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Sylvaroad rejuvenating agent's dosage based on rheological properties of aged asphalt binder

Dosagem do agente rejuvenescedor Sylvaroad com base nas propriedades reológicas de ligante asfáltico envelhecido

Felipe Tiago Joenck[1], **Vanessa Bacca Couto Joenck**[1], **Joe Arnaldo Villena Del Carpio**[1], **João Victor Staub de Melo**[2]

[1]Federal University of Paraná, Curitiba, Paraná, Brazil
[2]Federal University of Santa Catarina, Florianópolis, Santa Catarina, Brazil

Contact: felipe.joenck@gmail.com, (FTJ); vanessa.bacca@gmail.com, (VBCJ); joe.villena@ufpr.br, (JAVDC); joao.victor@ufsc.br, (JVSM)

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ABSTRACT
The technique of using rejuvenating agents (RA) to recover aged binders' properties has still a lot to develop since the RA amount directly affects binders' performance on rutting and fatigue, main factors of pavement degradation. This research evaluated dosage methods of RA Sylvaroad RP1000 in a service-aged binder, based on rheological parameters. The binder was extracted from Reclaimed Asphalt Pavement (RAP) using centrifugation and rotoevaporation. Samples with 0, 3, 5, 7, and 10% RA were produced and subjected to Rolling Thin Film Oven Test (RTFOT) and rheological tests on the Dynamic Shear Rheometer (DSR). The optimum dosage was assessed, based on virgin binder High-temperature Performance Grade (PGH). Susceptibility parameters to fatigue and rutting were also measured by Multiple Stress Creep and Recovery (MSCR) and Linear Amplitude Sweep (LAS) tests, respectively. Results showed that the RA was effective, and the dose able to recover virgin binder's PGH was 10.6% of the aged binder mass. Non-recoverable compliance, stress recovery rate and fatigue damage tolerance results showed this content would lead to satisfactory and superior results to those of the virgin binder (50/70 penetration grade). However, a lower content would already be able to recover aged binder's properties and improve its performance.

RESUMO
A técnica de utilização de agentes rejuvenescedores (AR) na recuperação das propriedades de ligantes envelhecidos ainda tem muito a ser desenvolvida, considerando que a quantidade de AR afeta diretamente o desempenho dos ligantes à fadiga e deformação permanente, principais fatores de degradação do pavimento. Esta pesquisa avaliou os métodos de dosagem de AR Sylvaroad RP1000 em ligante envelhecido, com base em parâmetros reológicos. O ligante foi extraído de mistura asfáltica fresada (RAP), por meio de centrifugação e rotoevaporação. Foram produzidas amostras com 0, 3, 5, 7 e 10% de AR e submetidas a ensaios na Estufa de Película Delgada Rotacional (RTFOT) e Reômetro de Cisalhamento Dinâmico (DSR). A dosagem ideal foi avaliada com base no *Performance Grade* de alta temperatura (PGH) do ligante virgem. Os parâmetros de suscetibilidade à deformação permanente e à fadiga também foram obtidos pelos ensaios de Fluência e Recuperação sob Tensão Múltipla (MSCR) e Varredura de Amplitude Linear (LAS). Os resultados mostraram a eficácia do AR, cujo teor capaz de recuperar o PGH do ligante virgem foi de 10,6% da massa do ligante envelhecido. Este teor apresentou resultados satisfatórios de compliância não recuperável, taxa de recuperação e resistência à fadiga, inclusive superiores aos do ligante virgem.

1. INTRODUCTION

The use of Reclaimed Asphalt Pavement (RAP) has grown substantially in recent years. In countries such as France, the Netherlands, and Germany, the available percentage of RAP used in recycling is

64%, 76%, and 90%, respectively (Noferini et al., 2017), and around 99% in Japan and the United States (West and Copeland, 2015). In Brazil, however, the use of RAP in new pavements is not yet consolidated; the few experiments carried out generally used low contents of this material. For the routine use of high RAP contents (greater than 25%), several issues still need to be addressed (Zaumanis, Mallick and Frank, 2014), including rejuvenation of asphalt binders through rejuvenating agents (RA), or just rejuvenators. These agents consist of organic oils or petroleum derivatives, capable of recovering a binder's characteristics, such as penetration, softening point and viscosity, and also rheological parameters, such as dynamic shear modulus and phase angle (Arámbula-Mercado et al., 2018).

Petroleum-based rejuvenators are the most common, but they have complications that need to be considered. This type of RA tends to volatilize more easily at high temperatures, which means poor regeneration efficiency. Also, they are derived from a non-renewable material and thus do not satisfy the requirements of sustainable development (Fang et al., 2021). Bio-rejuvenators, however, are green, environmental protection and renewable material, which has a very wide application prospect in the regeneration of asphalt materials, with satisfactory results obtained (Fang et al., 2021; Wang et al., 2023). Various bio-based oils are being produced and studied nowadays, such as vegetable oils, castor oil, waste engine oil, waste cooking oils, sludge oils (Fang et al., 2021; Wang et al., 2023; Deng et al., 2022; Yaro et al., 2023; Cao et al., 2018), and also crude tall oils, such as Sylvaroad RP 1000, which is a renewable raw material, by-product of the paper industry. Besides being made of renewable raw materials, Sylvaroad RP 1000 is non-hazardous, safe to handle and environmentally friendly. By allowing the use of high percentages of RAP in new mixtures, it can result in significant cost savings due to a reduced need for new materials and potential disposal fees (Kraton Corporation, 2017).

For the proper use of RA in recycled asphalt mixtures, dosage must be carefully determined, as it directly affects their performance. Excessive RA amounts will overly soften the binder, which impacts performance concerning permanent deformation, while insufficient amounts will not have the desired effect on improving cracking resistance (Arámbula-Mercado et al., 2018). Recovering virgin binder's properties through blending charts is a commonly used method for dosing RA. Recovering binder's viscosity and penetration, for example, may not be the most suitable for mixtures with high RAP contents, as it leads to higher RA contents (Gadler, 2018). Recent research tends to use rheological properties as a parameter for RA dosage (Arámbula-Mercado et al., 2018; Im, Karki and Zhou, 2016; Kaseer et al., 2018a; Asadi, Tabatabaee and Hajj, 2021), as they could be more related to binder performance at different temperatures. The Superpave methodology parameters have been used for this purpose, with emphasis on the $G^*/\sin\delta$, used for determining the binders' Performance Grade (PG).

Satisfactory results were obtained using virgin binder High-temperature Performance Grade (PGH) as a parameter for RA dosage. Researchers concluded that it is the most effective method when compared to restoring the Low-temperature Performance Grade (PGL) and the ΔTc parameter, which represents the temperature difference in the Bending Beam Rheometer test (Arámbula-Mercado et al., 2018). The virgin binder PGH dosage method was also validated by comparing it with the RA dosage used in projects executed by the transportation departments of five states in the United States (Kaseer et al., 2018a). Results presented a better performance of mixtures dosed by PGH, compared to the ones dosed by the transportation departments' methodologies. Also, higher RA contents were found using PGH recovery.

However, the procedure for determining rejuvenator optimum content based on its fatigue and cracking resistance, using the results of Linear Amplitude Sweep test (LAS), represents advancement beyond that of the PG-based methods (Asadi, Tabatabaee and Hajj, 2021). Thus, depending on which virgin binder parameter is to be recovered through the RA dosage, significant differences between the optimum contents can be found. Furthermore, it should be considered that plant-based organic rejuvenators require lower doses than petroleum-based ones to cause the same effect on the aged binder (Kaseer et al., 2018b; Zaumanis et al., 2014; Bajaj et al., 2020). The efficiency of organic agents — a group that includes tall oil, vegetable oils, and bio-based oils — is one of the reasons why these products have become common in research on the subject.

Developing asphalt concrete pavements resistant to both cracking and permanent deformation is a constant challenge in the industry (Espinoza-Luque, Al-Qadi and Ozer 2018), and the same is true for recycled asphalt mixtures using RA. Optimizing the rejuvenating agent's dosing process is, therefore, a topic that needs to be further studied in order to improve rejuvenated binders' behavior and recycled asphalt mixtures' performance. Other studies have already proved the PG-based methods to be efficient (Arámbula-Mercado et al., 2018; Kaseer et al., 2018a) and LAS-based methods (Asadi, Tabatabaee and Hajj, 2021). None of them, however, compared the optimum dosages obtained by PGH, Linear Amplitude Sweep (LAS) and also Multiple Stress Creep and Recovery (MSCR) tests simultaneously, especially considering that the susceptibility to fatigue and rutting (measured by these two tests, respectively) are the main performance parameters of an asphalt binder, responsible to ensure its service life extension.

Thus, this research proposes to investigate the effects of RA Sylvaroad RP1000 on the rheological properties of fatigue resistance and permanent deformation of a recovered binder. Also, it intends to determine the optimal dosage based on these performance trials, using LAS and MSCR tests, comparing it to a PG-based dosage.

2. MATERIALS

The main materials required to conduct this research were the aged asphalt binder, extracted and recovered from milled material (RAP), a virgin binder commercially called CAP 50/70, hereinafter referred to as Pen 50/70, and a plant-based rejuvenating agent. Each of these materials' characteristics is detailed below.

2.1. Asphalt binders

The virgin asphalt binder used as a control sample was Pen 50/70, which is the most common binder available on the Brazilian market, widely used in road maintenance and conservation works, so named due to its penetration range of results. The quantity required for the research was courtesy of CBB Asfaltos, located in the municipality of Araucária, state of Paraná, Brazil. Binder characteristics are presented in Table 1.

The aged asphalt binder was extracted from RAP, obtained from the Road Department of Parana (DER/PR), as a result of milling service for maintenance and conservation of the PR-412 roadway, in the municipality of Pontal do Paraná, Paraná, Brazil. As informed by the department inspector, it is estimated that the milled pavement has been in service for 11 years and was originally produced with conventional Pen 50/70 binder, without modification.

Table 1: Physical properties of the Pen 50/70 asphalt binder.

Characteristics	Method	Specification	Result	Unit
Penetration	ASTM D5 (ASTM, 2020b)	50 to 70	52	0.1 mm
Softening point	ASTM D36 (ASTM, 2016a)	46 minutes	51	ºC
Brookfield 135 ºC sp21 20rpm viscosity	ASTM D4402 (ASTM, 2006)	274 minutes	331	cp
Brookfield 150 ºC sp21 50rpm viscosity	ASTM D4402 (ASTM, 2006)	112 minutes	166	cp
Brookfield 177 ºC sp21 100rpm viscosity	ASTM D4402 (ASTM, 2006)	57 to 285	62	cp
RTFOT retained penetration	ASTM D5 (ASTM, 2020b)	55 minutes	64	%
RTFOT - Ductility at 25 ºC	ASTM D113 (ASTM, 2017)	20 minutes	> 100	cm
RTFOT mass variation in %	ASTM D2872 (ASTM, 2019)	-0.50 to 0.50	-0.06	%
Ductility at 25 ºC	ASTM D113 (2017)	60 minutes	> 100	cm
Flash point	ASTM D92 (ASTM, 2018a)	235 minutes	340	ºC
Relative density at 20/4 ºC	ASTM D70 (ASTM, 2018b)	-	0.993	-

2.2. Rejuvenating agent

The rejuvenating agent used in this research is an organic product obtained from pine oil, commercially called Sylvaroad RP1000. Unlike petroleum-based rejuvenators, Sylvaroad is an additive produced from crude sulfated turpentine oil, a chemical extracted from pine wood deriving from pulp and paper industry. According to the manufacturing company, Arizona Chemical (acquired in 2016 by Kraton Corporation), Sylvaroad is a liquid additive that reactivates the aged RAP binder and increases its flexibility; consequently improving cracking resistance and allowing larger amounts of RAP to be added to the mix. The product has a high flash point, 280 ºC on average, and is not dangerous at high temperatures, which allows it to be added directly to the RAP in hot, warm, or cold mixtures. Three years were spent creating this product, which was developed to interact with the RAP binder at a molecular level, not just reducing its viscosity, as regular oils do (Smith, 2015). This molecular interaction, however, is not detailed by the manufacturer, but literature shows that tall oils (such as Sylvaroad) are proven to perform better than petroleum-based products (Gadler, 2018).

3. METHODS

The present research sought to determine the optimal RA content in an aged binder, taking the virgin binder's PGH recovery as a parameter and considering the analysis of fatigue damage tolerance properties, non-recoverable creep compliance, and recovery rate (%R). To achieve the proposed objectives, it was necessary to extract and recover aged binder from RAP and to carry out rheological tests in the Dynamic Shear Rheometer (DSR) equipment, on both original and short-term aged samples, to further determine the optimum content by a blending chart. The flow chart in Figure 1 summarizes the research steps, detailed in sequence.

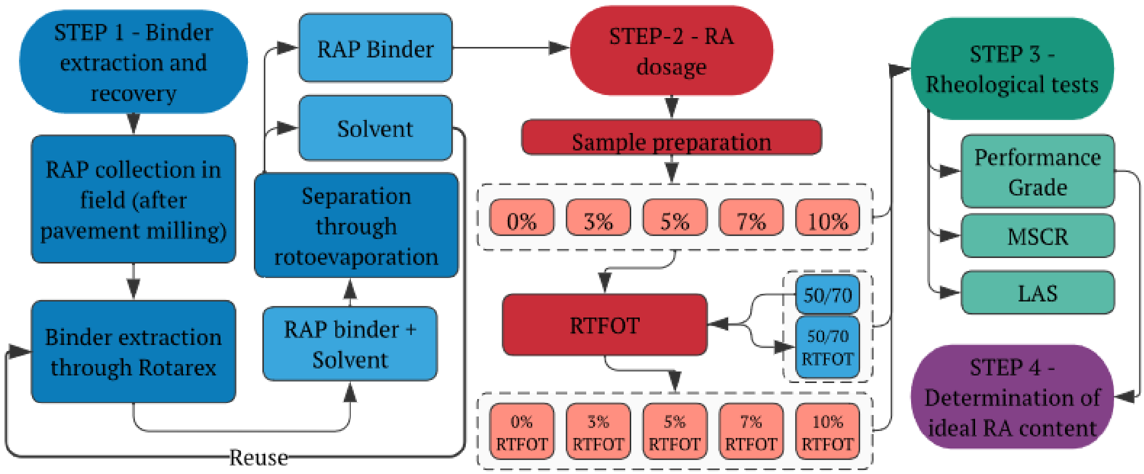

Figure 1. Flowchart of the research methods.

3.1. Step 1 – Binder extraction and recovery

Before the extraction, RAP preparation began with drying it at 40 °C in an oven (to simulate air drying) for at least 24 hours, until the complete removal of moisture. Subsequently, RAP was fractioned into fine and coarse fractions, using sieve No.4. Lumps larger than 25 mm were discarded, as they could harm the extraction process, requiring greater use of solvents. Samples of each fraction were then reduced by quartering into approximately 1000 g, to maintain homogeneity. RAP binder extraction was performed using a centrifuge equipment, with both coarse and fine RAP samples. Since no ash content tests would be conducted and to prevent fines from remaining in the resultant binder (after recovery), two paper filter units were used in the equipment sample container, instead of just one. The chosen solvent was dichloromethane, for being more effective in binder dissolution (Mikhailenko, Webber and Baaj, 2021) and for having a boiling temperature of around 40 °C, which facilitates the binder's subsequent recovery and reduces its aging during the process.

The binder's recovery, which consists of its separation from the solvent, was carried out by a rotoevaporator. This method is preferred by researchers due to its ability to provide larger sample volumes, leave less residual solvent after the recovery process, and cause less age-hardening (Mikhailenko, Ateian and Baaj, 2020). In the rotoevaporation method, the solvent and asphalt binder solution is placed in a rotating flask and distilled through partial immersion in a temperature-controlled water bath. This method does not cause changes in binders' properties by the influence of temperature during distillation, as long as the glass joints of the equipment are well sealed (Hospodka, Hofko and Blab, 2018). Heating the water bath to a temperature of 50 °C was enough to evaporate the solvent. For safety, the flask with the solution (solvent+binder) was only filled halfway (250 mL), and after evaporation of most of the solvent, refilled and reinserted into the system, repeating the process until all the solvent had evaporated. Toward the end of the process, when the solution in the flask was highly viscous and no solvent was visibly condensing into the collection container, the temperature was raised to 80 °C for 60 min to ensure complete evaporation of any remaining solvent. After the end of the process, the flask was taken to the oven at 80 °C and remained inverted for 24 hours so the binder could be completely drained to the sampler. The main steps of the extraction and recovery process are shown in Figure 2.

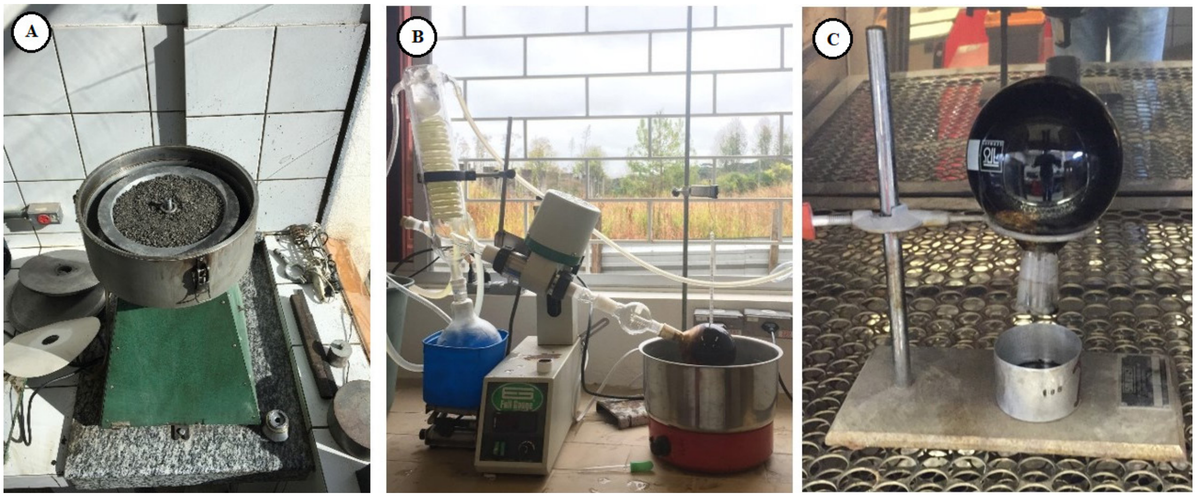

Figure 2. (A) Centrifuge extraction process; (B) Solvent separation in the rotoevaporator, by simple distillation at 50 ºC water bath; (c) Binder spilling from the flask for 24 hours, inside the oven at 80 ºC.

12 extractions were performed to obtain a sufficient amount of binder for the analyses (600 g). Penetration tests were performed according to ASTM D5 (2020b) after each recovery, for binder sample control, in order to keep them homogeneous before moving on to the dosing stage and the rheological tests. Penetration results that were too high or different from the average, called outliers, could be an indicator of residual solvent presence. When these situations occurred, samples were again submitted to rotoevaporation for additional 2 hours. After this process, if penetration results remained high, samples were then discarded. Finally, after the invalid samples were discarded, all the binders obtained in each recovery process were unified and manually mixed at the temperature of 80 ºC, so they could be submitted again to the rotoevaporation test for 4 hours, at an 80 ºC water bath. This last process aimed to eliminate all residual solvent to obtain a homogeneous binder, while maintaining all its original properties, therefore eliminating variables that could arise from the extraction and recovery processes.

3.2. Step 2 – RA dosage

The Performance Grade binder classification system is defined by ASTM D6373 (2016b) standard, based on the idea that the properties of asphalt binder should be related to the conditions in which it will be used (Speight, 2016). Binders' graded designations are related to the pavement's high design temperature, at a depth of 20 mm below the surface, and the low design temperature, at the surface itself (Nikolaides, 2015). The degree of binder performance required is then expressed, for example, in PG 64-28 format; which designates a suitable binder for an environment where the maximum temperature of the pavement does not exceed 64 ºC (at a depth of 20 mm), and is not lower than -28 ºC on the surface (Huang, 2004). The maximum temperature in the given example (64 ºC) is known as PGH (High-Temperature Performance Grade) and the minimum temperature (-28 ºC) as PGL (Low-Temperature Performance Grade). The objective of this research is to find the ideal dosage so that the continuous aged binder's PGH is reestablished at the same value as the virgin binder's PGH, using a blending chart.

To achieve that, the unified RAP binder was divided into 10 samples so it could receive five different RA contents (two samples for each content) based on binder's weight. Contents of 0, 3, 5, 7, and 10% RA were established according to the manufacturer's protocol. It is known that contents of up to 10% of this kind of RA are already sufficient to restore an aged binder's properties, such as penetration and viscosity (Porot and Bell, 2019). This RA content was then set as the highest for the blending chart's production. The process of dosing RA on the scale is shown in Figure 3. The mixing procedure followed the manufacturer's protocol: the RA was directly added to the preheated binder (at 130 ºC) and manually mixed for 30 seconds. The binder was then reheated for 10 minutes and homogenized for another 30 seconds every 5 minutes.

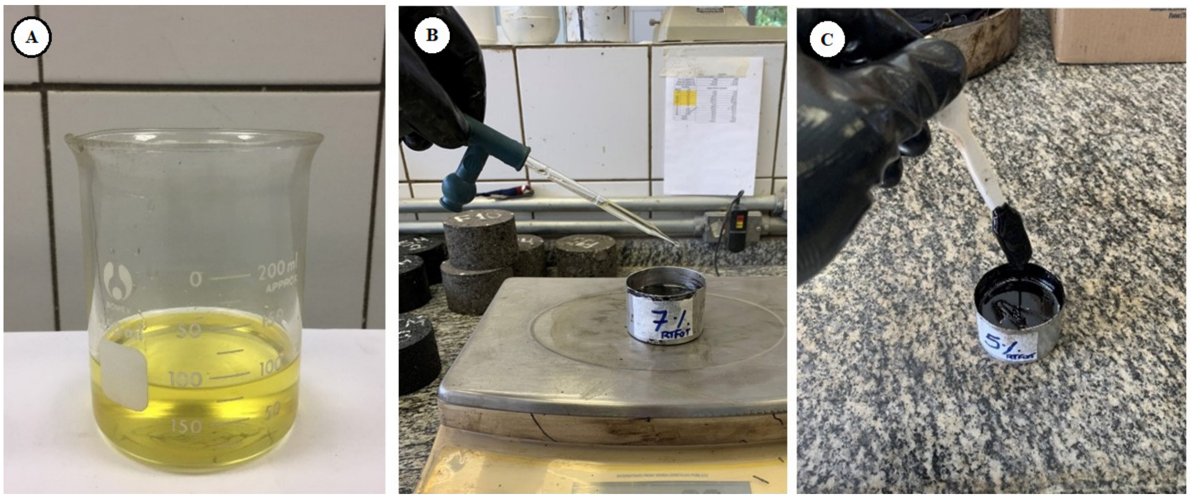

Figure 3. (A) RA Sylvaroad RP 1000 in original condition; (B) RA dosage; (C) RA and binder manual mixing.

3.3. Step 3 – Rheological tests

One sample of each RA content was subjected to short-term ageing in Rolling Thin Film Oven Test (RTFOT), according to ASTM D2872 (2019), for 85 minutes at 163 ºC, while other sample was kept in its original condition. Pen 50/70 virgin binder received the same treatment for comparison. All samples, in both original and aged conditions, were then subjected to the Dynamic Shear Rheometer testing, according to ASTM D7175 (ASTM, 2015), to obtain their G*/sinδ parameter and determine PGH. The G*/sinδ parameter's minimum values set to determine the samples' failure temperature were 1.0 kPa for the binder's original condition and 2.2 kPa for the RTFOT aged condition, as specified by ASTM D6373 (ASTM, 2016b) and ASTM D7643 (ASTM, 2016c). With these results in hand, it was possible to perform a graphical analysis and determine the approximate content to recover virgin binder's PGH.

In terms of non-recoverable compliance and recoverable rate in stress, the rejuvenated binders' performance was evaluated using Multiple Stress Creep and Recovery (MSCR) tests, carried out on the samples after their RTFOT aging. The test was performed at 64 ºC (virgin binder PG) with a 25 mm diameter geometry and a 1.0 mm gap. Twenty 0.1 kPa load cycles were applied with 1.0 s of load application and 9.0 s of recovery, along with 10 further 3.2 kPa load cycles, as determined by ASTM D7405 (ASTM, 2020a). The elastic response of each binder to shear is obtained through its recovery rate (%R) and the non-recoverable compliance (Jnr).

The material's fatigue damage tolerance was analyzed using Linear Amplitude Sweep (LAS) tests at 20 ºC, on samples with 8.0 mm diameter geometry and 2.0 mm gap, after the RTFOT test. According to AASHTO T 391 (AASHTO, 2020), the curve's A and B parameters were determined through Viscoelastic Continuum Damage analysis (VECD), and the number of cycles to failure (Nf) of different strain amplitudes was calculated. The failure criterion adopted was a 35% reduction in the parameter |G*|. sinδ, according to the aforementioned standard.

4. RESULTS AND DISCUSSIONS

4.1. Extraction control by penetration

As shown in Figure 4, discarding the outliers, the extraction/recovery samples' penetration results ranged from 24 to 36 mm/10.

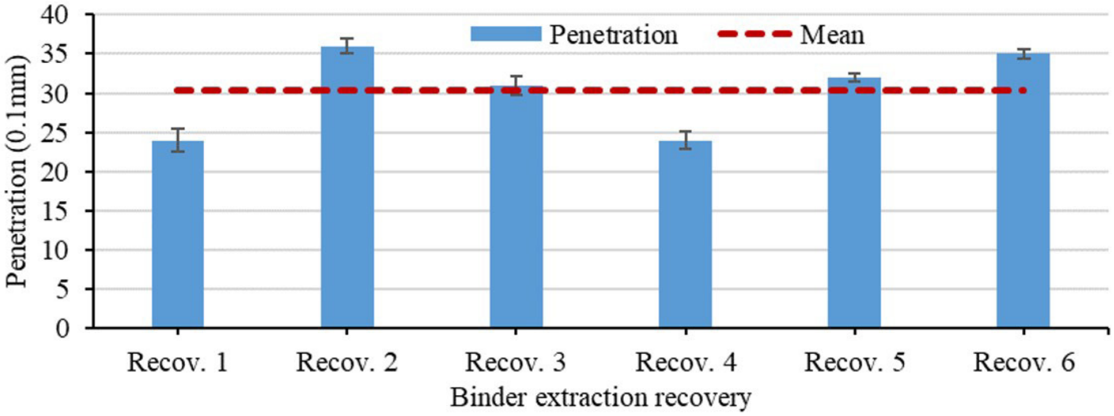

Figure 4. Result of the valid samples' penetration after each binder extraction and recovery from the RAP. Error bars correspond to standard deviation.

After all extractions/recoveries were turned into a single homogeneous sample, the result was a binder whose penetration was approximately 32 mm/10. It's worth mentioning that Pen 50/70's penetration should vary between 50 and 70 mm/10 when virgin; thus, it can be seen that this is indeed an aged binder.

4.2. Step 4 – Optimum content determination

The results obtained for the High-temperature Performance Grade (PGH) are presented in Table 2. Sample RAP + 0% binder, extracted from the RAP without RA addition, showed greater variation between the original condition results and the results after RTFOT when compared to the virgin binder Pen 50/70. For the latter, PGH determined for both conditions varied only at approximately 2 ºC, whereas for the RAP + 0% sample, the variation was 4 ºC. This could indicate the presence of residual solvent in the samples; however, such conclusion cannot be directly drawn since the samples with added RA presented a variation below 2 ºC.

The results showed that even a 10% RA content, which is considered high, was not able to recover virgin binder's PGH in both original and RTFOT aged conditions. It is also verified that the samples phase angle (δ) at 64 ºC increased with the increase of RA, decreasing after short-term aging. Even with 10% RA, this parameter did not exceed that of the virgin binder, denoting a better elastic response of the rejuvenated binder compared to the virgin one. The difference in binder phase angle after RTFOT indicates that RA addition weakens and delays its aging process. Temperature and rejuvenator properties and quantity play a significant role in this rheological response (Yaro et al., 2023).

Higher PGHs also mean lower G*/sinδ parameter (also called rutting index or anti-rutting coefficient), which value takes into consideration both phase angle (δ) and complex shear modulus (G*). The higher G*/sinδ value displays the stronger the anti-deformation ability of asphalt (Deng et al., 2022). The increase in RA content represents a decrease in the value of G*/

sinδ, considering that the addition of light components to the binder affects its viscosity and elasticity. Other researchers have also achieved similar conclusions with bio-oils (Deng et al., 2022; Wang et al., 2023), the challenge being to avoid excessive reduction in the rutting resistance, to be better analyzed with MSCR tests, ahead in this article.

Table 2: DSR test results and determined PGH values.

Binder	Sample	Parameters at 64°C - Original		Original PGH	Original PGH Average	Parameters at 64°C - after RTFOT		PGH after RTFOT**	Average PGH after RTFOT
		δ	G*/sinδ			δ	G*/sinδ		
Pen 50/70	1	88.10	1.12	64.88	64.91	86.70	1.95	63.18	63.20
	2	88.20	1.13	64.94		86.70	1.96	63.21	
RAP + 0%	1	79.80	10.20	81.88	82.25	72.20	42.70	86.69	86.60
	2	79.40	9.64	82.61		72.30	41.40	86.52	
RAP + 3%	1	81.10	5.45	77.76	77.75	75.80	15.50	79.33	79.33
	2	81.10	5.49	77.75		75.80	15.60	79.34	
RAP + 5%	1	82.90	3.14	73.50	74.23	77.30	10.00	75.96	75.96
	2	82.10	3.85	74.95		77.30	10.10	75.96	
RAP + 7%	1	84.00	2.06	70.09	69.94	79.00	6.17	72.15	71.94
	2	84.10	2.00	69.79		79.30	5.78	71.74	
RAP + 10%	1	85.00	1.38	66.78	66.73	81.40	3.21	67.14	67.14
	2	84.90	1.36	66.67		81.50	3.21	67.14	

Failure temperature set with the G/sinδ = 1.0 kPa parameter; ** Failure temperature set with the G*/sinδ = 2.2 kPa parameter

Figure 5 shows that PGH results approximate a linear function with adequate accuracy. They are represented by the equations shown in the graph. It also shows rejuvenated binders' penetration results, whose RA content correlation is exponential, as suggested by Zaumanis, Mallick and Frank (2014). The straight line of the binder's original condition shown in Figure 5 was used to estimate the ideal RA content, able to recover virgin binder's PGH (65.1). This choice was motivated for two reasons: first, the RTFOT aged condition would generate higher contents, considering the Pen 50/70's and the RAP + 10%'s most distant results; second, the fact that, in this case, the original condition was determinant for the definition of the rejuvenated binders' PGH since it presented the lowest values between the two conditions, according to ASTM D7643 (2016c). Therefore, the ideal RA content, defined by extrapolation of the straight line, was 10.6% (PGH = 65.1).

It is important to highlight that, regarding the production of the initial samples, 10% RA content reduced binder's viscosity too much and increased the penetration to 145 x 0.1 mm, which made it difficult to perform the DSR tests, since the sample spilled out of the sampler when heated. An ideal content of 10.6% could therefore be considered high if the property to be recovered was the virgin binders' viscosity or penetration. These parameters have already been used in other studies (Gadler, 2018; Zaumanis, Mallick and Frank, 2014) and are more practical alternatives for RA measurement. In this study, however, if virgin binder's penetration (52 x 0.1 mm) was used as the parameter to be recovered, the ideal RA content would be around 2.8% only; that is, 3.8 times lower than the RA content needed for PGH recovery.

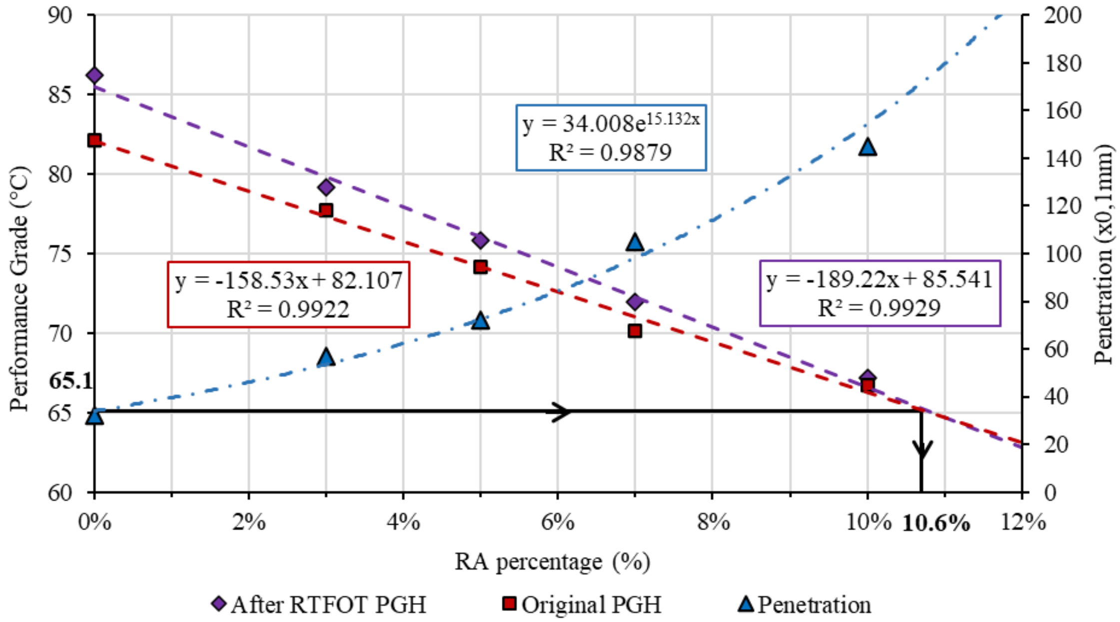

Figure 5. Variation of Performance Grade and penetration with RA content.

4.3 Permanent deformation susceptibility

The permanent deformation susceptibility was evaluated through MSCR tests, also performed in the DSR equipment. The Jnr parameter refers to the unrecoverable residual strain generated by the unit stress level, corresponding to plastic deformation, which should be prevented at high temperatures (Wang et al., 2023). The MSCR test results are shown in Table 3.

Table 3: MSCR test results, performed at 64 °C (virgin binder PGH).

Binder	$\%R_{0.1}$ (%)	$J_{nr0.1}$ (kPa^{-1})	$\%R_{3.2}$ (%)	$J_{nr3.2}$ (kPa^{-1})
Pen 50/70 RTFOT	0.09	4.825	-0.14	5.24
RAP + 0% RTFOT	28.35	0.126	26.96	0.129
RAP + 3% RTFOT	18.3	0.399	14.59	0.425
RAP + 5% RTFOT	14.78	0.685	9.02	0.759
RAP + 7% RTFOT	14.35	0.833	7.96	0.95
RAP + 10% RTFOT	5.09	2.42	1.9	2.755

Results show that Jnr increases with the increment of RA, considering that adding bio-oils raises the light components and reduces the elasticity of asphalt (Wang et al., 2023). By analyzing Table 3, it can be noted that, although the maximum RA content of 10% used appeared to be excessive, the results pointed out that the Jnr determined for this content was not affected to the point of being lesser than the virgin binder's (Pen 50/70). It demonstrates a better behavior at permanent deformation of the rejuvenated sample.

The correlations between rejuvenating agent content and the variables Jnr and %R can be approximated by an exponential equation, represented in Figure 6. These equations showed that the corresponding value of Jnr for the estimated content of 10.6% would be 3.29 kPa^{-1} at a tension of 3.2 kPa, while the %R would be 2.17% at this same tension.

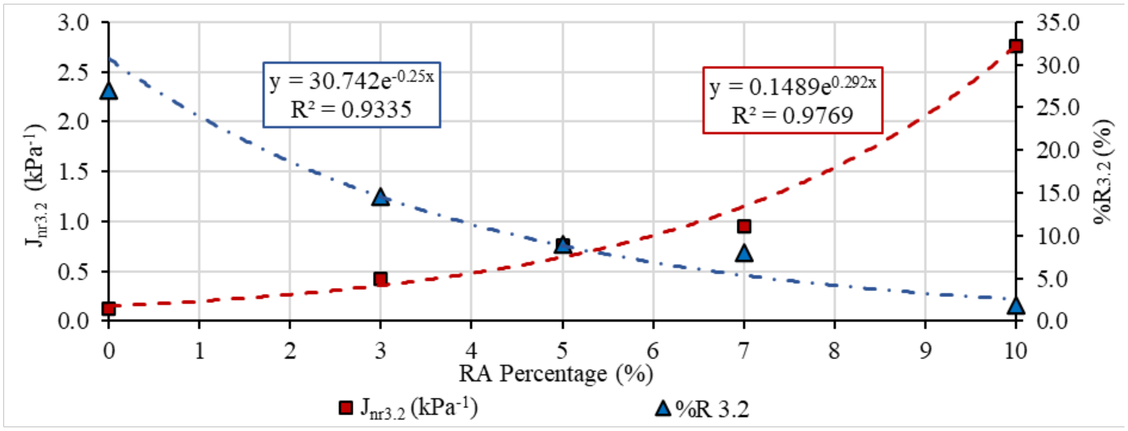

Figure 6. Graph of the relationship between recovery rate (%R) and non-recoverable compliance (Jnr) with RA content.

The AASHTO M332 standard (AASHTO, 2018) establishes the following traffic classification based on Jnr: standard traffic (Jnr3.2 ≤ 4.5 kPa^{-1}), heavy traffic (Jnr3.2 ≤ 2.0 kPa^{-1}), very heavy traffic (Jnr3.2 ≤ 1.0 kPa^{-1}), and extremely heavy traffic (Jnr3.2 ≤ 0.5 kPa^{-1}). According to this classification, the Jnr found with an estimated RA content of 10.6% would be classified as standard traffic, and an RA content below 8.8% would produce a binder suitable for heavy traffic. To stay within the standard traffic limit, the RA content would need to be less than 11.7%. The Jnr3.2 for the Pen 50/70 resulted in 5.24 kPa^{-1}, which is not suitable for standard traffic according to the aforementioned standard. The RA content that would reestablish this value in the aged binder would be 12.2%, but, by adopting such high content, the performance would worsen since it would make the binder unfit for standard traffic. It can also be noted that the %R for the Pen 50/70 resulted in a negative value, which demonstrates test conditions (defined by the AASHTO standard) for itself exceeded the effective stress range of the material.

4.4. Fatigue damage tolerance

The summarized results of LAS fatigue tolerance analysis are presented in Table 4 and Figure 7. The table shows the number of cycles for failure at amplitudes of 2.5, 5.0, and 15.0% since other amplitudes follow the same trend. Fatigue life of rejuvenated asphalt increases exponentially as the dosage of RA increases (Cao et al., 2018), and it is worth mentioning that amplitudes above 10% are more representative of fatigue life assessment (Chen, Zhang and Bahia, 2021).

Table 4: Summary of the results of the linear amplitude scanning (LAS) test.

Sample	Parameter A	Parameter B	Nf (2.5%)	Nf (5.0%)	Nf (15.0%)	initial G*/sin δ	initial G*
Pen 50/70	63,811	-2.74	5,162	770	38	13.69	18.24
RAP + 0%	378,021	-4.43	6,538	304	2	31.50	57.81
RAP + 3%	265,818	-3.72	8,787	666	11	17.01	27.05
RAP + 5%	305,170	-3.46	12,800	1,162	26	12.40	18.55
RAP + 7%	370,139	-3.40	16,365	1,547	37	9.81	14.25
RAP + 10%	230,397	-2.95	15,409	1,991	78	4.96	6.52

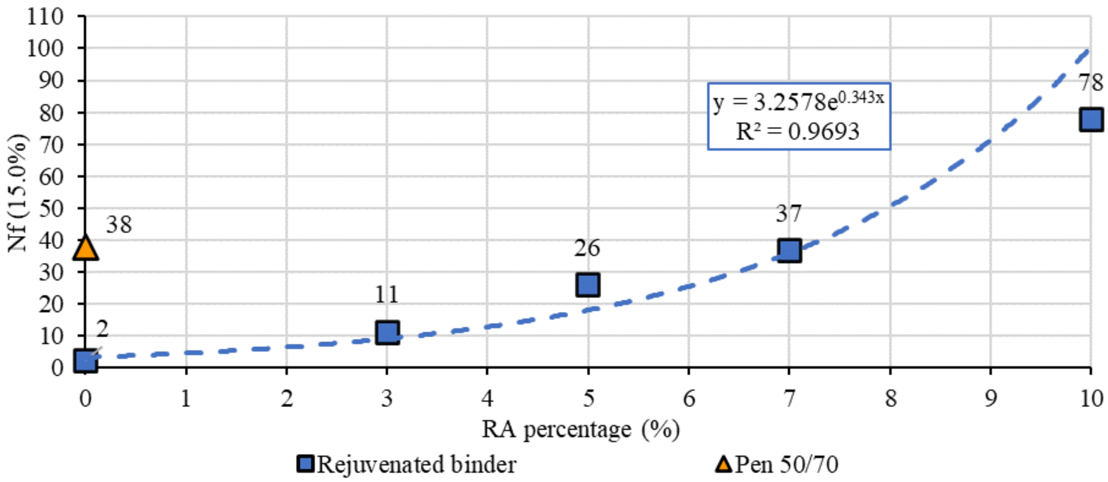

Figure 7. Fatigue life of both virgin and rejuvenated binders at 15% amplitude.

From the results it can be seen that the binders' fatigue damage tolerance (represented by Nf) increased with the addition of RA; this improvement is proportional to the amount of RA added. Considering that the test is performed with short-term aged (RTFOT) samples, it can be inferred that, in practice, the RA's effects would remain satisfactory after asphalt mixture production and compaction processes. Analyzing the Nf results for the 15.0% strain amplitude (Figure 7), for example, a content close to 7.0% (Nf = 37) would already be able to recover the same fatigue life of the virgin binder (Nf = 38). In this range of deformation, results are almost linear until reaching a content of 7.0%, with a greater jump for a 10.0% content. In 30.0% amplitude results, the same graphical analysis showed that a content slightly above 8.0% would be sufficient to recover Pen 50/70's fatigue life. The resulting accuracy for this amplitude, however, would not be adequate for the analysis, considering that the rupture would occur within a few cycles.

Figure 8 presents the fatigue life curves of virgin binder and RAP binder with the different contents of RA, at all the amplitudes evaluated. It can be seen that, at amplitudes above 15.0%, only the straight line for the 10% content exhibited improved behavior to Pen 50/70 in terms of damage tolerance. Hence, the PGH estimated content (10.6%) would result in a binder with adequate behavior to fatigue, surpassing virgin binder's performance.

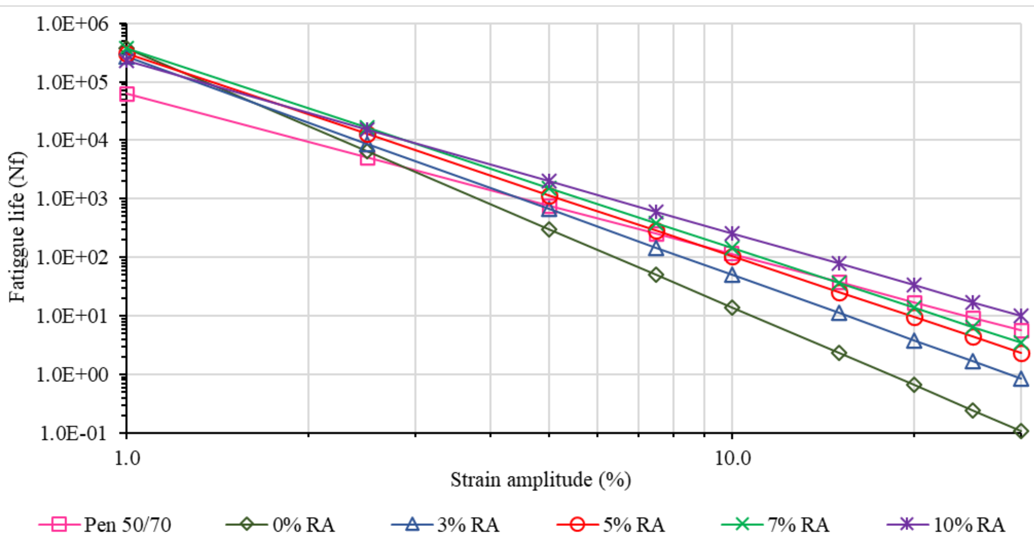

Figure 8. Fatigue life of both virgin and rejuvenated binders.

5. CONCLUSION

This research presented the effects of adding different levels of RA on the rheological properties of the RAP extracted binder. Dosing was carried out using the PGH recovery method of the virgin binder (CAP/ 50/70). The results of rheological characterization tests carried out at DSR (PG, MSCR, and LAS) allowed the following conclusions:

Higher RA contents are required to recover the virgin binder's PGH than for its penetration recovery. The estimated RA content required to recover PGH was 10.6%, which is 3.8 times higher than the content required for penetration recovery. This percentage of RA (10.6%) would result in a binder whose viscosity would be too low, with a penetration of around 145 x 0.1 mm. However, binder's performance to non-recoverable compliance, estimated through the MSCR test, proved to be satisfactory for standard traffic and even superior to that of Pen 50/70. The RA content threshold for the rejuvenated binder to meet standard traffic would be 11.7%, while the RA content needed to match the Pen 50/70's results would be 12.2%.

As expected, the binders' fatigue damage tolerance increased with RA addition; this improvement was proportional to the added RA content. Levels above 7.0% would already be able to match virgin binder's resistance. The estimated content of 10.6% exceeded this resistance.

Thus, it is possible to state that dosing RA by the virgin binder's PGH would be a suitable method for recovering RAP extracted binder's fatigue damage tolerance and non-recoverable compliance. Even after short-term aging, the method improved RAP binder's behavior to the Pen 50/70 virgin binder. However, when evaluating the properties of susceptibility to permanent deformation and fatigue life of rejuvenated binders, it is safer to use lower contents — considering that contents from 7.0% to 8.0% in this research would be enough to raise the aged binder's performance to that of a Pen 50/70 virgin binder, in terms of fatigue life. This RA content reduction would result in savings regarding the production of asphalt mixtures compared to direct dosage by the PGH and would bring greater gains in terms of susceptibility to permanent deformation, considering that lower content would result in superior binders, suitable for heavy traffic.

Further investigation into the interaction between asphalt binders and tall oils is suggested, in order to improve RA efficiency and rejuvenated binders' performance. Furthermore, analysis of binder behavior in terms of fractures and moisture sensitivity is also suggested, as a complement to the performance evaluation.

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