EVALUATION OF MOISTURE DAMAGE IN ASPHALT CONTAINING CASHEW NUT SHELL LIQUID (CNSL) MODIFIED BITUMEN

Edeilto Almeida Ribeiro[1]*, Fernando Felipe Lopes Antunes*[1]*, Verônica Teixeira Franco Castelo Branco*[1]*, Sandra Aguiar Soares*[2]*, Jorge Barbosa Soares*[1]
[1]*Departamento de Engenharia de Transportes, Universidade Federal do Ceará, Fortaleza, Brazil*
[2]*Departamento de Química Orgânica e Inorgânica, Universidade Federal do Ceará, Fortaleza, Brazil*

ABSTRACT

Moisture damage is a frequent distress on pavements and is considered a major contributor to premature deterioration. Physicochemical properties of the cashew nut shell liquid (CNSL) showed to be potentially useful to improve the adhesion between bitumen and aggregates. The main objective of this study is to evaluate the potential of the CNSL modified bitumen to increase the asphalt resistance to moisture damage, and as a promoter of adhesiveness between bitumen and aggregates. Pure and modified bitumen with different CNSL contents were characterized in terms of chemical and rheological properties. The aggregates were characterized by X-ray fluorescence. Asphalts were designed using the Superpave methodology. The modified Lottman and resilient modulus results for the asphalt containing bitumen modified with CNSL were compared to those of two distinct asphalts: one with unmodified bitumen and natural aggregates, and another with the same constituents but with 2% of lime as a filer. The asphalts stripping resistances were also characterized through digital image processing (DIP). The findings showed that the CNSL decreased the bitumen viscosity. Both, pure and modified bitumen, were classified as PG 70-28. The findings showed that the asphalt with the bitumen modified with CNSL had better stripping resistance and satisfactory mechanical properties if compared with the other two asphalts. It is expected that the CNSL can be used as an additive to prevent striping in asphalts contributing to its moisture damage resistance.

Keywords: Asphalt, Moisture Damage, Cashew Nut Shell Liquid (CNSL)

1. INTRODUCTION

Stripping, is often associated with high traffic volume, weather and material properties. It has been deteriorating pavements and increasing maintenance and rehabilitation costs. A deteriorated pavement affects the number of accidents and the pollutants level. Road infrastructure quality is directly related to the economic and social country development [1].

Asphalt pavement surface courses are generally designed to have air voids contents around 7%, with an average of 4% due to the traffic densification, and may have 2% at the end of its life. The loss of adhesion between bitumen and aggregates when the air voids or moisture are excessive affects performance [2]. This phenomenon does not happen in the entire asphalt layer, but in specific areas with air voids saturated with water or steam water [2]. The deleterious effect of water in asphalt is defined as a progressive functional deterioration caused by loss of adhesive bond between bitumen and aggregates and/or loss of cohesive strength within the bitumen film itself [3]. The adhesion phenomenon involves the development of electrostatic interactions that depend on the bitumen and aggregates chemical nature [4].

There are nonpolar hydrocarbons compounds present in bitumen whereas the polar surface of the aggregate may have positive or negative charges. In order to avoid moisture damage in pavements, additives to increase the chemical compatibility between bitumen and aggregates can be used. Hydrated lime is the additive most commonly used [5]. The tests used to evaluate the moisture damage in asphalts are generally divided into two categories: (i) tests on loose asphalts and (ii) tests on compacted asphalts. Most of these tests are empirical and not related to the compatibility between bitumen and aggregates [6].

The cashew tree (*Anacardium occidentale L.*) is found in Africa, Asia, Vietnam, India, Central America and Brazil. The true fruit from cashew tree is the nut and the “cashew apple” (penduncle) is the pseudo-fruit. Brazil leads the world in the production of cashew nut (70% of the total production). The cashew culture is one of the main agronomic activities in Northeast Brazil, specially concentrated in the states of Ceará, Piauí and Rio Grande do Norte [7]. The cashew nut and the cashew apple are used in human nutrition. In the cashew nut there is a dark black liquid, caustic, and flammable known as cashew nut shell liquid (CNSL) that represents 25% by weight of the cashew nuts [8]. This liquid has been used in industry as an antioxidant for fuels and lubricants, it is also used in manufacture of cement, varnishes and paints, and has fundamental importance for the polymer industry. The CNSL is rich in unsaturated long-chain and natural phenolic compounds: anacardic acid, cardanol, cardol, 2-metilcardol, polymeric materials (Figure 1).

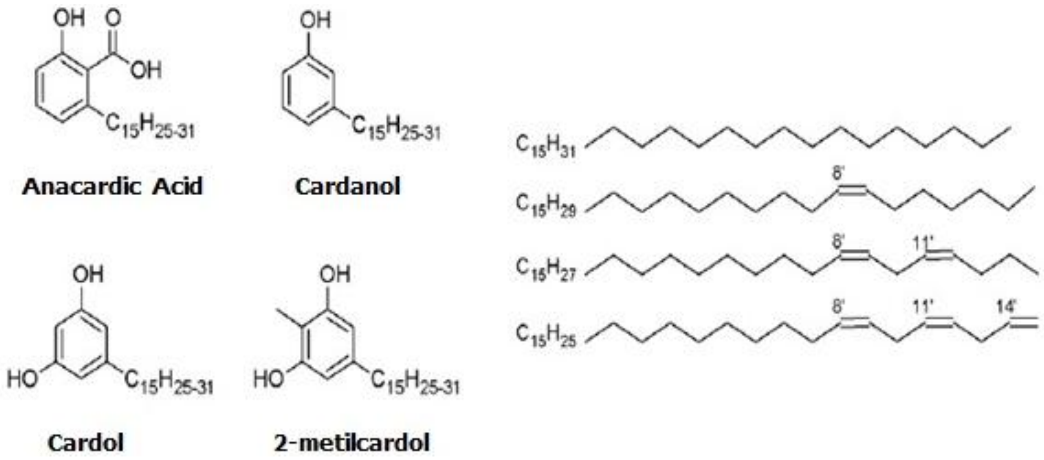


Figure 1 – CNSL chemical composition [8]

The CNSL extraction process can be done in two ways: (i) using solvents (natural CNSL) or (ii) using high temperatures during the industrial processing of cashew nuts (technical CNSL). At high temperatures (180ºC), the process leads to decarboxylation of the anacardic acid to form cardanol (Figure 2) [9].

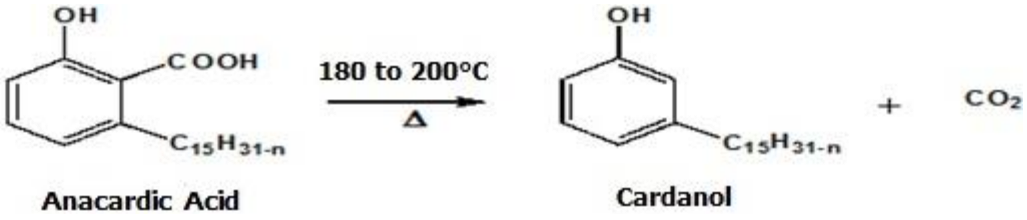


Figure 2 – Decarboxylation process [9]

The decarboxylation process explains the difference between natural and technical CNSL. Natural CNSL has high amounts of natural anacardic acid and no polymeric material in its constitution. Technical CNSL has a high cardanol content and also polymeric material (Table 1) [8].

Table 1 - Natural and technical CNSL compositions [8]

Phenolics Components	Natural (%)	Technical (%)
Anacardic Acid	71.7 – 82.0	1.1 – 1.8
Cardanol	1.6 – 9.2	67.8 – 94.6
Cardol	13.8 – 20.1	3.8 – 18.9
2-metilcardol	1.6 – 3.9	1.2 – 4.1
Others	0 – 2.0	0 – 7.4

The CNSL antioxidant activity is due to the presence of phenolic groups in its constitution. CNSL is a natural source for unsaturated long- chain phenols. CNSL also has surfactants properties that can potentially contribute to improve the compatibility between bitumen and polymers and can also be used substituting traditional petroleum solvents with lower pollutant impact [10]. The objective of this study is to verify the CNSL as a potential bitumen modifier based on its surfactant characteristics that can contribute to improve the chemical compatibility between bitumen and aggregates in the presence of water.

2. EXPERIMENTAL

The pure bitumen with a 50/70 penetration grade was produced by Petrobras. The bitumen was modified with technical CNSL supplied by Iracema Cashew Nut Company. CNSL modified bitumen was prepared using a shear laboratory mixer at 1,000 rpm. Four CNSL contents were used: 0.5, 1.0, 1.5 and 2.0% by weight of bitumen. The pure bitumen was heated at 160°C during one hour. Granitic aggregates were used for the asphalts. A gradation with 19.1mm nominal maximum aggregate size was selected (Figure 3). It was used hydrated lime, 2% by weight of aggregates, as the filler for one of the investigated asphalts.

The asphalt containing bitumen modified with CNSL was compared to two other asphalts designed using the Superpave methodology. Specimens were separated in three groups. The first group is the asphalt containing unmodified bitumen (B). The second group is the asphalt containing bitumen modified with 2% of CNSL (B + 2% CNSL). The third group is the asphalt using unmodified bitumen and 2% of lime (B + 2% Lime). Table 2 shows the design properties of the investigated asphalts.

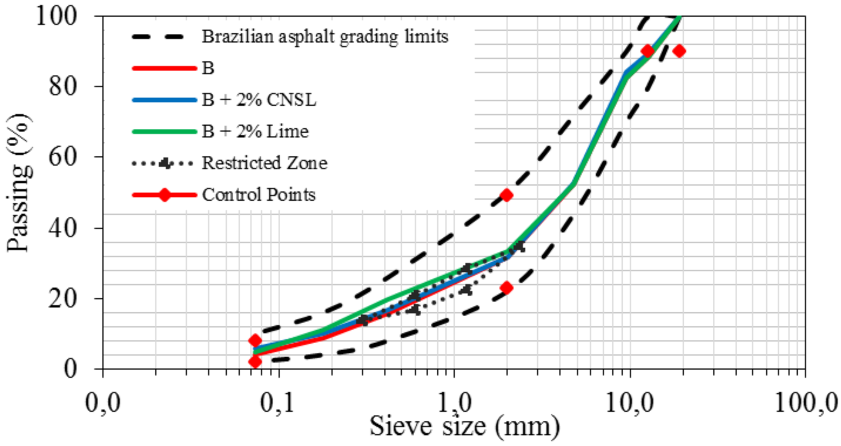


Figure 3 - Asphalts gradation

Table 2 - Asphalt design parameters

Asphalt parameters	B	B + 2% CNSL	B + 2% Lime
Bitumen content (%)	6.0	6.0	5.5
Air voids (%)	4.3	3.7	3.6
G_{mm}	2.414	2.414	2.441
G_{mb}	2.311	2.324	2.345

2. RESULTS AND DISCUSSION

2.1 Viscosity

The viscosity was measured using a rotational viscometer [11]. The measurements were performed at 135, 150 and 175°C (Figure 4). The results illustrate a notable decrease in the viscosity for the modified bitumen. CNSL surfactant properties possibly caused a molecular rearrangement reducing their resistance to flow [12]. This decrease in viscosity can lead to lower energy costs.

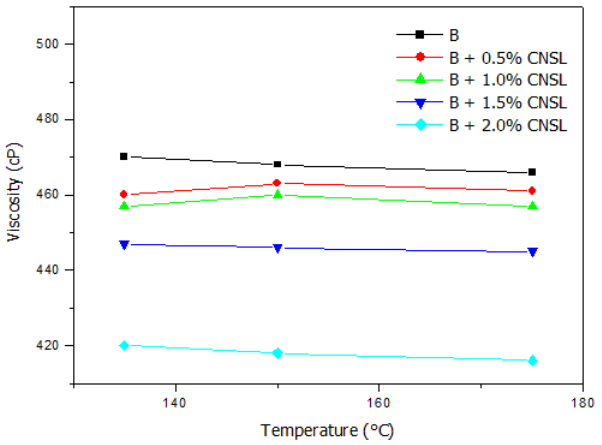


Figure 4 - Viscosity as a function of temperature for the pure and the modified bitumen samples

2.2 Mixing and Compaction Temperatures

Mixing and compaction temperatures were determined using the rotational viscometer results and the dynamic shear rheometer (DSR) according to Casola method procedure [13]. The viscosity values data were correlated with the mixing and compaction temperatures through the logarithmic curve of viscosity versus temperature [14]. In the Casola procedure, the mixing and compaction temperatures were determined using a frequency sweep test from 0.1 to 100 rad/s and a specific temperature range (40 to 100°C). The values were obtained using the equations below, where *w* corresponds to the phase angle at 86°C.

$$\text{Mixing temperature (°F)} = 325 \times w^{-0,0135} \quad (1)$$

$$\text{Compaction temperature (°F)} = 300 \times w^{-0,012} \quad (2)$$

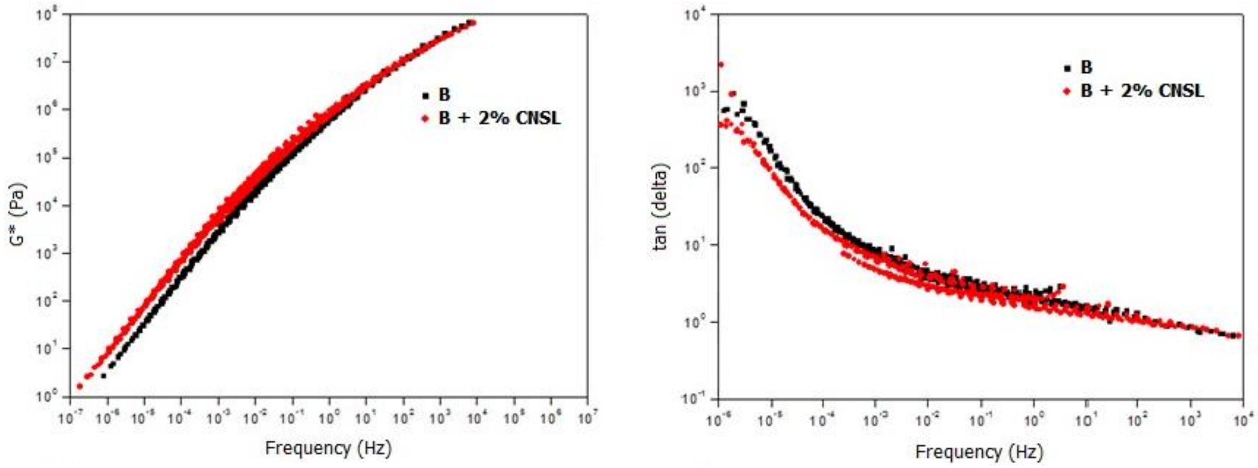
Sample B + 2% CNSL provided a smaller reduction in the mixing and compaction temperatures (Table 3). The temperature values for B + 2.0% CNSL were the lowest. The analysis for the Casola procedure was performed only for the 2% content. These results show that the CNSL addition may reduce energy costs, decreasing volatiles emission, and excessive oxidation.

Table 3 - Mixing and compaction temperatures

Bitumen	**Rotational viscometer procedure**		**DSR - Casola procedure**	
	Mixing Temperature (°C)	**Compaction Temperature (°C)**	**Mixing Temperature (°C)**	**Compaction Temperature (°C)**
B	154 – 161	143 – 147	154	142
B + 0.5% CNSL	154 – 161	143 – 147	-	-
B + 1.0% CNSL	154 – 160	143 – 147	-	-
B + 1.5% CNSL	153 – 159	142 – 147	-	-
B + 2.0% CNSL	152 – 158	141 – 146	152	140

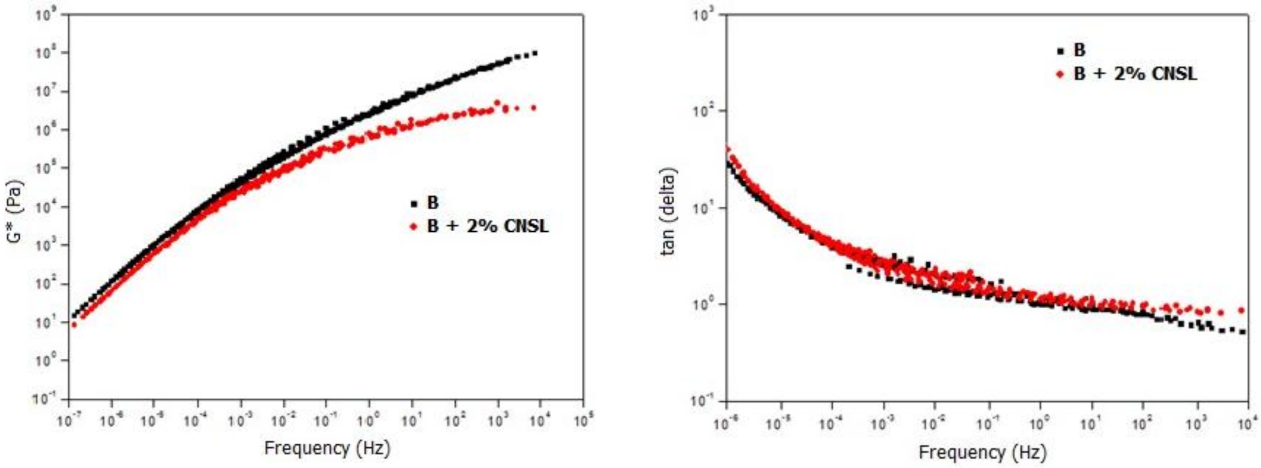
2.4 Complex Modulus, Phase Angle Master Curves and Bitumen Performance Grade (PG)

DRS measurements were performed in a stress-controlled equipment with parallels plates of 25mm and 8mm diameter. Complex modulus and phase angle as a function of temperature were measured [15,16]. The effect of temperature was analyzed from 0.01 to 10Hz, at high (> 45°C) and low temperatures (40 to -10°C). Master curves were constructed for a reference temperature of 25°C [17]. Figures 5 and 6 illustrate the analysis for B and B + 2% CNSL samples before and after RTFOT aging [20].



(a) (b)

Figure 5 - Master curves for B and B + 2% CNSL samples before RTFOT: (a) G^* versus frequency (Hz) and (b) tan (δ) versus frequency (Hz)



(a) (b)

Figure 6 - Master curves for B and B + 2% CNSL samples after RTFOT: (a) G^* versus frequency (Hz) and (b) tan (δ) versus frequency (Hz)

For low temperatures (high frequencies), the two curves are practically overlapped (Figure 5a). Bitumen B + 2% CNSL increased the G^* value when compared to B at intermediate temperatures, and especially for high temperatures (low frequencies). This fact is possibly associated with the oligomerization reaction at high temperatures (low frequencies). Probably during oligomerization the double bonds connections present in the aliphatic cardanol chains were broken causing its alignment [19].

It is observed a variation of tan δ for samples B and B + 2% CNSL (Figure 5b). Sample B + 2% CNSL showed a small increase in elasticity when compared to sample B, and this variation was observed only for high temperatures (low frequencies). This increase in elasticity may be advantageous with respect to the material durability [17].

It is observed that the RTFOT aging decreased the bitumen stiffness for sample B + 2% CNSL for intermediate and low temperatures (intermediate to high frequencies) comparative to sample B (Figure 6a). These results are not in agreement with other studies that pointed that the consequences of aging are less significant for intermediate and low temperatures [11,17]. CNSL probably acts as an aging retardant. The significant decrease in stiffness for B + 2% CNSL sample at low temperatures can be also due to physical or chemical interaction between CNSL and bitumen/polar groups causing resistance to age hardening.

It was not observed significant difference in the behavior of bitumen samples analyzed after RTFOT (Figure 6b). This indicates that for high frequencies (low temperatures), there is a slight increase in the curve for sample B + 2% CNSL. This fact is due to the material lower stability at high frequencies (low temperatures) [12,20]. B and B + 2% CNSL samples were both classified as PG 70-28.

2.5 Aggregates' Chemical Characterization

The aggregates were submitted to chemical characterization using X-ray fluorescence (Table 4). Results are consistent with the nature of the aggregates due to the high silicon (Si) content. Researchers have observed that the asphalts containing granitic aggregates had a higher susceptibility to moisture damage [21,22]. High Si content makes the aggregate more acidic requiring a basic component to increase the adhesion at the bitumen/aggregates interface. Acid aggregates, such as granite, have high Si content and negatively affect the development and maintenance of the adhesive bonds with bitumen. Aggregates of basic nature, such as basalt, have low Si content, and tend to develop stronger adhesive bonds with bitumen.

Table 4 - Aggregates' chemical characterization

Chemical Element	Content (%)
Silicon (Si)	61.90
Potassium (K)	18.00
Aluminum (Al)	12.14
Calcium (Ca)	5.32
Iron (Fe)	2.59
Rubidium (Rb)	0.05

2.6 Digital Image Processing (DIP)

It was used a DIP technique to visually classify the asphalts with respect to stripping after the conditioning used for the modified *Lottman* test. A total of five images, for each asphalt, were taken. The pictures were cropped to eliminate the area without particles and then received a treatment to be transformed to black and white scale. In order to calculate the area of each color, the ImageTool software was used. The color of each pixel in the image was identified and provides the percentage of black (without stripping) and white (with stripping) areas pixels. Sample B had a higher percentage of white area (16.3%) (Figure 7). It means that this material had less stripping resistance compared to others asphalts. Sample B + 2% CNSL had a higher black area (91.4%). A higher bitumen coverage on the aggregates' suggests that the CNSL can increased the asphalt stripping resistance.

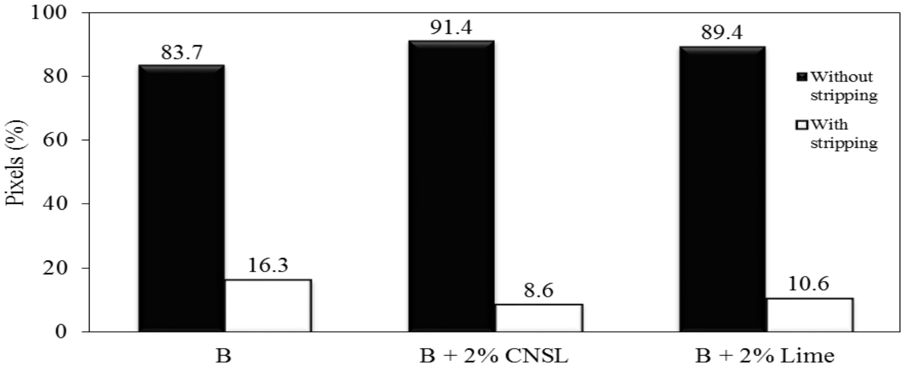


Figure 7 – Asphalts specimens DIP

2.7 Asphalt Modified *Lottman* Test

The modified *Lottman* test was performed on specimens with 7±1.0% air void content [37]. Five specimens were selected as unconditioned. Other five specimens were saturated with water (55-80% saturation level) followed by a freeze cycle (-18°C for 16h) and having a warmer-water soaking cycle (60°C water bath for 24h). These specimens were then tested using the indirect tensile strength (ITS) test at 25°C. The moisture susceptibility for the compacted specimens was evaluated using the tensile strength ratio (TSR):

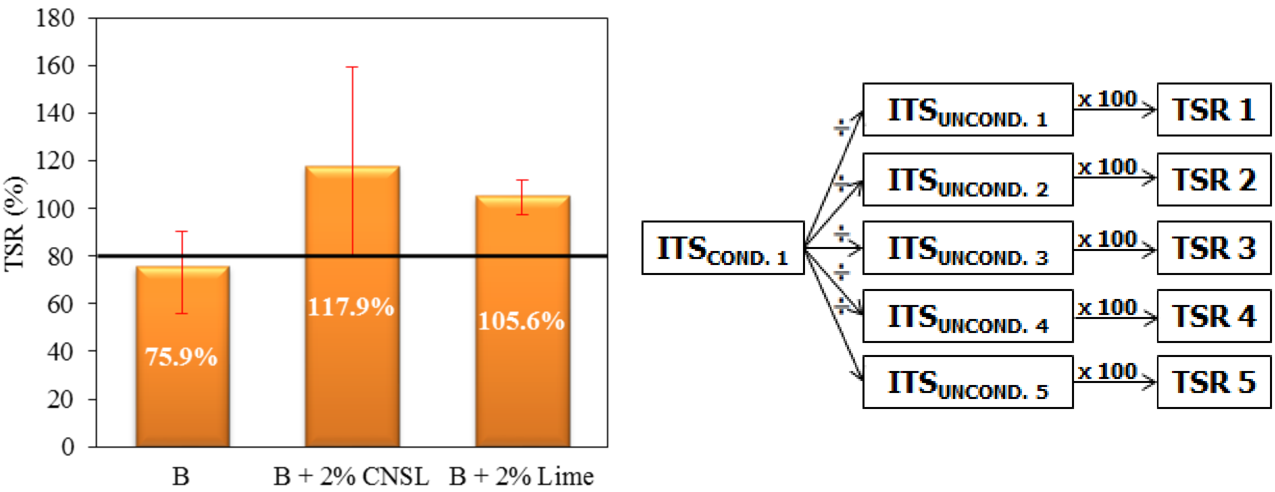
$$TSR = \frac{ITS_{cond}}{ITS_{uncond}} \times 100 \tag{3}$$

Where $ITS_{uncond.}$ and $ITS_{cond.}$ are the average ITS for the unconditioned and conditioned specimens, respectively.
Due the results variability, it was also determined a variation range between conditioned and unconditioned samples. It has been obtained the ITS range. The results are presented in Table 5 and Figure 8. The horizontal line indicates the limit of TSR = 80% adopted in this study [23;24].

Table 5- Modified *Lottman* test results

Asphalt	Bitumen Content (%)	Air Voids (%)	Indirect Tensile Strenght (ITS)						TSR (%)	TSR Variation Range (%)
			Unconditioned			Conditioned				
			Average (MPa)	SD*	CV (%)	Average (MPa)	SD	CV (%)		
B	6.0	7	0.66	0.05	8	0.50	0.06	11	75.5	55.7 - 90.2
B + 2% CNSL	6.0	7	0.65	0.05	8	0.76	0.06	9	117.6	79.4 - 159.4
B + 2% Lime	5.5	7	0.57	0.02	4	0.61	0.01	2	105.6	97.2 - 111.8

* Standard Deviation



(a) (b)

Figure 8- Modified *Lottman* test results

Only the asphalt with the unmodified bitumen was considered unacceptable (TSR lower than 80%). Asphalt B + 2% CNSL had the higher TSR. This asphalt showed TSR value approximately 42% higher compared to the asphalt B and 12% higher than asphalt B + 2% Lime. This is possibly due to the CNSL surfactant properties that can provide a better compatibility between bitumen and aggregates [25].

Asphalts B + 2% CNSL and B + 2% Lime presented TSR values higher than 100%. Some researchers also observed this [2, 22, 29]. This is possibly associated with ITS test variability and with asphalt parameters variation such as air void content (7±1.0%) and saturation level (55-80%) [27]. Water effect is dependent on the sample internal structure (degree of connectivity and air voids size) [27]. These factors may have facilitated the water contact in the aggregate/bitumen interface as well as the displacement of the bitumen film in the investigated asphalts. Asphalt B + 2% CNSL had a higher TSR whereas B + 2% Lime had the lowest TSR variation range. The variation range shows the degree of empiricism of this test [27].

2.8 Resilient Modulus (RM) Test

The RM test was performed according to [28] for specimens with 4% air void content. Similarly to the conditioning process applied in the modified *Lottman* test, five specimens were selected as unconditioned. Another five specimens were selected to be conditioned. All specimens were tested at 25°C and the RM ratio (RMR) was calculated:

$$RMR = \frac{RM_{cond}}{RM_{uncond}} \times 100 \qquad (4)$$

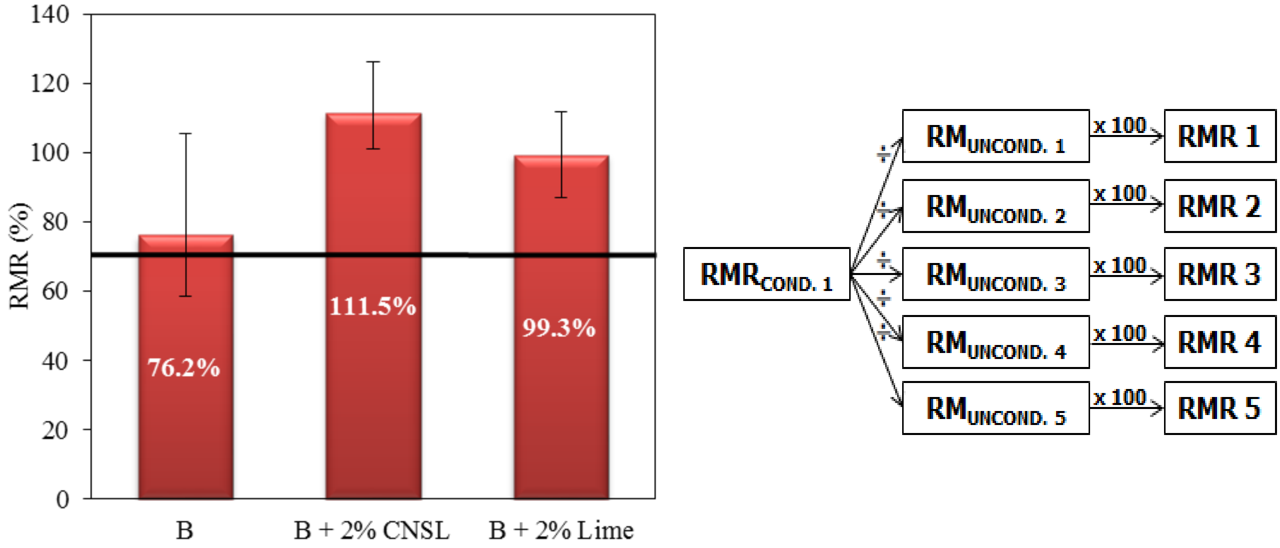
Where $RM_{uncond.}$ and $RM_{cond.}$ are the average RM for the unconditioned and the conditioned specimens, respectively.

It was also determined a variation range between conditioned and unconditioned samples. The results are presented in Table 6 and Figure 9. The horizontal line indicates the limit of RMR = 70% adopted in this study [22, 29, 30].

Table 6 – Resilient modulus (RM) test results

Asphalt	Bitumen Content (%)	Air Voids (%)	Resilient Modulus (RM)						RMR (%)	RMR Variation Range (%)
			Unconditioned			Conditioned				
			Average (MPa)	SD	CV (%)	Average (MPa)	SD	CV (%)		
B	6.0	7	2,829	327	12	2,157	222	10	76.2	58.4 - 105.5
B + 2% CNSL	6.0	7	3,090	125	4	3,446	160	5	111.5	101.1 - 126.1
B + 2% Lime	5.5	7	3,758	109	3	3,731	320	9	99.3	87.0 - 111.8

All asphalts analyzed are considered acceptable (RMR higher than 70%) (Table 6 and Figure 9). Asphalt B + 2% CNSL performed better. It had a higher RMR value approximately 35% higher compared to the asphalt B and 12% higher compared to the asphalt B + 2% Lime. The B + 2% CNSL presented higher RM values for the conditioned compared to RM values for the unconditioned subset. This fact was also observed by others researches [2, 29, 30]. Possibly this phenomenon is associated with the RM test variability and, as this test is performed at low stress levels, the parameter may have not been affected by the presence of water. Asphalts B + 2% CNSL and B + 2% Lime showed RMR variation ranges nearly the same: 24.8% and 25.0%, respectively, whereas asphalt B had the highest RMR variation range (47.1%).



(a) (b)

Figure 9 - RM results for the asphalts analyzed

3. CONCLUSIONS

CNSL was evaluated as a bitumen modifier with respect to resistance to moisture damage. CNSL has shown promise as a bitumen additive decreasing the mixing and compaction temperatures and bitumen viscosity. In respect to complex modulus master curves, bitumen B + 2% CNSL non-aged presented an increase in the stiffness up to intermediate temperatures. For low temperatures, the behavior remained the same compared to non-aged bitumen B. After the RTFOT aging, the curves were overlapped in most of the temperature spectrum, with small differences at low temperatures for the bitumen B + 2% CNSL compared to bitumen B. There were not significant changes in the PG of the analyzed bitumen samples and both were classified as PG 70-28. After aging, it was verified overlapping curves in most of the measured temperature spectrum, with small differences at low temperatures for the bitumen B + 2% CNSL compared to B.

The fluorescence X-ray test confirmed the aggregates acid composition due to the high silicon content. DPI conducted for the asphalt samples after the ITS tests showed that asphalt B + 2% CNSL had a greater stripping resistance.

For the modified *Lottman* test, only asphalt B was not considered acceptable. The B + 2% CNSL showed the higher TSR. This fact may have been caused by CNSL surfactant properties that may provide greater chemical compatibility between modified bitumen and aggregates. For the MR test, the analyzed asphalts were considered acceptable. The asphalt B + 2% CNSL showed higher RMR values.

The mechanical results showed that asphalt B + 2% CNSL presented better performance compared to asphalts B and B + 2% Lime. The CNSL advantage is the fact that this material is natural and abundant in Brazil in relation to other synthetic additives with considerable cost. It is expected that this additive can be used in asphalts in order to improve the affinity between aggregates and bitumen, to provide better adhesion and greater resistance to breakdown, and to generate asphalts lower susceptible to the detrimental water effect.

4. ACKNOWLEDGEMENTS

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