RESEARCH ARTICLE OPEN ACCESS

The mechanical performance of lignin-modified asphalt binders

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ABSTRACT
Asphalt is one of the most used materials for covering roads. Asphalt pavement can be degraded by the cyclical stress caused by passing vehicles. For this reason, several materials have been tested to improve the properties of asphalt mixtures, including lignin. In this sense, this work aimed to evaluate the mechanical properties of asphalt binders modified with lignin, such as the Marshall stability, compressive strength, and resilience modulus. The most suitable binder content for the performed tests was 5.21 wt.% of asphalt mixture. The addition of lignin in the oil binder present in the asphalt did not cause significant changes in Marshall stability and creep nor in the tensile strength. However, in the resilience test, samples modified with lignin, especially with 6 wt.% content in the asphalt binder, showed a greater resistance to plastic deformation. Thus, one can infer that the addition of lignin in the asphalt binder did not deteriorate its mechanical properties.

KEYWORDS**:** asphalt, binder, lignin, Marshall test, tensile test.

Date of Submission: 13-02-2021 Date of Acceptance: 27-02-2021

I. INTRODUCTION

Asphalt is one of the most used pavement materials and is applied mainly for soil waterproofing. Asphalt pavement provides a smooth surface that is resistant to skidding and deterioration because of bad weather and defrosting chemicals [1].

Asphalt pavement can be degraded by the cyclical stress caused by passing vehicles. Under this regime, cracks that are easily propagated can be formed in case of any kind of degradation effect on the bituminous mixture such as binder oxidation and volatile loss [2,3].

The binder aging is one of the most common causes of reduced asphalt performance, which attracts researchers' attention to the area to change this situation. The asphalt binder comes from oil refining and is composed of several hydrocarbons of varying formulas that, under the influence of heat, ultraviolet (UV) radiation and oxygen, can oxidize and stiffen the asphalt [4,5].

Currently, several materials have been tested to improve the properties of asphalt mixtures, such as: natural asphalts, natural and artificial polymers [6-8], hydrated lime [4], lignin [9], polyphosphoric acid [4,8], UV radiation absorbers and antioxidants [9-11]. Other variables have been explored for the optimization of asphalt, such as its granulometry, the amount added and the methodology for adding these compounds.

Zhang *et al.* [12], for example, analyzed the addition of styrene-butadiene-styrene (SBS) associated with sulfur in the asphalt binder. Before aging, the addition of sulfur to the asphalt modified with SBS significantly improved the dynamic viscosity and thermal stability because of the formation of a structural network vulcanized with SBS. After aging, though, the addition of sulfur did not show favorable results for dynamic viscosity and thermal stability.

Among the studied additives for asphalt binder, lignin has recently attracted increasing interest in the scientific scenario. Lignin is a polymer present in plants with structural (provides rigidity to the cell wall), chemical (oxidation resistance) and microbiological functions [13-16]. This natural polymer is a complex phenolic compound that is only less abundant in plants than cellulose. Present in the plant cell wall, the structure of lignin is not yet completely known for presenting variations in its chain, which depend on several parameters, such as the type of plant, plant life span, geographic location and so on [17].

Nowadays, there are few studies in the literature using lignin as an antioxidant additive for asphalt binders. These studies have shown promising results, concerning abrasion resistance and adhesion improvements. The interest in the use of this biopolymer contemplates an environmental appeal since the paper and cellulose industry have been looking for alternatives to commercialize lignin. Most of the by-products of this type of industry are lignin-based materials and due to its high strength and structural variety, it finds few applications as a product with high added value. Mostly, lignin constitutes the incinerated biomass to generate energy for the industry itself [18,19].

The fact that some studies in the literature indicate that lignin has antioxidant properties, which could delay the oxidative aging of asphalt, increases the interest in its use [20]. Since there is no additive in widespread use acting as an antioxidant, lignin can be a promising alternative [20].

Fayzrakhmanova *et al.* [21] studied the variation in binder adhesion with the addition of lignin at different levels (0 to 100%) and concluded that the adhesion increased up to 10% of lignin but decreased for higher levels in the binder. The authors justified this behavior by the presence of functional groups in lignin that increased the adhesion on the surface of the asphaltene micelles to approximately 10% of lignin when this compound is saturated. The resistance of the bituminous mixture at levels greater than 10 wt.% of lignin addition reduces to values close to the initial resistance of the lignin. In other words, this polymer becomes determinant to the resistance of the mixture.

The work of Fu *et al.* [22] also demonstrated the effect of adding lignin and anti-furrow agent to a bituminous mixture. The authors reported an increase in the performance of the binder with the addition of lignin fibers, such as dynamic stability. However, this performance was lower with the increase in lignin content. The optimum content found in the bituminous mixture was 0.36 wt.% of lignin and 0.40 wt.% of the anti-furrow agent.

Batista *et al.* [20] studied the antioxidant effect of lignin in the binder, also called Petroleum Asphalt Cement (PAC). For this, penetration test, softening point, Brookfield viscosity, dynamic shear test (RCD), and thermal analysis with thermogravimetry (TG-DTG) were performed. The results indicated that the addition of lignin increased viscosity and reduced the penetration capability, besides working satisfactorily as an antioxidant agent.

In this context, the present study aimed to evaluate the lignin-modified asphalt binders using the measurement of the following mechanical properties: Marshall stability, radial compression resistance and resilience modulus. The measurement of these properties is important to evaluate the behavior of the bituminous mixture during its service life in pavements. Furthermore, it is important to determine the optimum lignin content in the binder to obtain the most suitable properties for application in asphaltic pavements.

II. MATERIALS AND METHODS

2.1 Characterization of binders

The commercial Kraft lignin was supplied by the Suzano Paper and Pulp industry in Brazil. The properties of the lignin are shown in Table 1. The asphalt binder, PAC 50/70, was supplied by the Gabriel Passos refinery (Petrobras) in Brazil, together with its characterization data, obtained in accordance with the National Department of Transport Infrastructure-DNIT 095/2006 Standard. The physicochemical properties of the binder are shown in Table 2.

Table 1: Physicochemical properties of commercial Kraft lignin.

PARAMETERS	RESULTS	UNIT
Average molar mass in number (Mn)	2260 ± 32	g mol^{-1}
Average molar mass by mass (Mw)	3378 ± 79	g mol^{-1}
Polydispersity (Mw / Mn)	1.5	-
Ash content at 800ºC, 12 h	3.3	%
pH	3.5	-
Moisture content	3.8	%

Table 2: Physicochemical properties of PAC 50/70.

PROPERTIES	RESULTS	UNIT
Mass variation	-0.182	%
Ductility at 25°C	> 150	cm
Increase in softening point	3.9	ºC
Penetration retained	57	%
Thermal susceptibility index	-1.1	-
Solubility of trichlorethylene	99.9	% w/w
Softening point	49.8	ºC
Penetration	54	mm

Flash point	328	ºC
Density	1.0069	g cm^{-3}
Brookfield viscosity at 135°C	337.5	cP
Brookfield viscosity at 150°C	168.6	cP
Brookfield viscosity at 177°C	62.64	cP

2.2 Particle size analysis

The specimens were produced with gneiss samples supplied by a Santiago Mining industry (Brazil), according to the DNER-PRO 120/97 Standard. The samples were mixed and quartered according to the DNER-PRO 199/96 Standard. According to the granulometry, they were classified as crushed stone 1, crushed stone 0 and stone powder, as defined in the DNIT-ES 031/2006 Standard.

The samples were dried in the oven at 110 ± 5°C, cooled to room temperature, and passed through a series of sieves to perform the granulometric analysis and. The mass retained in each sieve was measured and classified, according to the DNIT-ES 031/2006 Standard.

2.3 Marshall method

The Marshall method of asphalt mixtures followed the NBR 15785:2010 Brazilian Standard. Firstly, the conventional binder was heated to 140°C, the aggregates were heated to 150°C and both mixed for 20 minutes. The dough was placed in an iron mold and five specimens were obtained from 75 strokes per face. In this testing step, no lignin was added. The composition of the binder in each part is shown in Table 3.

To choose which binder content should be used, the volumetric parameters were calculated: total void volume (Vv), voids in mineral aggregate (VMA) and bitumen void ratio (BVR). Vv and BVR were used to define the ideal content. The tests described in the following sections were carried out using this ideal binder content.

Table 3: Composition of the specimens.

CAP CONTENT (%)	CAP MASS (g)	AGGREGATE MASS (g)	MASS FOR 1 SPECIMEN (g)
4.53	57	1200	1257
4.76	60	1200	1260
4.99	63	1200	1263
5.21	66	1200	1266
5.44	69	1200	1269

2.4 Mechanical tests

After the determination of ideal binder content, twenty-four specimens were produced, six with conventional binder, six with ligand containing 1 wt.% lignin, six with asphalt binder containing 4 wt.% lignin and six with binder containing 6 wt.% lignin. The Marshall test, tensile test by diametrical compression and resilience test were performed in duplicate for each lignin content.

Marshal test followed the NBR 15785/2010 Brazilian Standard. Marshall specimens with the necessary composition in each test were produced with 100 mm in diameter and 63.5 mm in height. The equipment consists of two heads, the upper fixed and the lower mobile, moving at a rate of 5 cm min^{-1} at 60°C. The maximum load applied to the body is defined as Marshall stability, measured in units of force (kgf or N) and the maximum displacement of the head constitutes the Marshall fluency, measured in millimeters.

The resistance to diametrical compression test was performed according to the NBR 15087/2012 Brazilian standard. The specimens had a diameter of 10.0 ± 0.2 cm and the length varied from 3.5 cm to 6.5 cm. All measurements were performed in duplicate.

The specimens were cooled to a temperature of 25.0 ± 0.1°C for two hours. Subsequently, they were placed in horizontal position between the press plates and were compressed at a speed of 0.8 mm s^{-1} until the material ruptured in the vertical diametrical plane. The tensile strength by diametrical compression was calculated according to Eq. (1):

$$\sigma = \frac{2F}{\pi DH}$$

On what:
σ = tensile strength, in kgf cm^{-2};
F = breaking load, in kgf;
D = diameter of the specimen, in cm;
H = height of the specimen, in cm.

The resilience modulus test was performed according to the NBR 16018:2011 Brazilian standard. The specimen was placed at the base of the press and supported on the lower concave frieze. The loading piston with the upper frieze was seated in contact with the specimen, diametrically opposite to the lower frieze. The load used was 10% of the value of the previous test. Linear Variable Displacement Transducers (LVDT) were connected to the specimen and its behavior under the stress was recorded on a microcomputer. The applied stress and the corresponding deformation were measured.

III. RESULTS AND DISCUSSION

3.1 Granulometry

One of the most important parameters in the production of HMA (hot mix asphalt) is the granulometry of the aggregates, as this will directly influence the properties of the asphalt. The appropriate granulometry, besides the PAC, must provide a mixture that supports the loads that are requested, in addition to not deteriorating early due to climate and traffic changes. To ensure minimum conditions of use, some regulations standardize the size range that must be obtained to produce asphalt. In this sense, the DNIT-ES 031/2006 standard defines the granulometric ranges A, B and C, based on the amount of through mass in each ASTM series sieve. Series A features a coarser grain sample, series B, intermediate grain size and series C, finer grain size. There are the upper and lower limits of the retained mass in each sieve, allowing the characterization of the samples according to the classification above.

Thus, a granulometric analysis was performed to characterize the sample obtained from the mining company. Fig. 1 presents the results regarding the working range, upper limit, and lower limit of the granulometric range C according to the DNIT-ES 031/2006 standard. When carrying out the tests and comparing them with the standard, it was concluded that the gravel used in this work belongs to the granulometric range C, as observed by the working range of Fig. 1 and, therefore, is allowed for use in the production of the asphalt mixture. As this strip presents finer granulometry, the tendency for the asphalt is to present a higher density, with greater contact between the particles and smaller voids in the mineral aggregate, providing better stability.

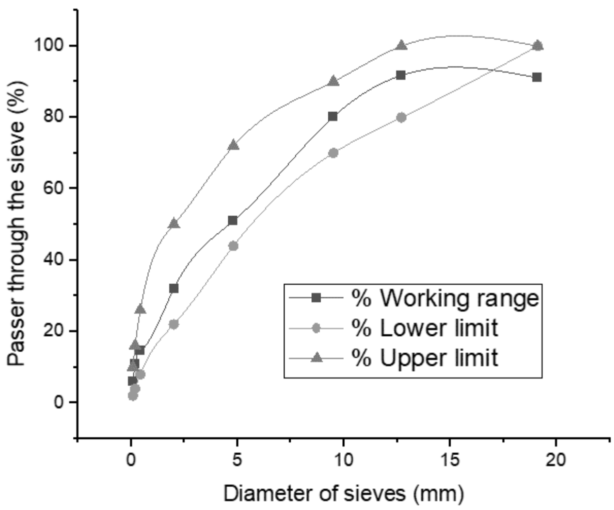

Figure 1: granulometric analysis of this work based on the DNIT-ES 031/2006 Brazilian Standard

3.2 Marshall dosage

Asphalt needs to exhibit some characteristics that allow it to perform satisfactorily in service. For this, it is necessary to define an optimum binder content that promotes a balance of the desired properties. For the determination of this content, specimens were made from the mixture of the binder in different levels and the aggregates. Based on the contents of the binders and on the densities of each component of the asphalt mixture, the following volumetric properties were calculated:

- Apparent density (d);
- Theoretical maximum density (TMD);
- Volume of voids (Vv);
- Bitumen voids (VFB);
- Voids in mineral aggregate (VMA);
- Bitumen / voids ratio (BVR).

To define the most suitable content of PAC, the method that considers only two volumetric parameters was used, Vv and BVR. The other parameters are used to calculate these two main ones. Those two parameters describe air voids, that are necessary within the compacted mixture to allow thermal expansion of the binders and to support the slight compaction caused by traffic. Besides that, Sheng *et al.* [23] discussed the relationship between void volume, water permeability in asphalt and, consequently, durability. Thus, the DNIT 031/2006 standard defines the ideal values for the two volumetric parameters Vv and BVR, with the value of Vv being 3% and 5% and the value of BVR must be between 75% and 82%. Based on this, the optimum content of binder chosen for the asphalt mixture was 5.21 wt.%. The data obtained in the volumetric analysis are shown in Table 4.

Table 4: Volumetric parameters calculated to determine the optimum content of the asphalt mixture.

CAP CONTENT IN THE MIXTURE	VV (%)	BVR (%)
4.53	5.97	64.03
4.73	5.95	65.13
4.99	5.22	69.14
5.21	3.80	76.48
5.44	3.47	78.80

3.3 Marshall method

The Marshall test is performed to determine the stability and creep of bituminous mixtures. Marshall stability is the maximum resistance to radial compression, while creep is the total deformation presented by the specimen until the maximum load is applied. Arabani and Tahami [24] stated that stability indicates the resistance of the asphalt mixture to pressure, horizontal tension and shear induced by the compression load.

Fig. 2 presents the Marshall stability values for mixtures with different contents of lignin concerning the binder (1 wt.%, 4 wt.% and 6 wt.%). The asphaltic mixtures with a lignin content of 1 wt.% and 6 wt.% had a reduction in the stability value, observing the average values. The mixture with a 4% lignin content showed an increase in stability of 13%, which represented the best result. However, in general, there was no improvement in Marshall stability with the addition of different levels of lignin. Anyway, all values are in accordance with the DNIT 031/2006 Brazilian Standard, which establishes a minimum value for stability of 500 kgf.

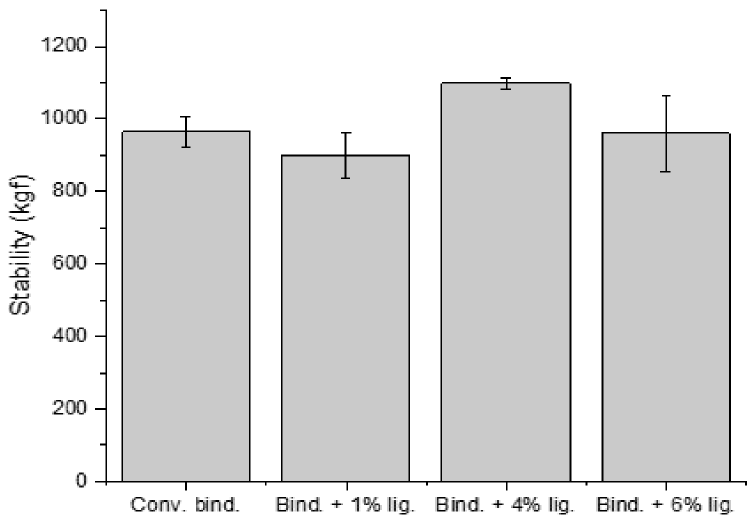

Figure 2: Marshall stability of conventional ligand and lignin-modified ligand.

According to Chen *et al.* [25], the effect of increasing stability is due to the addition of lignin fibers, which can increase the adhesion of the components of the asphalt mixture, in addition to serving as a hindrance for the propagation of cracks when acting as a “bridge”, which was called the "bridging cracking effect". Thus, lignin fibers promote increased resistance to the wheel track, which is related to the vehicle traffic, also preventing the advance of cracks that weaken the material. However, the asphalt is non-uniform and multiphase, requiring binders to allow cohesion between the phases. In this sense, uneven dispersion of lignin occurs throughout the mixture, promoting differences in the performance of the material. Also, according to the authors, if the lignin content is greater than 10 wt.%, the effect of the dispersion through the asphalt will cause notable weaknesses.

The work developed by Sheng *et al.* [23] evaluated the addition of mineral fibers, polyester, flocculant lignin separately and together in the Stone Matrix Asphalt (SMA). In their findings, the authors observed that the addition of these fibers improved Marshall stability. In the case of lignin fibers, with an analysis range of 0.1% to 0.5%, values of 8.5 to 12.5 kN were obtained, approximately. Moreover, Panda *et al.* [26] added mature coconut fibers to the SMA, produced with a binder with viscosity grade 30 (VG-30) and aggregates of larger particle sizes, improving of the Marshall stability of the mixture up to 0.5% fiber content, with a reduction after that value. The maximum value found in the Marshall test was approximately 11 kN.

Marshall fluency values are shown in Fig. 3 and there is a reduction in binder performance compared to the conventional binder. The reductions for 1 wt.%, 4 wt.% and 6 wt.% of lignin were 5.6%, 6.8% and 12.8%, respectively.

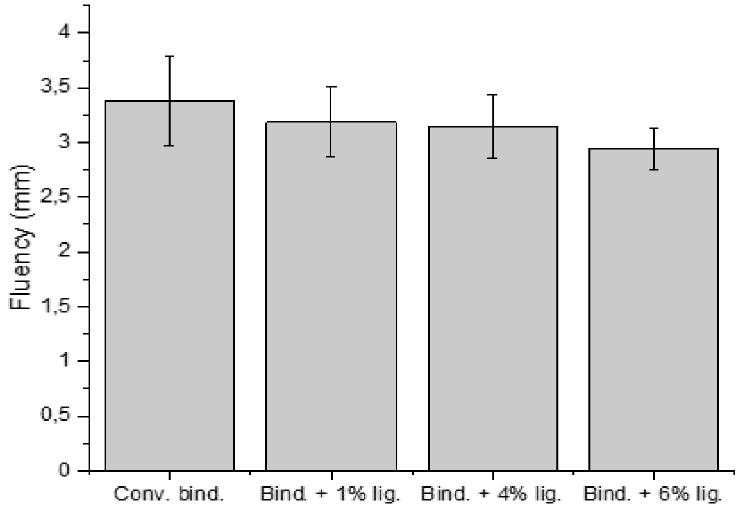

Figure 3: Marshall fluency of conventional binder and lignin-modified binder

According to Chen *et al.* [25], the increase in fluency value indicates that the resistance to crack propagation also increases. Although the results show that there was no variation in the fluency value, they are in accordance with the limits reported by Panda *et al.* [26], who recommended values between 2 mm and 4 mm. The results of that study indicated stabilization in the fluency values between 0% and 0.5% of coconut fiber content, reaching values close to 3 mm.

According to Arabani and Tahami [24], the Marshall coefficient (CM), obtained by the ratio between stability and fluency Marshall, indicates the rigidity and resistance to permanent deformation obtained by the asphalt mixture. If there is an increase in the value of this coefficient with the addition of lignin fibers, this is probably due to the increase in adhesion between binder and aggregate, since these fibers, because they have a porous structure, adsorb the binder and decrease the free content in the mixture. This phenomenon makes the material more resistant to withstand loads.

From this and observing the Marshall coefficient values for the conventional and lignin-modified binder shown in Fig. 4, there was practically no change with the addition of 1 wt.% of lignin. Nevertheless, there was an increase of 20% for mixing with 4 wt.% of lignin and a 13% increase for the 6 wt.% lignin content. Due to increases in the performance of the mixture, it will be possible to improve the rigidity and resistance to permanent asphalt deformation with the addition of lignin in levels of 4 wt.% and 6 wt.%.

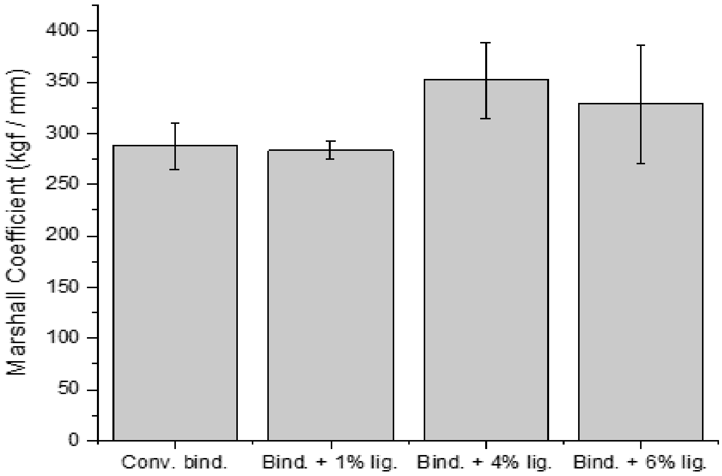

Figure 4: Marshall coefficient for the sample of conventional and modified lignin binder.

3.4 Tensile strength test by diametrical compression

Fig. 5 shows the values recorded in the tensile test by diametrical compression in bituminous mixtures with conventional binder and lignin-modified binders.

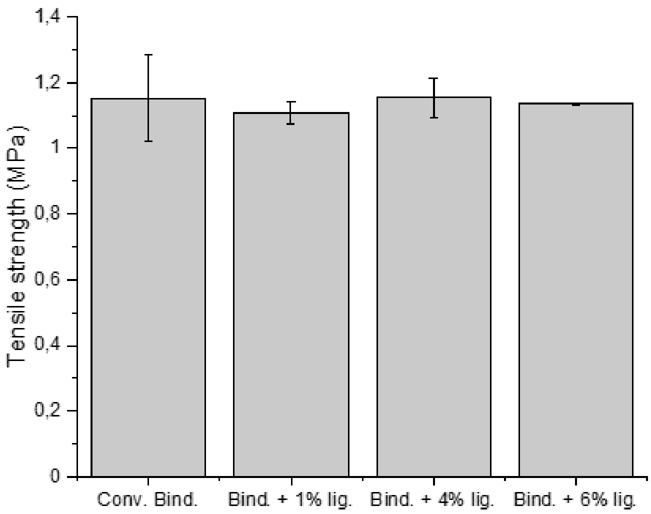

Figure 5: tensile strength by diametrical compression of conventional binder and lignin- modified binder.

Peng *et al.* [27] studied the changes in mechanical properties in asphalt modified with lignin-based polyurethane. The tensile strength values of the mixture are presented in the study, obtaining a maximum value of 311.1 kPa for the addition of 10 wt.% of this additive, in contrast to the values of 244.4 kPa obtained without its presence, before UV aging process. The authors stated that the increase in the polyurethane content in the asphalt mixture increases its viscosity and, thus, increases the cohesion between the particles of the material, reflecting in the improvement in the tensile strength. Furthermore, in the work of Abdelsalam *et al.* [28] the effects on the mechanical properties of asphalt modified with diatomite (DMAM), lignin fiber (LFMAM) and diatomite-lignin fiber (DLFMAM) were studied. The tensile strength showed higher values in the presence of fibers when compared to asphalt without added fibers. The values presented were 0.711 MPa for the control asphalt mix (CAM) 0.738 MPa for DMAM, 0.748 MPa for LFMAM and 0.773 MPa for DLFMAM.

In this work, there were no changes in tensile strength with the addition of lignin in the bituminous mixture. The found values agree with the NBR 15087/2012 standard, which establishes a minimum value for the tensile strength of 0.65 MPa.

3.5 Resilience modulus

The values obtained in the resilience module test for the conventional binder and for lignin-modified binders are shown in Fig. 6.

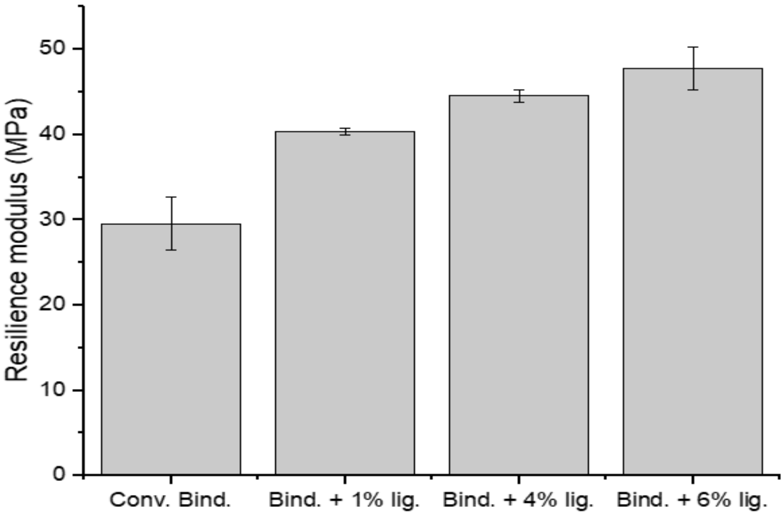

Figure 6: resilience module of conventional ligand and ligand modified with lignin.

Pourtahmasb *et al.* [29] reported that the resilience module (MR) test can be used to represent the asphalt mix conditions subjected to traffic on pavements and offers the possibility to compare the behavior of asphalt under various conditions and stress states. Furthermore, according to Karami *et al.* [30], mixtures with superior MR can distribute the loads over a wider area and, possibly, be more resistant to crack deformation. Accordingly, it is shown that there was an advantage in adding certain levels of lignin to the bituminous mixture, as it increased the MR. The most satisfactory result was obtained from the mixture with 6 wt.% lignin content, which exhibited an MR increase of 45% concerning the conventional binder.

The MR analysis indicates that there is a better response of the asphalt to the successive stresses caused by the traffic, when absorbing the impact and returning to its initial state. Therefore, there is a reduction of permanent deformations in its structure, increasing its durability. Briefly, there was an increase in the flexibility of the asphalt due to the addition of lignin, especially with the content of 6 wt.%, possibly caused by the increased adhesion of the components.

IV. CONCLUSION

The present work aimed to evaluate the mechanical properties of the bituminous mixture at different lignin and binder contents. Analyzing the Marshall test, the most suitable binder content was 5.21 wt.% of asphalt mixture.

The addition of lignin in the asphalt binder did not cause changes in Marshall stability and creep nor in the tensile strength in relation to the resilience module. Thus, the samples modified with lignin, especially with a content of 6 wt.%, presented greater resistance to plastic deformation and, therefore, possibly would better withstand the stresses typically imposed on the roads. Possibly, with the addition of lignin, there was an increase in adhesion between the binder and the aggregates in the asphalt, promoting better results than those obtained without the addition of the polymer.

In conclusion, lignin can be used as an additive in the asphalt binder for maintaining the mechanical properties already presented by the asphalt or, even, improving the resistance to plastic deformation. Linked to its antioxidant action and to the possibility of its use from the residues of the cellulose and biorefineries industries, lignin becomes more advantageous than some other additives described in the literature and used currently. The data obtained in the literature and those presented in this work show its application in modified asphalt for paving purposes should increase the useful life of the material by improving its resistance to continuous car traffic.

ACKNOWLEDGEMENT

The authors would like to thank to the company Suzano, a Brazilian paper and pulp industry.

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