



Rheological behavior of asphalt binders modified with fourth generation rubber asphalts (pellets)

Comportamento reológico de ligantes asfálticos modificados com asfaltos-borracha de quarta geração (pellets)

Ivana Gomes Maia¹, Laura Maria Goretti da Motta², Leni Figueiredo Mathias Leite², Roberto Quental Coutinho¹

¹Federal University of Pernambuco, Recife, Pernambuco – Brazil

²Federal University of Rio de Janeiro, Rio de Janeiro, Rio de Janeiro – Brazil

Contato: ivana.maia@ufpe.br,  (IGM); laura@coc.ufrj.br,  (LMGM); lenimathias@coc.ufrj.br (LFML); roberto.coutinho@ufpe.br (RQC)

Submitted:

28 August, 2021

Accepted for publication:

9 January, 2023

Published:

20 June, 2023

Editor in charge:

Jorge Barbosa Soares

Keywords:

Asphalt rubber pellets.
Sustainability.
Laboratory evaluation.
Rheology.

Palavras-chaves:

Pellets de asfalto-borracha.
Sustentabilidade.
Ensaios.
Reologia.

DOI: 10.58922/transportes.v31i2.2692

ABSTRACT

Among the materials used as asphalt binder modifiers, waste tire granulated rubber has shown good results in terms of improving resistance to permanent deformation, fatigue cracking and aging. The 4th generation rubber-asphalts are sold in the form of pellets. This configuration facilitates the process of mixing the pellets with binder and mineral aggregates, making the process of obtaining rubber-asphalt faster and sustainable technology with low pollutant emissions. The 4th generation of asphalt-rubber is related to the production of mixtures and not asphalt binders. However, the purpose of this study was to evaluate the asphalt-rubber behavior in the laboratory, using the criteria and tests required by the ASTM 8239/21 specification, in addition to fatigue resistance tests (LAS) and storage stability. A comparison was made between the rheological properties of a sample of commercial asphalt-rubber AB-08 and the properties of two 4th generation modifiers (PelletPAVE-Plus and PelletPAV) with CAP 50/70 in proportions of 20% and 30%, with respect to the binder's total mass. The results obtained showed that the properties of the modified binders are superior to those of conventional asphalt, but inferior to those of conventional AB-08 asphalt rubber.

RESUMO

Entre materiais empregados como modificadores de ligantes asfálticos, a borracha granulada de pneu inservível tem apresentado bons resultados em termos de melhoria da resistência à deformação permanente, às trincas por fadiga e ao envelhecimento. Os asfaltos-borracha de 4^a geração são comercializados em forma de pellets. Esta configuração facilita o processo de mistura dos pellets com ligante e agregados minerais, tornando o processo de obtenção do asfalto-borracha mais rápido e sustentável com baixa emissão de poluentes. A 4^a geração de asfalto-borracha se relaciona à produção de misturas e não de ligantes asfálticos. No entanto, este trabalho teve por objetivo avaliar o comportamento dos ligantes asfaltos-borracha em laboratório, utilizando os critérios e ensaios requeridos pela especificação ASTM 8239/21, além de ensaios de resistência à fadiga (LAS) e estabilidade à estocagem. Foi realizada uma comparação entre as propriedades reológicas de uma amostra de asfalto-borracha comercial AB-08 e as propriedades de dois modificadores de 4^a geração (PelletPAVE-Plus e PelletPAV) com CAP 50/70 nas proporções de 20% e 30%, com relação à massa total do ligante. Os resultados mostraram que as propriedades dos ligantes com pellets são superiores às do CAP, porém inferiores às do asfalto-borracha convencional AB-08.



1. INTRODUCTION

A pavement structure must be able to meet the requirements of comfort, safety, savings, climate change and traffic loads. In order to prevent faults through fatigue and permanent deformation, the thickness of the layers can be increased, or the conventional asphalt mix substituted by a higher quality, reducing the stresses and deformations impacting on the structure.

In Brazil, since 1990, the Petrobras Research Center (Cenpes) and some universities began examining the behavior of waste tire rubber as an asphalt binder modifier (Fontes, 2009). Leite (1999) reported that in order to increase resistance against aging, fatigue and permanent deformation, different polymers or granulated tire rubber were added to the petroleum asphalt cement (PAC). The granulated tire rubber is used as one of the durable versatile modifiers compared to the conventional binder. Asphalt rubber performs better against fatigue and permanent deformation, as demonstrated by Leite (1999), Oda (2000), Bertollo (2002), Specht (2004), Dantas Neto (2004), Lima (2008), Camargo (2016) and Nunes (2017).

The new generation of asphalt rubber (4th generation) is obtained by adding tire rubber dust in pellet form to a conventional asphalt binder. The US 2010/0056669 patent mentions that the pellets consist of asphalt cement, ground tire rubber (15%-30% in weight), fines from hydrated lime, mineral rocks, waxes and (Bailey, 2010). Another US Phoenix Industries patent, US 8404164/2013 also addresses pelletized asphalt rubber (Sockwell, 2013).

Literature on the use of asphalt rubber pellets shows that tire rubber is a sustainable alternative to maintain the cold stored product, without requiring stirred tanks and extra equipment (Nanjegowda and Biligiri, 2020).

The presence of solid components (fillers) in these pellets hardens the material making it non-sticky. However, they may harm the quality and not reproduce the positive effects of the tire rubber as an asphalt binder modifier. The benefits regarding the sustainability of the process, such as no need for hot storage and mixing equipment, may not be worth it, if the product lacks suitable properties. Greca Asfaltos (2021) states that 13,000 km of asphalt rubber was applied to Brazilian pavements over the last 20 years. Several asphalt distributors produce asphalt rubber, and Ecorodovias is the concessionaire that most deploys asphalt rubber in repair work.

The use of asphalt rubber extends from South, Southeast to Northern Brazil, without the risk of separating phases in the tank truck, despite long distances to the jobsite (Ecorodovias, 2021).

This study compares the rheological properties of the asphalt rubber AB-08 binder with the asphalt-rubber resulting from CAP 50/70 mixtures with two types of pellets produced by Phoenix Industries, called PelletPAVE Plus and PelletPAV. The pellets were added to the 20% and 30% proportions (in relation to the total binder mass). The samples of the four pellet-modified binders and the AB-08 sample were tested according to the ASTM 8239/21 (ASTM, 2021) specifications and endurance test by linear amplitude scanning (LAS). It is expected that comparing the performance-related results regarding endurance and permanent deformation could indicate the characteristics of the tire rubber pellets in the pavement.

2. BIBLIOGRAPHIC REVIEW

The physical requirements of the tire-rubber modified binders are standardized in the USA by ASTM D 6114/19 (ASTM, 2019), which establishes three types of parameters (I, II and III) for apparent viscosity at 175 °C in a rotational viscosimeter; penetration at 25 °C and 4 °C; softening point; resilience; flashpoint and penetration retained at 4 °C after aging in the fine-film oven. In Brazil, the ANP Technical Regulation no. 39 dated 2008 provides asphalt-rubber specifications in Brazilian territory. In 2009 DNIT standardized wet asphalt rubber in the DNIT Specification 111/2009-EM (DNIT, 2009).

Two processes exist to add tire rubber to the asphalt mixes: wet and dry. In the dry method, the rubber granules are added to the plant as part of the aggregates (1st generation). In the wet process, the binder may be modified in the asphalt concrete plant immediately before releasing the aggregates into the mixer (continuous blend – 2nd generation), or right after production of the base binder in the refinery (terminal blend - 3rd generation).

Bernucci, Motta and Ceratti (2022) believe the non-storable system must be applied immediately after production, due to its instability, showing different characteristics from the asphalt carried in tanks with stirring and recirculation. However, although they facilitate the asphalt-rubber application, the terminal blend mixtures might possibly show a poorer mechanical performance than the continuous blend mixtures (Shatnawi, 2011).

Considering the challenges in using asphalt rubber obtained from the wet process in plant, the storable asphalt rubber produced by the asphalt distributors in Brazil is commonly used.

The benefits of asphalt rubber are as follows: crack reduction, aging resistance; thinner cladding in relation to conventional binders; good resistance to skidding and permanent deformation, cutting maintenance costs and using discarded tires (Caltrans, 2003; Way et al., 2012). The restrictions include higher costs, more limited temperature requirements, higher binder content, odor and air quality problems (Lo Presti, 2013; NDOT, 2016).

The 4th generation asphalt rubber or pellets are marketed as small pellets for easier application. They are an option to generate terminal blend or continuous blend binders, when requiring elevated rubber content and high viscosity for special requirements.

In the “pelletized” process, the tire rubber, polymers and additives are added to the base binder before the pelletization process. The resulting material is bagged and stored dry at room temperature until it is used.

The pellets supplied by Phoenix Industries are produced from the mixture of ground tire rubber (15%-25% in the mixture) with asphalt cement at 175 °C et al., Kim (2014) reports that in the reactor’s outlet there is a pelletizer attached to a cooler, with a filler feed to form the pellets. Amirghanian, Xiao and Sockwell (2015) confirm this production process, stating that the filler is hydrated lime, and after pellet formation they are coated to prevent coagulation. The pellets are used in continuous mixtures, added directly to the binder and or aggregates and immediately applied, not requiring storage stability.

There are only a few records of rheological studies using pellet-modifying binders. However, many authors ratify that the tire-rubber modification of the asphalt improves the anti-fatigue and permanent deformation performance (Airey, 1997; Dantas Neto, 2004; Specht, 2004; Camargo, 2016; Nunes 2017; Wang et al., 2018; Klinsky et al., 2020).

Amirkhanian, Xiao and Sockwell (2015), Nunes (2017), and Amirkhanian (2018) suggest that pellets may be used successfully in open mixtures, substituting the terminal blending asphalt rubber.

Laboratory tests using a traffic simulator in mixtures dosed by the Marshall method showed that a porous friction mixture with 2% pellets demonstrated good fracture energy and excellent drainage capacity in 160 mL/min of heavy rainfall (Song et al., 2022).

In Kuwait, several analyses were performed to optimize the asphalt mixture dosage by the Marshall method or gyratory compaction (Zoorob, Al-Bahar and Al-Otaibi, 2018; Al-Baghli, 2020; 2022; Alkheder, 2021). Experimental stretches were created with different sections to compare the performance of mixtures in restored pavements with low to medium traffic (Al-Baghli, Awadh and Zoorob, 2022).

On a national level, pellets were applied in different conservation operations by the Nova Dutra concessionaire on the BR-116 highway, and by Arteris concessionaire on the BR-381 highway since 2013.

3. MATERIALS & METHODS

3.1. Materials

Four types of asphalt binders were used in this study:

- Conventional CAP 50/70, from Reduc;
- Conventional asphalt rubber, AB-08, produced by Stratura;
- Asphalt rubber pellets: “PelletPAVE-Plus” (PPP) and “PelletPAV” (PP), supplied by Phoenix Industries/USA, used in a continuous wet process (Figure 1).



Figure 1. Asphalt rubber pellet samples used in this study - PelletPAVE-Plus (left) and PelletPAV (right)

According to the manufacturer, the PP is used in gap-graded mixtures at a ratio of 20%-40% (in total binder weight), meeting the rubber-modified binder ASTM D 6114/19 (ASTM, 2019) requirements for viscosity, penetration, softening point and resilience.

The PPP is a hybrid asphalt rubber, namely, containing an SBS copolymer in its formulation, and also 12%-15% of a fine rubber powder, for use in dense asphalt mixtures.

The modified binders used in the study herein were named as follows:

1. CAP 50/70 + PelletPAVE-Plus with 20% added in relation to the binder's total mass: CAP 50/70 + 20% PPP;
2. CAP 50/70 + PelletPAVE-Plus with 30% added in relation to the binder's total mass: CAP 50/70 + 30% PPP;
3. CAP 50/70 + PelletPAV with 20% added in relation to the binder's total mass: CAP 50/70 + 20% PP and
4. CAP 50/70 + PelletPAV with 30% added in relation to the binder's total mass: CAP 50/70 + 30% PP.

The modified binders were prepared from CAP 50/70 by incorporating pellets at 20% and 30% ratios to the binder's total mass, as recommended by the manufacturer and analyzed by the ASTM D 8239/21 (ASTM, 2021) standard and by the LAS test.

In relation to the grading range of the rubber used in the pellet composition, the PP consists of rubber particles matching the grade in Table 1. The difference between the PP and PPP is the percentage of fines, higher in the PPP, but not available in this grade.

Table 1: Grade characteristics of tire rubber used in asphalt rubber pellets

Screen size	Tire rubber PP (%)
N°8	100
N°10	90-100
N°16	60-85
N°30	15-40
N°50	5-15
N°100	0-3
N°200	0-1

3.2. Methodology

The Superpave ASTM 8239/21 (ASTM, 2021) specification is based on rheological properties, climate conditions and traffic volume in order to select the asphalt binder. It is based on tests that simulate different binder aging stages: short term related to the milling and application of the asphalt mixture (rolling thin-film oven test, RTFOT) and long-term working life (pressure aging vessel-PAV).

Tests to determine the degree of performance at a high, intermediary and low temperature, in this US specification, carried out on the dynamic shear rheometer (DSR), ensure the resistance to permanent deformation and fatigue and in the bending beam rheometer (BBR), to heat checking. There also is the rotational viscosity test (ASTM D4402) (ASTM, 2015a), associated with the milling and compaction temperatures of the asphalt mixture. And lastly, the test to determine the permanent deformation resistance [Multiple Stress Creep and Recovery-MSCR; AASHTO M332-21 (AASHTO, 2021); ASTM 7405-20 (ASTM, 2020); DNIT 423/2020-ME (DNIT, 2020)].

The ASTM 8239/21 (ASTM, 2021) specification does not yet consider the linear amplitude sweep test (LAS) for fatigue resistance, AASHTO T 391-20 (AASHTO, 2020). However, this test has been used in Brazil since 2014 (Martins, 2014; Nascimento, 2015; Specht et al., 2019). Petrobras has an LAS database showing a correlation of the test on the binder with the performance of asphalt mixtures in the direct tensile test and with the

cracked field area. The National Department of Highways (DNIT) published standard DNIT 439/2022–ME (DNIT, 2022), adapted from AASHTO T 391-20 (AASHTO, 2020) with regard to the defining time criterion when damage occurs. In the Brazilian version, the damage occurs when there is maximum pseudo-energy (Chen et al., 2021).

Figure 2 provides the flowchart of this study. High temperature PG, MSCR and LAS tests were performed on dynamic shear rheometer (DSR) equipment, in order to predict the samples' rheological behavior. Penetration, softening point and apparent viscosity tests under the asphalt specification (DNIT 111/2009-EM) (DNIT, 2009) and storage stability (NBR 15086).

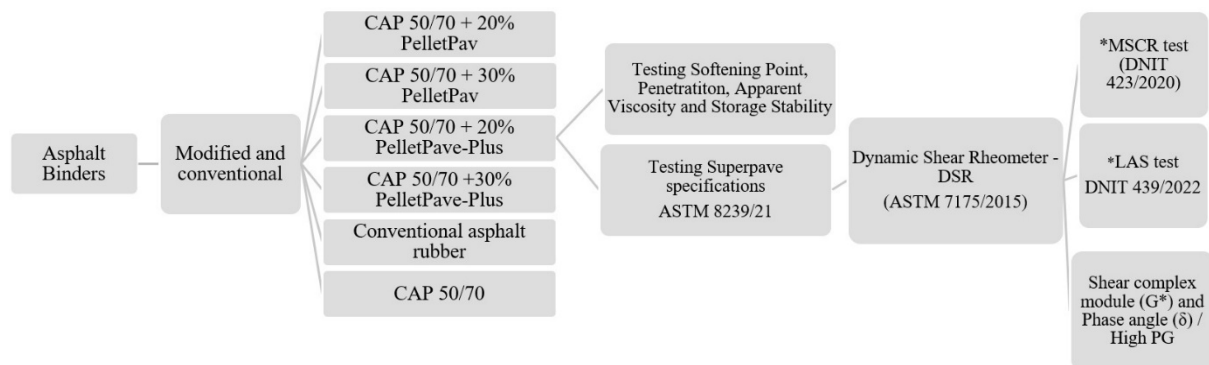


Figure 2. General flowchart of the tests on which this study was based

In this study herein the modified binders were tested in virgin condition and aged for a short time in rolling thin-film oven tests (RTFOT) (NBR 15235/2009 – ABNT, 2009) in duplicate, measuring the following parameters:

- $G^*/\text{sen } \delta$ before RTFOT at 58 °C, 64 °C, 70 °C, 76 °C and 82 °C;
- $G^*/\text{sen } \delta$ after RTFOT at 58 °C, 64 °C, 70 °C, 76 °C and 82 °C after RTFOT;
- J_{nr} after RTFOT at 70 °C;
- LAS after RTFOT at 19 °C
- Softening point (SP);
- Penetration at 25 °C, 100 g, 5 s;
- Apparent viscosity at 175 °C.

3.2.1. Performance Grading (PG ASTM 7175; ASTM, 2015b)

To obtain the binder PG, it is necessary to determine the complex shear module (G^*) and the phase angle (δ) on a virgin and aged sample in the RTFOT (ASTM D 8239-21) (ASTM, 2021). They are obtained in the DSR by applying shear stresses oscillating to the frequency of 10 rad/s in a sample set between two parallel plates, at the initial temperature of 58 °C and increasing intervals of 6 °C (ASTM D 7175/15) (ASTM, 2015b).

The performance grading PG is based on properties of binders related to the pavement temperatures and traffic volume.

In the PG classification the binder must meet the limits proposed at a certain temperature, compared with the climate conditions of the site to be paved (Leite, 1999). The classes are grouped by the pavement's expected maximum and minimum (PG). For

the study herein no check was made for the low temperature that in the PG are negative, considering the major part of the territory has a tropical climate.

3.2.2. Multiple Stress Creep & Recovery - MSCR [ASTM 7405-20 (ASTM, 2020); AASHTO M332-21 (AASHTO, 2021); and DNIT 423/2020-ME (DNIT, 2020)]

The DSR parameter for permanent deformation ($G^*/\text{sen } \delta$) was replaced by the non-recoverable creep compliance (J_{nr}), obtained from the multiple stress creep and recovery test (MSCR), by virtue of determining the former in the linear viscoelasticity range, incompatible with the reality in the field (Martins, 2014).

The MSCR test was performed for the binders' permanent deformation resistance, on the rheometer with parallel plate geometry, 25 mm in diameter, between 1 mm plate in samples aged in the RTFOT for a high temperature of PG classification. There are 20 creep and recovery cycles for a stress of 100 Pa, the first 10 cycles for conditioning the sample and the last 10 for data analysis. After the 20 creep and recovery cycles for the stress of 100 Pa, 10 creep and recovery cycles were carried out for a stress of 3200 Pa.

3.2.3. Linear Amplitude Sweep - LAS [DNIT 439/2022; DNIT (2022)]

This test assesses the asphalt fatigue resistance by cyclic loading using a linear increase of the amplitude load. The amplitude sweep is performed in the DSR at a 19 °C intermediate temperature of the pavement, representing the fatigue damage rather than the parameter expressed by $G^*/\text{sen } \delta$ (Pamplona, 2013; Martins, 2014). The binder is tested after short-term aging.

The LAS test procedure is split into two stages. The first is a frequency sweep for viscoelastic properties in the linear region, determining the parameters α and m that assess the undamaged material. The second stage using an oscillating shear in the 10 Hz frequency. The loading scheme consists of the continuous oscillating deformation sweep, linearly increasing from 0.1% to 30%, up to 3100 loading cycles. The shear stress and deformation peaks are recorded every 10 load cycles (one second) together with a dynamic shear module ($|G^*|$) and phase angle (δ).

Five criteria exist to determine the number of cycles until damage: 35% reduction in $|G^*|/\text{sen } \delta$; maximum stress; peak at $C \times N$; peak at the phase angle and maximum pseudo-energy. The ability to separate modified from unmodified binders currently uses the criterion of maximum pseudo-energy (W_S^R maximum).

The LAS test estimates the time when the damage occurs with the criterion relating to the maximum pseudo-energy and then the binder fatigue factor (BFF). The results are analyzed using the principle of viscoelastic continuous damage (VECD).

Adopting the maximum pseudo-energy criterion determines the value (W_S^R), and confirms the time corresponding to C_f , calculating in a worksheet the constants C_1 , C_2 , A and B and final number of cycles, F_n .

For asphalt mixtures, the mixture fatigue factor (MFF) is determined in the direct tensile test, referring to the area between 100 and 200 $\mu\epsilon$ deformations and corresponding cycles. The MFF presents a good correlation with the cracked area of

asphalt coatings. It was found that there is a correlation between MFF and BFF, defined as the area between 1.25% and 2.5% deformation, in the graph N versus deformation, binder fatigue factor at 19 °C (BFF_{19°C}), calculated according to Equation 1 below:

$$\text{BFF} = \frac{(\log(N_{f,1.25\%}) + \log(N_{f,2.5\%}))}{2} \times (\log(0.025) - \log(0.0125)) \quad (1)$$

3.3. Adding process of pellets to asphalt binders

To include pellets in CAP 50/70 a paddle mixer was used for stirring in the heating chamber. The temperature recommended by the manufacturer for mixing is between 165 °C and 175 °C. Figure 3 shows the mixing equipment, prepared samples and DSR equipment for the MSCR and LAS tests.

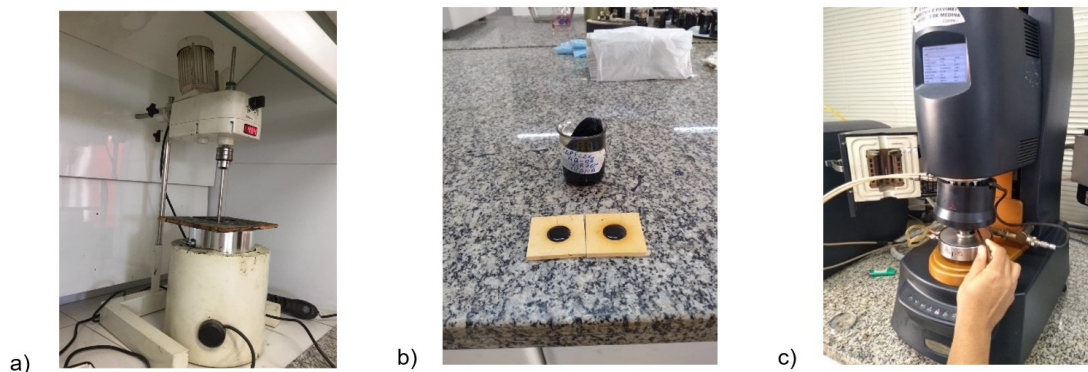


Figure 3. (a) Low shear stirring mixer with propeller, (b) 25 mm samples prepared and (c) dynamic shear rheometer

In order to modify CAP with pellets, CAP 50/70 was heated at 170 °C for one hour and then each pellet sample was added. For the 20% pellet proportion, 1600 g of CAP was added to the gallon mixer, placed inside the heating chamber, fitted with an agitator and gradually adding the cold pellets (400 g) to guarantee good homogenization, stirring for half an hour. For the 30% pellet proportion, 1400 g of CAP and 600 g of pellets were added to the gallon. In both cases an aliquot was taken from a beaker (500 g) to perform the tests.

4. RESULTS ANALYSIS

4.1. Physical characterization

Table 2 shows the results of softening point, penetration and apparent viscosity for the studied samples, to compare with the limits of the asphalt rubber specifications in ASTM D 6114/19 (ASTM, 2019). It was found that the modification of the binder with 30% pellets was below the penetration limit of 25 (1/10 mm), while the 20% showed unsatisfactory softening point values below 54 °C. The viscosity values of all modified samples are below the required limits. In relation to the storage stability test, the modified sample with 20% pellets gave satisfactory results.

Table 2: Results of the physical characterization and storage tests of the binders used in this study

Characteristics	CAP 50/70	AB-08	50/70 + 20% PP	50/70 + 30% PP	50/70 + 20% PPP	50/70 + 30% PPP	ASTM D6114 (TYPE II)
Softening point °C	48	60	52	54	50	56	Min. 54
Penetration at 25 °C, 100 g, 5 s, (1/10mm)	-	27	-	21	-	22	Min. 25
Rolling viscosity, 177 °C (cP)	71	1117	-	1017	-	817	Min. 1500
Storage test °C			Top 50/50 Bottom 53/54	Top 52/52 Bottom 60/60	Top 50/51 Bottom 54/54	Top 54/54 Bottom 63/63	

4.2. Rheological characterization

4.2.1. Determining high temperature PG

The DSR test was performed on virgin and aged samples after RTFOT for the six binders examined in this study and the results are summarized in Table 3. The high temperature of PG was defined as that in which the $G^*/\sin(\delta)$ values are higher than 1.0 kPa and 2.2 kPa, for the samples before and after aging, respectively.

Table 3: $G^*/\sin(\delta)$ in function of the test temperature for virgin and aged samples in RTFOT

Test temperature (°C)	Binder	Virgin Sample			Aged Sample			PG grade (°C- high temperature)
		Complex module G^* (MPa)	Phase angle δ (°)	$G^*/\sin \delta$ (kPa)	Complex module G^* (MPa)	Phase angle δ (°)	$G^*/\sin \delta$ (kPa)	
58	CAP	1.90E-03	88.71	2.34	4.26E-03	87.11	4.26	58
64	50/70	8.00E-04	89.18	0.88	1.80E-03	88.15	1.83	
70		4.00E-04	89.43	0.40	8.00E-04	88.83	0.83	
70	AB-08	4.13E+03	71.70	4.41	8.30E+03	72.00	8.74	82
76		2.21E+03	69.53	2.46	4.48E+03	72.04	4.71	
82		1.35E+03	65.28	1.67	2.41E+03	75.57	2.49	
70	CAP	1.35E+03	75.32	1.46	2.60E+03	79.00	2.72	70
76	50/70 +	7.97E+02	72.60	0.86	1.35E+03	79.90	1.38	
82	20% PPP	4.97E+02	67.53	0.58	7.50E+02	79.49	0.76	
70	CAP	1.08E+03	86.64	1.08	3.70E+03	78.29	3.78	70
76	50/70 +	5.44E+02	87.43	0.55	1.85E+03	79.83	1.91	
82	30% PPP	2.90E+02	81.92	0.29	1.02E+03	80.47	1.05	
64	CAP	3.01E-03	64.70	3.57				64
70	50/70 +	7.29E-04	84.99	0.73	2.49E-03	81.35	2.52	
76	20% PP	3.80E-04	83.52	0.38	1.25E-03	82.87	1.26	
82		2.15E-04	82.81	0.22	6.75E-04	83.13	0.69	
70	CAP	1.39E-03	83.98	1.40	6.76E-03	62.90	7.64	70
76	50/70 +	7.36E-04	81.16	0.75	3.94E-03	60.09	4.62	
82	30% PP	4.28E-04	78.34	0.44	2.57E-03	54.42	3.26	

For CAP 50/70, high temperature PG grade was 58 °C. The parameter referring to permanent deformation resistance ($G^*/\sin(\delta)$) is greater for the aged samples, ratifying that the aging process hardens the material.

The AB-08 binder showed PG 82 to be very much higher than CAP 50/70, also confirmed by Specht (2004); Domingos (2011); Martins (2014) and Klinsky and Faria (2017), among others.

For CAP 50/70 + 20% PPP there was low repeatability of the results to determine the PG, but we can assume the more conservative figure of PG 70. PG 79 was obtained for CAP 50/70 + 30% PPP, less than AB-08, indicating less resistance to temperatures over 70 °C, common in the Brazilian coatings. This was, however, well above that obtained for CAP 50/70. It is worth mentioning that the low repeatability of the samples modified by pellets may be due to the large size of the rubber particle, since standard ASTM D 7175 (ASTM, 2015b) recommends the method not to be suggested for binders containing particles larger than 250 µm for spacing between rheometer plates. The results are influenced by the particle-to-particle interaction in the space between the plates. Perhaps larger spacing could accommodate the particles better. The largest part of the asphalt rubber (terminal blend) uses maximum size crumb rubber from screen 30. Studies are still in progress with other spacings and other geometry, but not yet complete. The 250 µm limitation for the largest rubber particle is based on the fact that the test requires the maximum particle size to be less than $\frac{1}{4}$ of the spacing (FHWA, 2014; Mturi, O'Connell and Zoorob, 2014; Bennert, 2013).

For CAP 50/70 + 20% PP low repeatability was also found in the tests for parameter $G^*/\sin \delta$. Values higher than 1.0 kPa were found at the temperature of 64 °C. CAP 50/70 + 30% PP showed PG 70 and low aging resistance. The PG was lower than AB-08, but higher than CAP 50/70.

Bearing in mind the shortage of Brazilian data involving binders modified by asphalt rubber pellet, no data are available for a comparative analysis. Since it is a new material, the studies have focused rather on the performance of the asphalt mixtures with pellets than the binder performance.

In this study, PG 70 was predominant in the binder with pellets.

4.2.2. MSCR at 70 °C after RTFOT

In general, the modified binders showed a drop in the non-recoverable compliance for the two stress levels when compared to 50/70, except for the two 50/70 + 20% pellet samples, which presented non-recoverable compliance values under stress of 3.2 kPa, higher than the conventional.

Figure 4 shows the average values of the parameters obtained for non-recoverable compliance and the recovery percentages for the 0.1 kPa and 3.2 kPa stresses.

CAP 50/70 + 30% PP provided good recovery results at the two tensile levels compared to the 50/70. The lower value of the non-recoverable compliance suggests better resistance to permanent deformation. No increase was observed in the other pellet binders in terms of performance parameters in this test. The AB-08 showed great improvement when compared to CAP 50/70, also reported by Subhy, Lo Presti and Airey (2015), Camargo (2016), Klinsky, Bardini and Faria (2020), and others.

Camargo (2016), Nunes (2017), Fengler (2018), Gaspar (2019) and others used 2 mm gaps between DSR plates, to determine high-temperature PG and MSCR with 25 mm plate to minimize the impact of the rubber particles on the results. Corrigan and Golalipour (2016) noted that the larger the spacing, the less the variability, smaller the complex module and

larger the phase angle. They demonstrated that the 1 mm spacing may be too small to accommodate the rubber particles, due to the particle size, concentration and reaction time.

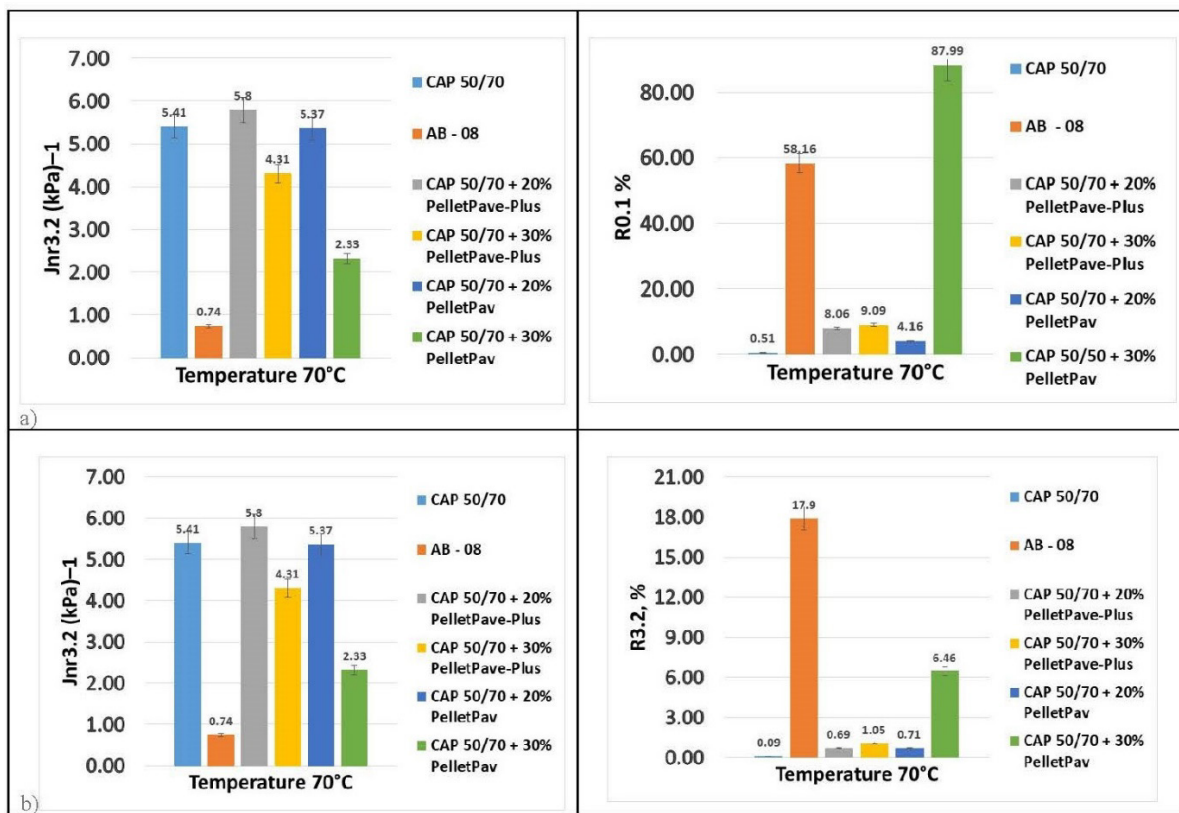


Figure 4. Non-recoverable compliance parameters and recovery percentages for 0.1 kPa and 3.2 kPa stresses after RTFOT at 70°C

Table 1 provides the grading of the rubber particles comprising the pellets under analysis. The PP grading is between 0.6 mm and 2 mm, similar to traditional asphalt rubber. We found that the size of the particles for the gap used in this study (1 mm) could have impacted the results.

Bernucci et al. (2020) reported that the increase in the gap does not seem to inhibit the effect of the asphalt rubber particles when 1 mm and 3 mm gaps were tested. Some rubber-modified binders could be tested correctly with the parallel-plate geometry while others may not. Corrigan and Golalipour (2016) say that problems with parallel-plate geometry involve: edge effect, particle interactions, grading, swelling and percentage.

Several studies address the assessment of the impact of rubber particle size on asphalt rubber properties (Subhy, Lo Presti and Airey (2015), Camargo (2016), Nunes (2017), Fengler (2018), Gaspar (2019), and others). So far there is no standardization for this item (75 µm to 2.36 mm) not distinguishing between small and large. However, Jones, Liang, Harvey (2017) conclude that digestion time, phase angle and fatigue resistance reduce with a smaller size of particle, while storage stability, viscosity, rigidity, permanent deformation resistance increase as the particle size decreases. In addition to particle size, the pellet mixing process in terms of stirring and temperature may not have been sufficient to digest

and swell the rubber, causing the presence of particulate (Bukowski, 2014). Asphalt rubber produced in this way could cause low-performing rheological properties.

4.2.3. LAS (Linear Amplitude Sweep)

The binder classification criteria based on the LAS test evaluate fatigue resistance. Table 4 presents the parameters m , α , C1, C2, Df, coefficients A and B, and BFFPSE, obtained in the LAS test.

Table 4: Coefficients obtained in the LAS test for the binders studied herein

Asphalt binders	Straight slope	Parameter	Regression coefficients		Damage failure	Fatigue curve coefficients		Binder fatigue factor
	m	α	C1	C2	Df	A	B	PSE Method
50/70	0.69	1.45	0.064	0.540	78.0	2.12E+05	-2.89	1.39
AB-08	0.50	2.00	0.140	0.360	197.0	4.32E+07	-4.02	1.98
50/70 + 20% PPP	0.61	1.64	0.080	0.50	78.5	4.50E+05	-3.28	1.46
50/70 + 30% PPP	0.58	1.70	0.084	0.49	80.5	6.73E+05	-3.44	1.5
50/70 + 20% PP	0.61	1.65	0.083	0.50	81.0	5.01E+05	-3.31	1.47
50/70 + 30% PP	0.56	1.80	0.087	0.47	96.8	1.53E+06	-3.60	1.59

Parameter B depends on the value of α , calculated from the straight slope (m). The smaller m values obtained for the modified binders in the study herein suggest that they are less sensitive to the deformation amplitude than the conventional.

For modified binders, an increase is found in parameter A and a decrease in B in relation to 50/70, also observed by Nuñez (2013), Martins (2014), Camargo (2016), Nunes (2017) and Wang et al. (2018). The rise in A and drop in B indicate longer fatigue life for the modified binders. The AB-08 presented the biggest decrease in B, followed by those modified by 30% pellets.

Figure 5a shows the binders' stress curves versus shear deformation of this study. In the early stage, the shear stress increases linearly with the added deformation overall. When they enter the non-linear region, stress peaks are reached at different deformation levels. Shear stress applied to 50/79 increases when deformation amplitude increases. The rubber asphalts have a stress peak in greater deformation amplitudes than the conventional binder, indicating more damage resistance.

The modified binders in this study, except for AB-08, have a higher stress peak than the 50/70 for similar deformation amplitudes. The pellet binders presented a slight improvement in fatigue life compared to the conventional, but the AB-08 had the best behavior. The damage characteristic curve, Figure 5b, shows that the integrity of the materials diminished with increased damage intensity.

Figure 6 shows the fatigue behavior, depending on three deformation levels (1.25%, 2.5% and 5.0%) in the linear viscoelastic range. The binder fatigue factor (BFF) was provided by the PSE method (Cenpes). It can be said that on average conventional asphalt rubber and pellet binders had longer fatigue life values (F_n) compared to CAP 50/70.

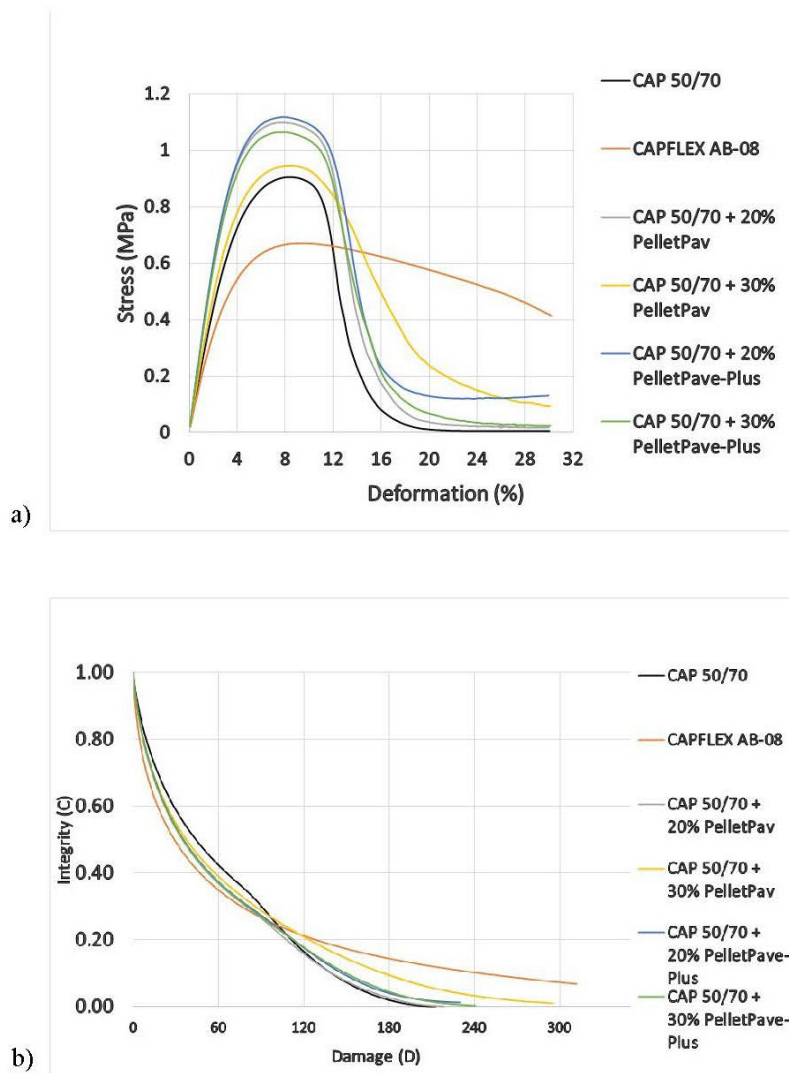


Figure 5. (a) Stress versus deformation curve and (b) Characteristic damage curve in LAS test

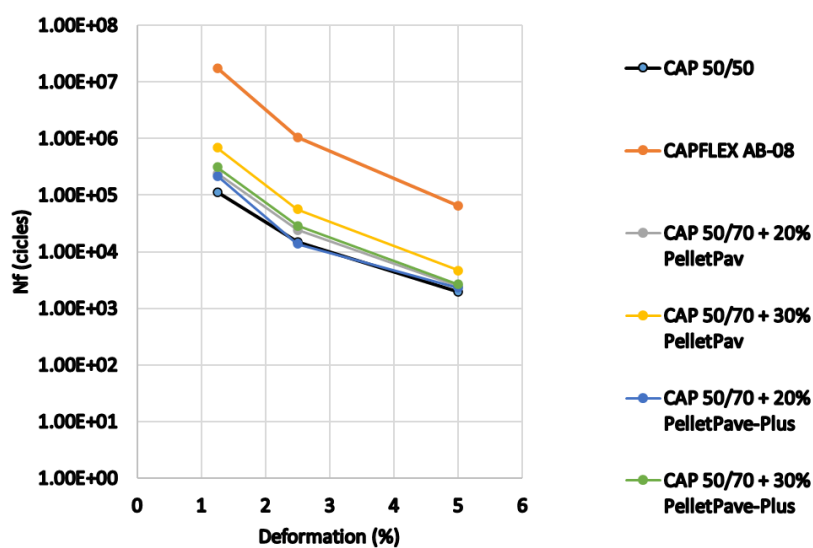


Figure 6. Fatigue life (Fn) according to the shear deformation for the binders studied herein

5. CONCLUSIONS

This study assessed physical and rheological characteristics of a CAP 50/70 modified with added 4th generation asphalt-rubber pellets (PP and PPP). The pellets produced asphalt-rubber PG inferior to AB-08, but superior to the conventional CAP.

Conventional asphalt rubber produced a higher PG 82 than PG 58 of CAP 50/70. For the binders with 20% pellets, the results indicate PG 70 and PG 64 higher than PG of CAP 50/70. The binders with 30% pellets produced PG 70.

The CAP 50/70 samples with 20% pellets presented storage stability and those with 30% pellets did not behave positively to storage, but in DSR indicated PG 70.

With regard to the recovery parameters and non-recoverable compliance in MSCR, there was no major improvement for the pellet binders compared to the conventional binder. Conventional asphalt rubber revealed a higher recovery value under stress and a lower non-recoverable compliance value.

An increase in parameter A and a decrease in parameter B were found in the modified binder LAS test in relation to the conventional binder. This shows a longer fatigue life for modified binders.

It is important to check whether the rubber particle size and pellet additives caused interference in the DSR tests, since the modified binders were compared to the asphalt rubber type storable terminal blending, in which the particle size is much smaller. Discussions have been raised about the use in DSR for asphalt rubber, since there are larger particles than the space allocated to the binder in the plate/plate geometry. Many adopt the concentric cylinder geometry to better assess asphalts with pellets.

The conclusion was that AB-08 performed very well in terms of permanent deformation and fatigue, better than 4th generation rubber asphalts in this study. However, the tests of the pellet binders were not conclusive and further investigation is required for the properties of asphalt mixtures prepared with all binders evaluated herein.

REFERENCES

- AASHTO (2020) *T 391-20: Standard Method of Test for Estimating Fatigue Resistance of Asphalt Binders Using the Linear Amplitude Sweep*. Washington, DC: AASHTO.
- AASHTO (2021) *M 332-21: Standard Specification for Performance-Graded Asphalt Binder Using Multiple Stress Creep Recovery (MSCR) Test*. Washington, DC: AASHTO.
- ABNT (2009) *NBR-15235: Materiais asfálticos - Determinação do Efeito do Calor e do Ar em uma Película Delgada Rotacional*. Rio de Janeiro: ABNT.
- Airey, G.D. (1997) *Rheological Characteristics of Polymer Modified and Aged Bitumens*. Thesis (PhD). University of Nottingham. Nottingham. Available at: <<https://eprints.nottingham.ac.uk/13431/>> (accessed 08/28/2021).
- Al-Baghli, H. (2020) Characterization and design of a crumb rubber modified asphalt mix formulation. *International Journal of Urban and Civil Engineering*, v. 14, n. 2, p. 2020.
- Al-Baghli, H. (2022) Optimizing PelletPAVE™ rubberized asphalt mix design using gyratory compaction and volumetrics. *International Journal of Urban and Civil Engineering*, v. 16, n. 3, p. 2022.
- Al-Baghli, H.; Z.S. Awadh and S.E. Zoorob (2022) Rubberized asphalt pilot road trial in Kuwait. In *Proceedings of the 5th International Conference on Rehabilitation and Maintenance in Civil Engineering ICRME 2021*. Singapore: Springer Nature, p. 117-28. DOI: 10.1007/978-981-16-9348-9_11.
- Alkheder, S. (2021) Pavement performance enhancement using waste rubber: green pavement design for Kuwait -, *International Journal of Environmental Research*, v. 15, n. 4, p. 733-50. DOI: 10.1007/s41742-021-00354-w.

- Amirkhanian, S.N. (2018) *The Use of Pelletized Asphalt Rubber as an Alternative to the Process Blending System*. Arizona: Asphalt Rubbe.
- Amirkhanian, S.N.; F. Xiao and K. Sockwell (2015) Performance properties of polymer modified pelletized asphalt mixtures. In Harvey, J.T. and K.F. Chou (eds.) *Airfield and Highway Pavements*. Reston: American Society of Civil Engineers. DOI: 10.1061/9780784479216.012.
- ASTM (2015a) *D 4402-15: Standard Test Method for Viscosity Determination of Asphalt at Elevated Temperatures Using a Rotational Viscosimeter*. West Conshohocken: ASTM.
- ASTM (2015b) *D7175-2015: Standard Test Method for Determining the Rheological Properties of asphalt Binder Using a Dynamic Shear Rheometer*. West Conshohocken: ASTM.
- ASTM (2019) *D 6114-19: Standard Specification for Asphalt-Rubber Binder*. West Conshohocken: ASTM.
- ASTM (2020) *7405-20: Standard Test Method for Multiple Stress Creep and Recovery (MSCR) of Asphalt Binder Using a Dynamic Shear Rheometer*. West Conshohocken: ASTM.
- ASTM (2021) *D 8239-21: Standard Specification for Performance-Graded Asphalt Binder Using the Multiple Stress Creep and Recovery*. West Conshohocken: ASTM.
- Bailey, W.R. (2010) Rubberized Asphalt Pellets - Patent US-2010/0056669-A1.
- Bennert, T. (2013) *Grade Determination of Crumb Rubber-Modified Performance Graded Asphalt Binder: Final Report (No. C-08-20)*. Piscataway: CAIT - Rutgers University.
- Bernucci, L.B.; G.S. Pinheiro; J.A. Rodrigues et al. (2020) Avaliação do efeito do gap da geometria de placas paralelas no comportamento reológico de um ligante modificado por borracha. In *34^o Congresso de Pesquisa e Ensino em Transporte da ANPET*. Rio de Janeiro: ANPET, p. 1376-87. Available at: <http://www.anpet.org.br/anais34/documentos/2020/Infraestrutura/Ligantes%20e%20Misturas%20Asf%C3%A1lticas%20III/2_359_AC.pdf> (accessed 09/01/2023).
- Bernucci, L.L.B.; L.M.G. Motta; J.A.P. Ceratti et al. (2022) *Pavimentação Asfáltica: Formação Básica para Engenheiros* (2nd ed.). Rio de Janeiro: Petrobrás.
- Bertollo, S.A.M. (2002) *Avaliação Laboratorial de Misturas Asfálticas Densas Modificadas com Borracha Reciclada de Pneus*. Tese (doutorado). Engenharia Civil, University of São Paulo, São Paulo. DOI: 10.11606/T.18.2002.tde-14122015-152916.
- Bukowski, J. (2014) *The Use of Recycled Tire Rubber to Modify Asphalt Binder and Mixtures* (FHWA-HIF-14-015). Washington, DC: FHWA.
- Caltrans (2003). *Asphalt Rubber Usage Guide*. California: Department of Transportation.
- Camargo, F.F. (2016) *Field and Laboratory Performance Evaluation of a Field-Blended Rubber Asphalt*. Thesis (PhD). Civil Engineering, Universidade de São Paulo, São Paulo. Available at: <<https://www.teses.usp.br/teses/disponiveis/3/3138/tde-01072016-111905/publico/FelipeFilizzolaCamargoCorr16.pdf>> (accessed 08/28/2021).
- Chen, H.; Y. Zhang and H.U. Bahia (2021) Estimating asphalt binder fatigue at multiple temperatures using a simplified pseudo-strain energy analysis approach in the LAS Test, *Construction & Building Materials*, v. 266, p. 120911. DOI: 10.1016/j.conbuildmat.2020.120911.
- Corrigan, M. and A. Golalipour (2016) *Ground Tire Rubber: Field Projects, Experiences and Lessons Learned*. Fall River: FHWA. (Asphalt Binder ETG Meeting Technical Report).
- Dantas Neto, S.A. (2004) *Avaliação das Propriedades dos Ligantes e das Misturas Asfálticas Modificados com Borracha Granulada de Pneus Usados*. Tese (doutorado). Engenharia Civil, Universidade de Brasília, Brasília, DF. Available at: <https://www.consulpav.com/docs/06-Tese_Doutoramento_Silvrano_Adonias_Dantas_Neto/Tese-SILVRANO_ADONIAS_DANTAS_NETO-024-2004.pdf> (accessed 08/28/2021).
- DNIT (2009) EM 111/2009: *Pavimentação flexível – Cimento asfáltico modificado por borracha de pneus inservíveis pelo processo via úmida do tipo “terminal blending”*. Brasília, DF: DNIT.
- DNIT (2020) ME 423/2020: *Pavimentação – Ligante Asfáltico – Fluência e Recuperação de Ligantes Asfálticos determinados sob tensão múltipla (MSCR)*. Brasília, DF: DNIT.
- DNIT (2022) ME 439/2022: *Pavimentação – Ligante asfáltico – Avaliação da resistência à fadiga de ligantes asfálticos usando varredura de amplitude linear (LAS – Linear Amplitude Sweep)*. Brasília, DF: DNIT.
- Domingos, M.D.I. (2011) *Caracterização do Comportamento Fluência Recuperação de Ligantes Asfálticos Modificados Virgens e Envelhecidos*. Dissertação (mestrado). Engenharia Civil, Universidade de São Paulo, São Paulo. DOI: 10.11606/D.18.2011.tde-12032012-144327

- Ecorodovias (2021) Relatório integrado. Available at: <<https://api.mziq.com/mzfilemanager/v2/d/7c109ecb-88c9-441f-91cb-66a8db417120/db3ecd97-dc7b-bc89-c55e-c1889945e6d5?origin=2>> (accessed 08/28/2021).
- Fengler, R.Z. (2018) *Caracterização de Ligantes e Misturas Asfálticas Modificados com a Adição de Trinidad Lake Asphalt*. Dissertação (metrado). Engenharia Civil, Universidade Federal do Rio de Janeiro, Rio de Janeiro. Available at: <<https://pantheon.ufrj.br/bitstream/11422/13418/1/RicardoZardinFengler.pdf>> (accessed 08/28/2021).
- FHWA (2014) *FHWA-HIF-14-015: The Use of Recycled Tire Rubber to Modify Asphalt Binder and Mixtures*. Washington, D.C.: Federal Highway Administration. Available at: <<https://www.fhwa.dot.gov/pavement/pubs/hif14015.pdf>> (accessed 16/04/2023).
- Fontes, L.P. (2009) *Optimização do Desempenho de Misturas Betuminosas com Betume Modificado com Borracha para Reabilitação de Pavimentos*. Tese (doutorado). Engenharia Civil, Universidade do Minho, Portugal. Available at: <<https://repositorium.sdum.uminho.pt/handle/1822/9601>> (accessed 08/28/2021).
- Gaspar, M.S. (2019) *Mecanismos de Interação e Ativação entre Ligantes na Reciclagem Asfáltica a Quente e Morna*. Dissertação (metrado). Engenharia Civil, Universidade de São Paulo, São Paulo. DOI: 10.11606/D.3.2019.tde-22082019-141237.
- Greca Asfaltos (2021) *ECOFLEX 20 anos de asfalto-borracha no Brasil 2001-2021*. Available at: <<https://fatoseasfaltos.com.br/>> (accessed 08/28/2021).
- Jones, D.; Y. Liang and J. Harvey (2017) *Performance Based Specifications, Literature Review on Increasing Crumb Rubber Usage by Adding Small Amounts of Crumb Rubber Modifier in Hot Mix Asphalt*. California: California Department of Transportation.
- Kim, R.Y. (2014) *Asphalt Pavements*. Boca Raton: CRC Press. DOI: 10.1201/b17219.
- Klinsky, L.M.G. and V.C. Faria (2017) *Asfalto-borracha: Revisão bibliográfica, avaliação reológica do ligante asfáltico e determinação dos parâmetros mecânicos das misturas asfálticas* (Report CCR-ND-AB-RF-DEZ/2017). Brasília, DF: ANTT.
- Klinsky, L.M.G.; V.S.D. Bardini and V.C. Faria (2020) Evaluation of permanent deformation of asphalt rubber using multiple stress creep recovery tests and flow number tests. *Transportes*, v. 28, n. 2, p. 76-86. DOI: 10.14295/transportes.v28i2.2110.
- Leite, L.F.M. (1999). *Estudos de Preparo e Caracterização de Asfaltos Modificados por Polímeros*. Tese (doutorado). Engenharia Civil, Universidade Federal do Rio de Janeiro, Rio de Janeiro.
- Lima, C.S. (2008). *Caracterização de Ligantes Asfálticos Modificados com Borracha de Pneu e Aditivos*. Dissertação (mestrado). Engenharia Civil, Universidade Federal do Ceará, Ceará. Available at: <https://repositorio.ufc.br/bitstream/riufc/4871/1/2008_dis_cslima.pdf> (accessed 08/28/2021).
- Lo Presti, D. (2013) Recycled tire rubber modified bitumens for road asphalt mixtures: a literature review, *Construction & Building Materials*, v. 49, p. 863-81. DOI: 10.1016/j.conbuildmat.2013.09.007.
- Martins, A.T. (2014) *Contribuição para a Validação do Ensaio de Resistência ao Dano por Fadiga para Ligantes Asfálticos*. Dissertação (mestrado). Engenharia Civil, Universidade Federal do Rio de Janeiro, Rio de Janeiro. Available at: <www.coc.ufrj.br/pt/documents2/mestrado/2014/2488-adriana-tinoco-martins-mestrado/file> (accessed 08/28/2021).
- Mturi, G.; J. O'Connell and S. Zoorob (2014) A study of crumb rubber modified bitumen used in South Africa, *Road Materials and Pavement Design*, v. 15, n. 4, p. 774-90. DOI: 10.1080/14680629.2014.910130.
- Nanjegowda, V.H. and K.P. Biligiri (2020) Recyclability of rubber in asphalt roadway systems: a review of applied research and advancement in technology, *Resources, Conservation and Recycling*, v. 155, p. 104655. DOI: 10.1016/j.resconrec.2019.104655.
- Nascimento, L.A.H. (2015) *Implementation and Validation of the Viscoelastic Continuum Damage Theory for Asphalt Mixture and Pavement Analysis in Brazil*. Dissertation (PhD). North Carolina State University. Raleigh. Available at: <<https://repository.lib.ncsu.edu/handle/1840.16/10651>> (accessed 16/04/2023).
- NDOT (2016) *Research Report N° 513-13-803: Evaluation of New Innovations in Rubber-Modified Asphalt Binders and Rubberized Asphalt Mixes for Nevada*. Carson: Nevada Department of Transportation.
- Nunes, L.C. (2017) *Fadiga de Misturas Asfálticas Descontínuas com Asfalto-Borracha de 4ª Geração*. Dissertação (mestrado). Engenharia Civil, Universidade de Brasília, Brasília, DF. Available at: <<https://repositorio.unb.br/handle/10482/23583>> (accessed 08/28/2021).
- Nuñez, J. (2013) *Caracterização à Fadiga de Ligantes Asfálticos Modificados Envelhecidos a Curto e Longo Prazo*. Dissertação (mestrado). Engenharia Civil, Universidade de São Paulo, São Paulo. DOI: 10.11606/D.18.2013.tde-19112013-171029.

- Oda, S. (2000) *Análise da Viabilidade Técnica da Utilização do Ligante Asfalto-Borracha em Obras de Pavimentação*. Tese (doutorado). Engenharia Civil, Universidade de São Paulo, São Paulo. DOI: 10.11606/T.18.2018.tde-30012018-151928.
- Pamplona, T.F. (2013) *Efeito da Adição de Ácido Polifosfórico em Ligantes Asfálticos de Diferentes Fontes*. Dissertação (mestrado), Engenharia Civil, Universidade de São Paulo, São Paulo. DOI: 10.11606/D.18.2013.tde-29112013-084714.
- Shatnawi, S. (2011) *Comparisons of Rubberized Asphalt Binders, Asphalt Rubber and Terminal Blend*. California: Shatec Engineering Consultants.
- Sockwell, K. (2013) Composition for pelletized bitumen and method for preparing the same - US Patent 8404164.
- Song, J.; J. Xie; S. Wu et al. (2022) Study on properties and improving mechanism of OGFC-13 asphalt mixtures modified by novel rubber pellets. *Construction & Building Materials*, v. 325, p. 126799. DOI: 10.1016/j.conbuildmat.2022.126799.
- Specht, L.P. (2004) *Avaliação de Misturas Asfálticas com Incorporação de Borracha Reciclada de Pneus*. Tese (Doutorado). Engenharia Civil, Universidade Federal do Rio Grande do Sul, Rio Grande do Sul. Available at: <<https://www.lume.ufrgs.br/handle/10183/5192>> (accessed 08/28/2021).
- Specht, L.P.; V.N. Ramos; A.A. Oliveira et al. (2019) Análise de parâmetros reológicos de rigidez de ligantes asfálticos por meio do reômetro de cisalhamento dinâmico (DSR). In *X Seminário de Engenharia Geotécnica do Rio Grande do Sul*. Santa Maria: Universidade Federal de Santa Maria.
- Subhy, A.; D. Lo Presti and G. Airey (2015) An investigation on using pre-treated tyre rubber as a replacement of synthetic polymers for bitumen modification, *Road Materials and Pavement Design*, v. 16, n. 1, p. 245-64. DOI: 10.1080/14680629.2015.1030826.
- Wang, H.; X. Liu; H. Zhang et al. (2018) Asphalt-rubber interaction and performance evaluation of rubberized asphalt binders containing non-foaming warm-mix additives, *Road Materials and Pavement Design*. DOI: 10.1080/14680629.2018.1561380.
- Way, G.B.; K.E. Kaloush and K.P. Biligiri (2012) *Asphalt-Rubber Standard Practice Guid*. Tempe: Rubber Pavement Association.
- Zoorob, S.E.; S.K. Al-Bahar and S.F. Al-Otaibi (2018) Design and optimization of a rubber-bitumen blend in preparation for a rubberized-asphalt road trial in the state of Kuwait. *MATEC Web of Conferences*, v. 195, p. 04001. DOI: 10.1051/mateconf/201819504001.