignin made PAC 30/45 more prone to oxidation, compromising its ability to improve the properties of asphalt mixtures exposed to short-term ageing.

The use of lignin is viable for improving the rheological properties of PAC 30/45 asphalt binder, providing greater rigidity, durability and resistance to ageing. It is recommended to explore other levels of lignin addition, investigating concentrations below 3% to confirm the benefits of small additions, and examining concentrations above 6% to better understand the oxidative tendency of this binder in larger quantities.

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