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Rheological investigation of asphalt binder modified with soybean oil sludge

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ABSTRACT: The utilization of alternative materials to asphalt binder in pavement aims to diminish the consumption of natural resources and energy, and in some cases, enhance pavement performance. This study thus examined the effects on physical and rheological properties of asphalt binder with 50/70 penetration, modified by soybean oil sludge at percentages of 1%, 3%, 5%, 7%, and 9% by binder weight. This modification was aimed at reducing viscosity for application in recycled asphalt mixtures. The asphalt binder was assessed through rotational viscosity tests, Performance Grade (PG), and Multiple Stress Creep Recovery in natura sludge and post-drying process. Results indicated that adding sludge led to increased viscosity in the asphalt binder. The modifier also couldn't lower the PG temperature of the pure binder. The modified binders exhibited greater susceptibility to permanent deformation, except for adding in natura sludge at 9% content. Consequently, soybean oil sludge didn't act as a viscosity-reducing material and thus didn't exhibit a rejuvenating effect for use in recycled asphalt mixtures.
Keywords: recycled mixtures; rejuvenating agent; viscosity; waste.

Investigação reológica do ligante asfáltico modificado com a borra do óleo de soja

RESUMO: A utilização de materiais alternativos ao ligante asfáltico na pavimentação tem por finalidade reduzir o consumo de recursos naturais, de energia e, em alguns casos, melhorar o desempenho do pavimento. Assim, este trabalho investigou os efeitos nas propriedades físicas e reológicas do ligante asfáltico com penetração 50/70 modificado pela borra do óleo de soja nos teores de 1%, 3%, 5%, 7% e 9% por peso do ligante, empregada como o objetivo de reduzir a viscosidade para o uso em misturas asfálticas recicladas. O ligante asfáltico foi avaliado por meio de testes de viscosidade rotacional, *performance grade* (PG) e *Multiple Stress Creep Recovery,* modificado com a borra *in natura* e após processo de secagem. Os resultados apontaram que a adição da borra gerou um ganho de viscosidade ao ligante asfáltico. O modificador também não foi capaz de reduzir a temperatura de PG do ligante puro. Os ligantes modificados se apresentaram mais suscetíveis à deformação permanente, com exceção da adição da borra *in natura* no teor de 9%. Assim, a borra de óleo de soja não se configurou como um material redutor de viscosidade, e com isso, não demonstrou um efeito rejuvenescedor para uso em misturas asfálticas recicladas.
Palavras-chave: agente rejuvenescedor; misturas recicladas; viscosidade; resíduos.

1. INTRODUCTION

In Brazil, approximately 95% of paved roads rely on asphalt coating. According to Zhang et al. (2018), the asphalt mixture is the predominant material for road surface layers globally. The key ingredient in producing this mixture is asphalt binder, a viscoelastic binder derived from petroleum. As Benachio et al. (2020) highlighted, road pavements consume around 30% of natural resources and contribute to 25% of construction industry-generated solid waste. This waste typically ends up in landfills.

Flexible pavements require maintenance, preservation, and reconstruction over their lifespan. During reconstruction, material from the asphalt surface layer is termed Reclaimed Asphalt Pavement (RAP). RAP has significant recycling potential, offering reduced environmental impact and costs when used as aggregate, leading to substantial reductions in Greenhouse Gas (GHG) emissions and energy consumption related to the pavement sector (AURANGZEB et al., 2014). Consequently, reusing RAP is crucial for formulating new asphalt mixtures (SILVA et al., 2012).

When incorporating RAP into asphalt mixtures at high levels exceeding 30%, the use of Rejuvenating Agents (RA) becomes necessary. These agents soften the rigid binder, offsetting RAP's inherent stiffness. The utilization of rejuvenating agents, termed bio-oils, sourced from organic plant materials and plant oil residues, has garnered attention among modern researchers. These modifiers have demonstrated notable outcomes, particularly restoring asphalt binder properties, including enhanced crack resistance and reduced binder viscosity (SANTOS; FAXINA, 2019).

These endeavors aim to introduce alternative materials to asphalt binders, reducing natural resource consumption, lowering energy usage, and, in some cases, improving pavement performance (PORTUGAL, 2016). In this context, Zargar et al. (2012) explored using residual cooking oil to rejuvenate aged asphalt binder. The authors observed a progressive decrease in complex shear modulus for the analyzed samples (virgin, aged, and rejuvenated binder with 1%, 2%, 3%, 4%, and 5% modifiers) as temperature increased from 30°C to 80°C. Additionally, the aged binder displayed a lower phase angle than virgin asphalt binder, increasing with the addition of residual cooking oil. The rheological and chemical analysis led the authors to conclude that residual cooking oil acts as a rejuvenator for aged binders, offering an environmentally and economically viable solution for waste reuse.

The literature features studies employing post-consumer residual cooking oil as a rejuvenating agent (SUN et al., 2017; LI et al., 2021; YAN et al., 2021). Melo Neto et al. (2022a) explored the use of fatty acid from soybean oil sludge, the residue after acidulation with hydrochloric acid. The authors indicated a rejuvenating effect on aged asphalt binders, although the production process requires appropriate treatment for the generated acidic water.

This research, therefore, investigated the application of in natura soybean oil sludge, without acidulation, as a viscosity-reducing agent for asphalt binder. Addressing the gaps in knowledge regarding the use of industrial soybean oil refining process residues as rejuvenating agents, this study examined the impact of adding in natura soybean oil sludge, both before and after oven-drying, on the rheological behavior of 50/70 penetration-grade asphalt binder at 1%, 3%, 5%, 7%, and 9% content levels by binder weight.

Recycling this residue, a byproduct of the neutralization process in soybean oil production, as a modifying agent for asphalt binder, benefits from its rich content of saponified fatty acids, cost-effectiveness, and wide availability in soybean oil and biodiesel industries, presenting itself as a sustainable raw material choice for reducing asphalt binder viscosity (SEIDEL; HADDOCK, 2014).

2. EXPERIMENTAL PROGRAM

The procedures implemented in this study adhered to the guidelines outlined by the American Society for Testing and Materials (ASTM) and the American Oil Chemists Society (AOCS). Each test was conducted in triplicate, and the average of the results for each test was provided.

2.1. Materials

The asphalt binder used in this study was supplied by Cordilheira Company, located in Campina Grande - PB. This binder was classified as a penetration grade of 50/70 with a maximum performance grade (PG) temperature of 64°C, representing the predominant binder type used in the Northeast region of Brazil. All tests conducted for the unmodified binder (AB) were also carried out on asphalt binder samples modified with soybean oil sludge.

Various characterization tests were conducted, including penetration, softening point, rotational viscosity, and performance grade (PG) assessments. These tests were performed on both virgin and aged binder samples through the Rolling Thin-Film Oven (RTFO) short-term aging process. Viscosity evaluations were executed using a Brookfield viscometer, while rheological analyses were carried out using a Dynamic Shear Rheometer (DSR) from the Discovery Hybrid Rheometer (DHR-1) series. The asphalt binder characterization outcomes are summarized in Table 1.

Table 1. Binder characterization.
Tabela 1. Caracterização do ligante.

Tests Conducted	Results (AB)	Standards Utilized
Penetration 0.1 mm (100g, 5s at 25ºC)	58.00	ASTM D5/D5M (2020)
Softening Point (ºC)	52.00	ASTM D36/D36M – 14 (2020)
Rotational Viscosity (cP)	135ºC – 401.00 150ºC – 198.00 177ºC – 72.75	ASTM D4402/D4402M (2015)
Maximum PG Temperature (°C)	64.00	ASTM D6373 (2021)
RTFO (Rolling Thin-Film Oven)		ASTM D2872 (2019)
Rotational Viscosity (cP)	135ºC – 557.50 150ºC – 269.00 177ºC – 94.00	ASTM D4402/D4402M (2015)
Maximum PG Temperature (°C)	64.00	ASTM D6373 (2021)
Multiple Stress Creep Recovery (MSCR)	Jnr at 0.1 kPa – 3.40 Jnr at 3.2 kPa – 5.30 % Recovery at 0.1 kPa – 5.03 % Recovery at 3.2 kPa – 0.37	ASTM D7405 (2020)

For the rejuvenating agent, soybean oil sludge from IMCOPA company located in Paraná was employed. The characteristic properties of this material were determined using tests specified by the American Oil Chemists Society (AOCS) and methodologies outlined by Da Fré (2009) and Araújo (2016), as detailed in Table 2.

Table 2. Characterization of soybean oil sludge.
Tabela 2. Caracterização da borra de óleo de soja.

Tests Conducted	Results	Standards Utilized
Free fatty acids in oleic acid (%)	0.68	AOCS Ca 5a-40 (2017)
Total fatty acids content (%)	41.59	AOCS G 3-53 (2017)
Oxidized fatty acids content (%)	1.22	AOCS G 3-53 (2017)
Unsaponifiable matter content (%)	0.87	AOCS Ca 6a-40 (2017)
Neutral oil content (%)	12.44	AOCS G5-40 (2017)
pH at 25°C	9.96	AOCS G 7-56 (2017)
Moisture and volatile content (%)	41.85	AOCS Ca 2c-25 (2017)

Based on the data presented in Table 2, the total content of fatty acids was determined to be 41.59%, which falls within the range of 35% to 50%, as stipulated by Swern (1982). Notably, the moisture content of the material exceeded 40%, a feature that could impact the outcomes of binder modification. The pH test conducted at 25°C indicated that the material exhibits a basic or alkaline nature, as evidenced by its pH value exceeding 7.

2.2. Methods

2.2.1. Drying of soybean oil sludge

As indicated in Table 2, the soybean oil sludge displayed a notable moisture content of 41.85%, constituting nearly half the total sludge added to the binder. This prompted the consideration that the inclusion of 1% sludge into the binder would effectively mean adding 0.58% actual sludge content, with the remaining 0.42% accounting for the moisture present in the sludge. In response, a drying protocol was implemented, as outlined in Figure 1, facilitating the examination of the behavior of the binder modified with both in natura and dried sludge.

This protocol was executed at the Pavement Engineering Laboratory (LEP) of the Federal University of Campina Grande (UFCG) over a span of 72 hours (3 days). Initially, two samples were weighed, positioned on aluminum foil, and subjected to drying in an oven set at a temperature of 65°C. After 17 hours, the samples underwent another weighing process. This weighing procedure was repeated at 40, 43, 65, 68, and 72 hours. The protocol concluded after 72 hours of drying, during which approximately 35% of the moisture content was extracted from the soybean oil sludge samples. Considering the boiling point of water at 100°C, exceeding the temperature employed in the protocol, a more extended duration would be necessary to eliminate moisture from the soybean oil sludge entirely. As such, the practical implementation of this material as a modifying agent for asphalt binder within large-scale asphalt mixture production is logistically unviable. The samples displayed the subsequent masses during the various weighings (Table 3).

Figure 1. Soybean oil sludge drying process.
Figura 1. Processo de secagem da borra de óleo de soja.

2.2.2. Modification of asphalt binder

For the study, percentages of 1%, 3%, 5%, 7%, and 9% of soybean oil sludge based on asphalt binder weight were examined, falling within the modifier range established by He *et al.* (2017). The FISATOM model 722D mechanical shaker was employed for the mixing process (modifier + binder). Initially, the binder was heated to 135°C for 90 minutes to achieve fluidity. Following preheating, the material was transferred to a Becker flask and positioned on the mechanical shaker at a rotation speed of 600 rpm to ensure thorough mixture homogeneity. As the temperature reached 140°C, the additives were added separately based on the pure binder weight, and the rotation speed was increased to 1000 rpm for 30 minutes for the 1%, 3%, 5%, 7%, and 9% contents. This technique was adopted based on studies by Faxina (2006), Souza (2012), and Melo Neto (2022). Table 4 presents the descriptions of the samples and their respective nomenclatures.

Table 3. Nomenclature of samples used in the research.
Tabela 3. Nomenclatura das amostras utilizadas na pesquisa.

Drying period	Sample 1 (g)	Sample 2 (g)
Without drying	288.76	288.18
17 hours	245.35	245.70
40 hours	214.11	216.19
43 hours	210.91	212.94
65 hours	194.33	196.79
68 hours	192.54	194.96
72 hours	190.59	193.03

Table 4. Nomenclature of samples used in the research.
Tabela 4. Nomenclatura das amostras utilizadas na pesquisa.

Samples	Nomenclatures
Penetration-grade 50/70 asphalt binder	AB
AB + 1% in natura soybean oil sludge	1% SS
AB + 3% in natura soybean oil sludge	3% SS
AB + 5% in natura soybean oil sludge	5% SS
AB + 7% in natura soybean oil sludge	7% SS
AB + 9% in natura soybean oil sludge	9% SS
AB + 1% dried soybean oil sludge	1% DSS
AB + 3% dried soybean oil sludge	3% DSS
AB + 5% dried soybean oil sludge	5% DSS
AB + 7% dried soybean oil sludge	7% DSS
AB + 9% dried soybean oil sludge	9% DSS

2.2.3. Test procedures

The modified asphalt binders were characterized at the Pavement Engineering Laboratory (LEP) of the Federal University of Campina Grande (UFCG). The samples underwent testing before and after the Rolling Thin Film Oven (RTFO) aging procedure (ASTM D2872, 2019) without any treatment and after the drying process.

Rotational viscosity analyses (ASTM D4402, 2015) involved placing the samples within a controlled-temperature container with a rotating rod set at a specified speed. The force needed to overcome the resistance posed by the binder's viscosity was determined based on the recorded rotation during the test.

For the Performance Grade (PG) test (ASTM D6373, 2021), the parameter G*/senδ was examined. The initial temperature was set at 46°C, with subsequent increments to temperatures of 52°C, 58°C, and 64°C. The maximum test temperature corresponded to the point of binder failure.

The Multiple Stress Creep Recovery (MSCR) test (ASTM D7405, 2020) was conducted at the PG temperatures of the asphalt binder. The goal was to compare pure binder samples with modified binder samples. This test assesses that lower Jnr values at 3.2 kPa indicate more viscous binders, while higher values suggest more fluid materials.

3. RESULTS

This section presents the outcomes obtained from the physical and rheological characterization of the asphalt binders modified in natura soybean oil sludge and after the drying procedure to eliminate moisture.

3.1. Rotational Viscosity

Table 5 displays the findings of the rotational viscosity assessment for the pure binders (AB) and binders altered with in natura soybean oil sludge (SS). The procedure adhered to the ASTM D4402 (2015) standard and aimed to ascertain the binder's viscosity and consistency.

Table 6 illustrates the viscosity outcomes for the pure binders and binders that underwent modification with dried soybean oil sludge (DSS).

Table 5. Results of the rotational viscosity test within natura sludge.
Tabela 5. Resultados do teste de viscosidade rotacional com borra *in natura*.

		Rotational Viscosity (cP)		
	°C	135.00	150.00	177.00
Tests Before RTFO	AB	401.00	198.00	72.75
	1%SS	397.50	194.00	71.25
	3%SS	356.25	181.50	69.50
	5%SS	354.30	182.50	680.00
	7%SS	428.75	211.00	104.75
	9%SS	446.25	182.50	72.25
	Limits	≥274.00	≥112.00	57.00 at
		Rotational Viscosity (cP)		
	°C	135.00	150.00	177.00
Tests After RTFO	AB	548.00	269.00	94.00
	1%SS	553.75	264.50	91.75
	3%SS	496.25	240.50	85.25
	5%SS	563.75	268.50	98.25
	7%SS	558.75	272.00	103.25
	9%SS	593.75	286.50	99.50
	Limits	≥55.00	NA	NA

NA = Not Applicable. NA = Não Aplicável.

Table 6. Results of the rotational viscosity test with dried sludge.
Tabela 6. Resultados do teste de viscosidade rotacional com borra seca.

		Rotational Viscosity (cP)		
	°C	135.00	150.00	177.00
Tests Before RTFO	AB	404.43	200.34	72.24
	1%DSS	398.53	193.65	71.06
	3%DSS	457.50	201.27	74.17
	5%DSS	674.57	248.47	119.61
	7%DSS	879.34	251.52	115.16
	9%DSS	401.00	198.00	73.00
	Limites	≥274.00	≥112.00	57.00 at
		Rotational Viscosity (cP)		
	°C	135.00	150.00	177.00
Tests After RTFO	AB	535.00	257.00	89.50
	1%DSS	518.48	249.24	88.82
	3%DSS	584.55	278.74	98.32
	5%DSS	705.53	326.60	114.25
	7%DSS	974.50	418.78	155.42
	9%DSS	548.00	269.00	94.00
	Limites	≥55.00	NA	NA

NA = Not Applicable. NA = Não Aplicável.

For both pre and post RTFO conditions, only the binder containing 1% sludge exhibited viscosity values lower than those of the pure asphalt binder. As the additive content increased, viscosity also increased. These findings differ from those obtained with the asphalt binder modified with soybean oil sludge fatty acid. Melo Neto (2022) reported a linear decrease in asphalt binder viscosity with increasing fatty acid content.

3.2. Performance Grade (PG)

In the performance grade tests, the values corresponding to the minimum PG temperatures were excluded due to the negligible likelihood of negative temperatures in tropical countries like Brazil. Figures 2 and 3 illustrate the variations in the G*/Sinδ parameter values across temperatures ranging from 46°C to 64°C. These figures pertain to the asphalt binders modified with in natura and dried soybean oil sludge, respectively, before and after the short-term aging process.

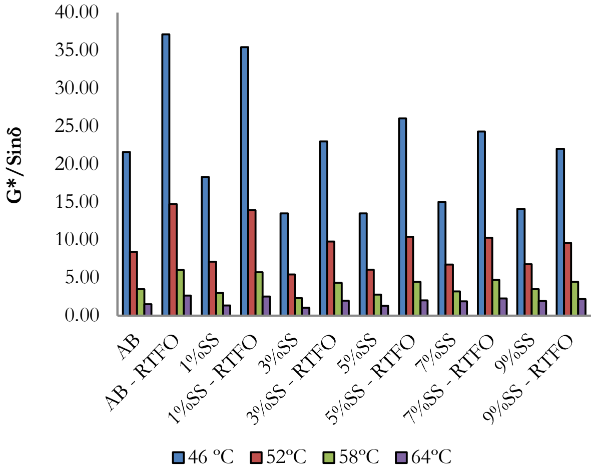


Figure 2. G*/Sinδ parameter versus temperature before and after short-term aging RTFO for binders modified with in natura sludge.
Figura 2. Parâmetro G*/Senδ versus temperatura antes e após o envelhecimento a curto prazo RTFO para os ligantes modificados com a borra *in natura*.

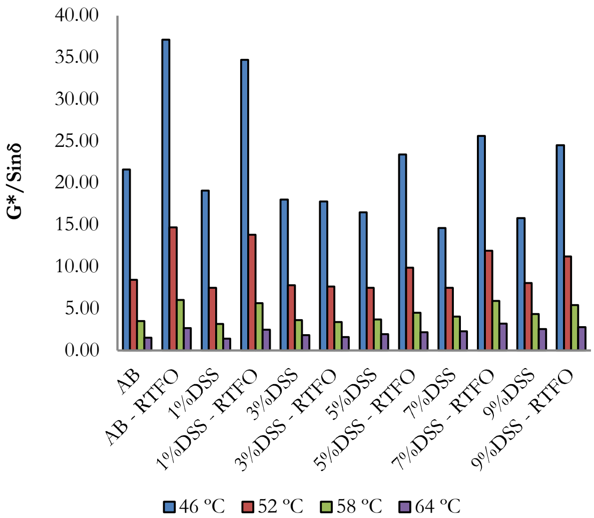
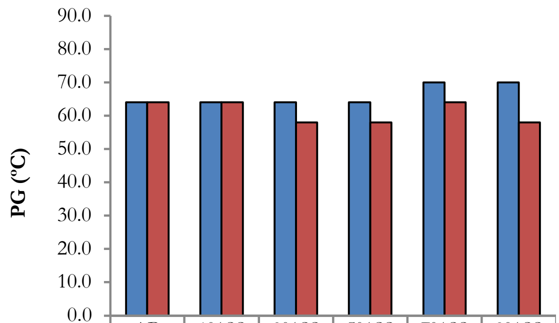


Figure 3. G*/Sinδ parameter versus temperature before and after short-term aging RTFO for binders modified with dried sludge.
Figura 3. Parâmetro G*/Senδ versus temperatura antes e após o envelhecimento a curto prazo RTFO para os ligantes modificados com a borra seca.

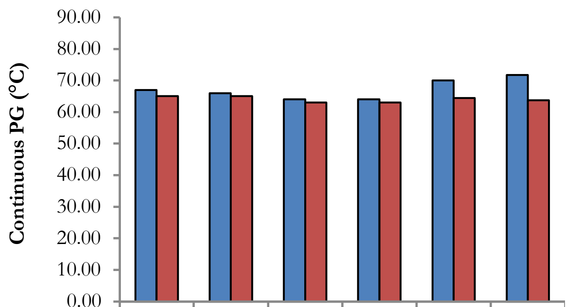
From the information depicted in Figures 2 and 3, it is evident that the G*/Sinδ parameter of the pure binders, both pre and post RTFO, exceeded those attained with the addition of soybean oil sludge under both considered conditions and lower temperatures (46°C and 52°C). At the temperature of 64°C, before the short-term aging process, the asphalt binder modified with in natura sludge at contents of 7% and 9% exhibited values surpassing those of the pure binder. The latter recorded a value of 1.53, while the former marked 1.87 and 1.88, respectively.

Regarding the binder amended with dried sludge, at the temperature of 64°C, binders containing percentages exceeding 3% displayed G*/Sinδ values surpassing the pure binder before RTFO. Figures 4 and 5 provide a graphical representation of the PG and continuous PG values for the pure asphalt binders and those modified with in natura soybean oil sludge, encompassing conditions before and after the short-term aging RTFO procedure.



	AB	1%SS	3%SS	5%SS	7%SS	9%SS
Before RTFO	64.00	64.00	64.00	64.00	70.00	70.00
After RTFO	64.00	64.00	58.00	58.00	64.00	58.00

Figure 4. Performance Grade of pure asphalt binders and binders modified with in natura soybean oil sludge before and after short-term aging RTFO.
Figura 4. Grau de desempenho dos ligantes asfálticos puro e modificados com a borra do óleo de soja *in natura* antes e após o envelhecimento a curto prazo RTFO.

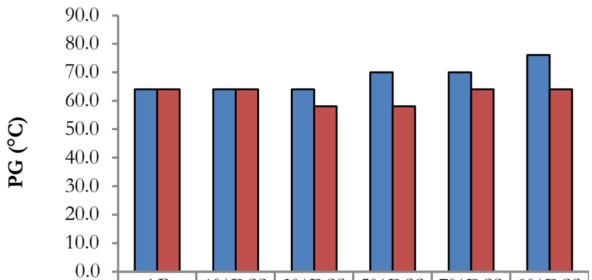


	AB	1%SS	3%SS	5%SS	7%SS	9%SS
Before RTFO	67.00	66.00	64.00	64.00	70.00	71.70
After RTFO	65.00	65.00	63.00	63.00	64.40	63.70

Figure 5. Continuous Performance Grade of pure asphalt binders and binders modified with in natura soybean oil sludge before and after short-term aging RTFO.
Figura 5. PG contínuo dos ligantes asfálticos puro e modificados com a borra do óleo de soja *in natura* antes e após o envelhecimento a curto prazo RTFO.

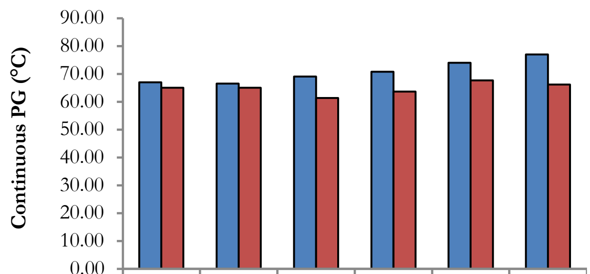
A discernible pattern emerges after thoroughly examining the outcomes depicted in Figure 4. The asphalt binder, subject to modification with 3% SS and 5% SS, exhibits a notable reduction in PG temperature post-aging, resulting in a shift from 64°C to 58°C. This shift signifies an increased susceptibility to oxidative effects, which, in turn, contributes to reduced deformability and enhanced rigidity at elevated temperatures. The specimen treated with 7% SS showcases a similar PG temperature shift following RTFO, marking a decrease of one interval, corresponding to a 6°C range, and descending from 70°C to 64°C. Conversely, the 9% SS sample demonstrates a more pronounced reduction, spanning two intervals and descending from 70°C to 58°C.

Figures 6 and 7 graphically depict the PG and continuous PG values of the unmodified pure asphalt binders and those that underwent modification by incorporating dried soybean oil sludge. These graphical representations encompass conditions before and after the short-term aging RTFO procedure, providing a comprehensive visual overview of the evolving performance of the binder.



	AB	1%DSS	3%DSS	5%DSS	7%DSS	9%DSS
Before RTFO	64.00	64.00	64.00	70.00	70.00	76.00
After RTFO	64.00	64.00	58.00	58.00	64.00	64.00

Figure 6. Performance Grade of pure asphalt binders and binders modified with dried soybean oil sludge before and after short-term aging RTFO.
Figura 6. Grau de desempenho dos ligantes asfálticos puro e modificados com a borra do óleo de soja seca antes e após o envelhecimento a curto prazo RTFO.



	AB	1%DSS	3%DSS	5%DSS	7%DSS	9%DSS
Before RTFO	67.00	66.60	69.10	70.80	74.00	77.00
After RTFO	65.00	65.00	61.40	63.70	67.70	66.20

Figure 7. Continuous Performance Grade of pure asphalt binders and binders modified with dried soybean oil sludge before and after short-term aging RTFO.
Figura 7. PG contínuo dos ligantes asfálticos puro e modificados com a borra do óleo de soja seca antes e após o envelhecimento a curto prazo RTFO.

Continuing the analysis concerning Figure 6, it becomes evident that including dried soybean oil sludge leads to consistent PG values when employing 1% DSS (64°C). Nonetheless, upon the incorporation of 3% sludge, there is a discernible reduction in PG temperature after the short-term aging RTFO assessment. Notably, the sample treated with 5% DSS displays an intriguing pattern, indicating an elevation in PG value pre-RTFO from 64°C to 70°C, encompassing a

6°C range. In cases involving the application of natural soybean oil sludge, an intriguing discrepancy at the 5% threshold emerges: the pre-RTFO value is 64°C for the 5%SS sample, whereas it registers at 70°C for the 5%DSS sample. This discrepancy suggests a more notable increase in asphalt binder stiffness with sludge utilization post the drying procedure. These findings bolster the observations from the rotational viscosity tests, reiterating that the drying process insignificantly interferes with soybean oil sludge's efficacy as a viscosity-reducing agent in asphalt binder.

Turning attention to the 7%DSS and 9%DSS samples, there surfaces an elevated PG before RTFO. Following the aging examination, both the 7%DSS and 9%DSS samples manifest a reduction in continuous PG temperature, recording reductions of 6.3°C and 10.8°C, respectively. This comprehensive analysis intimates that introducing soybean oil sludge yields negligible effects on the PG value when juxtaposed against the unadulterated binder sample.

Table 7 shows the temperature deviations prior to and post RTFO between PG and continuous PG values for the specimens under scrutiny. Notably, the transition from PG to continuous PG spans a spectrum of 0 to 5°C, findings that parallel the investigation by Melo Neto *et al.* (2022b) involving the analysis of the physical-rheological dynamics of asphalt binders modified with refined cotton oil. Their study echoes similar temperature variations, with the authors emphasizing that the amplitude of 6°C in PG variation also translates to the determination of lower performance grades than the authentic ones.

Table 7. Variation of PG and Continuous PG temperatures in asphalt binder samples.
Tabela 7. Variação das temperaturas de PG e PG contínuo nas amostras dos ligantes asfálticos.

Samples		Before RTFO	After RTFO
AB	(°C)	3.00	1.00
	(%)	4.69	1.56
1%SS	(°C)	2.00	1.00
	(%)	3.13	1.56
3%SS	(°C)	0.00	5.00
	(%)	0.00	8.62
5%SS	(°C)	0.00	5.00
	(%)	0.00	8.62
7%SS	(°C)	0.00	0.40
	(%)	0.00	0.63
9%SS	(°C)	1.70	5.70
	(%)	2.43	9.83
Samples		Before RTFO	After RTFO
AB	(°C)	3.00	1.00
	(%)	4.69	1.56
1%DSS	(°C)	2.60	1.00
	(%)	4.06	1.56
3%DSS	(°C)	5.10	3.40
	(%)	7.97	5.86
5%DSS	(°C)	0.80	5.70
	(%)	1.14	9.83
7%DSS	(°C)	4.00	3.70
	(%)	5.71	5.78
9%DSS	(°C)	1.00	2.20
	(%)	1.32	3.44

As depicted in Table 7, a notable observation emerges, underscoring the 7%SS sample for exhibiting the most minimal variance in both PG and continuous PG values. This finding designates it as the most indicative and faithful representation within the analyzed set. Remarkably, the PG classification value for this specific sample closely aligns with its actual failure point, signifying its heightened accuracy and reliability.

3.3. Multiple Stress Creep Recovery (MSCR)

The multiple stress creep and recovery test serves as a valuable tool for assessing critical parameters such as recovery percentage (%R), non-recoverable compliance (Jnr), and the percentage difference between non-recoverable compliances (Jnr, diff). These metrics provide insights into elasticity, susceptibility to permanent deformation accumulation, and sensitivity to increased stress levels. The test facilitates the determination of suitable traffic levels for the binder and its recovery behavior under the applied stresses corresponding to the chosen levels. Notably, higher Jnr values indicate a heightened susceptibility to permanent deformations.

Figures 8 and 9 impeccably portray the test outcomes conducted at the asphalt binder's maximum PG temperature (64°C) and its elastic recovery concerning binders modified with in-nature soybean oil sludge, respectively.

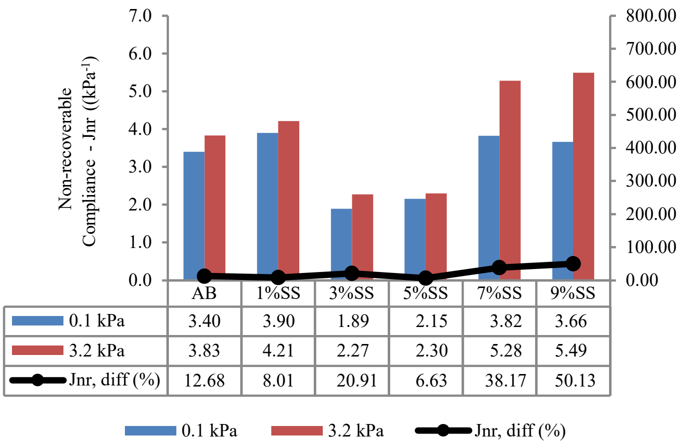


Figure 8. Non-recoverable compliances of pure and soybean oil sludge-modified binders.
Figura 8. Compliâncias não recuperáveis dos ligantes puro e modificados com a borra de óleo de soja *in natura*.

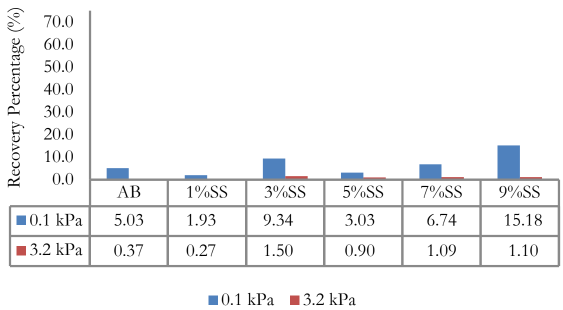


Figure 9. Elastic recovery at 0.1 kPa and 3.2 kPa for pure and soybean oil sludge-modified binders.
Figura 9. Recuperação elástica a 0.1 kPa e 3.2 kPa para os ligantes puro e modificados com a borra de óleo de soja *in natura*.

According to AASHTO M320 (2021), a relationship can be established between the values obtained for Jnr at 3.2 kPa and the traffic grade to which the binder belongs. This classification is shown in Table 8. Based on the values obtained for Jnr at 3.2 kPa, binders with 1%, 3%, and 5% can be classified for standard traffic use. However, the additions of 7%SS and 9%SS had Jnr values above the specified limit, making them unsuitable for highway use.

Table 8. Classification of traffic level based on Jnr values.
Tabela 8. Classificação do nível de tráfego com base nos valores de Jnr.

Property	Jnr (kPa)	Traffic Type	Traffic level
Jnr up to 3.2 kPa at maximum PG temperature	2.0 - 4.0	Standard	< 10 million
	1.0 - 2.0	Heavy	> 10 million
	0.5 - 1.0	Very Heavy	> 30 million
	0.0 - 0.5	Extremely Heavy	> 100 million

When analyzing the recovery percentage, as shown in Figure 9, it can be observed that the highest (%R) values are for the additions of 3%SS, 7%SS, and 9%SS, with values above those of the pure binder, both for Jnr at 0.1 kPa and Jnr at 3.2 kPa. Figures 10 and 11 present the Jnr test data conducted at the maximum PG temperature of the asphalt binder (64°C) and the elastic recovery for binders modified with dried soybean oil sludge.

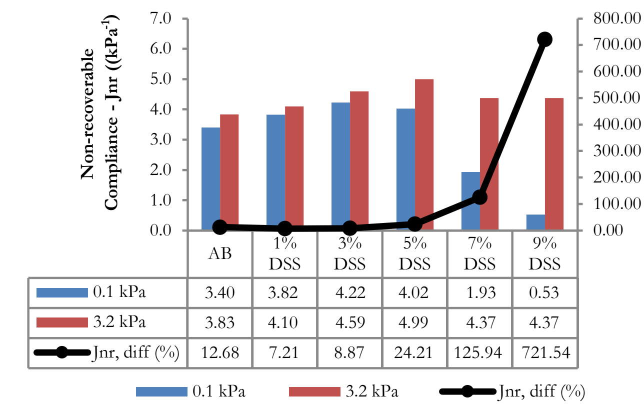


Figure 10. Non-recoverable compliances of pure binders and binders modified with dried soybean oil sludge.
Figura 10. Compliâncias não recuperáveis dos ligantes puro e modificados com a borra de óleo de soja seca.

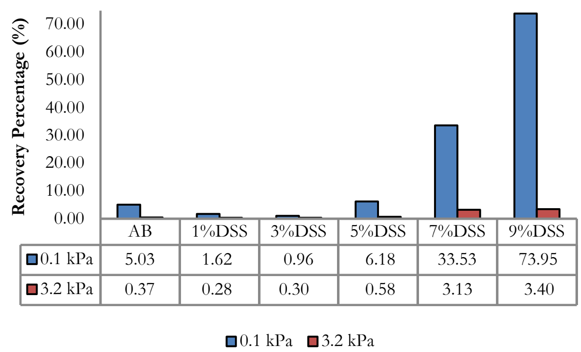


Figure 11. Elastic recovery at 0.1 kPa and 3.2 kPa for pure binders and binders modified with dried soybean oil sludge.
Figura 11. Recuperação elástica a 0.1 kPa e 3.2 kPa para os ligantes puro e modificados com a borra de óleo de soja seca.

According to Figure 10, it can be observed that with the incorporation of dried soybean oil sludge at 1%, 3%, and 5%, the values of Jnr at 3.2 kPa tend to increase.

Regarding the percentages of 7%DSS and 9%DSS, a decrease in the value of Jnr at 3.2 kPa is noticeable compared to the previous percentages. This behavior was unexpected due to the stiffness gain observed in the rotational viscosity and PG tests for these additions.

4. DISCUSSION

4.1. Rotational Viscosity

Based on the results presented in Table 5, a reduction in asphalt binder viscosity was observed for additions of up to 5% soybean oil sludge (SS), particularly at the lower test temperatures, consistent with the findings reported by Melo Neto et al. (2022a). However, 7% and 9% increased viscosity compared to the pure asphalt binder before the short-term aging procedure (RTFO). Following the RTFO test, only the 3% addition exhibited lower viscosity than the pure binder at all three temperatures. Overall, all percentages yielded results within the limits stipulated by the standard.

In the case of binders modified with dry soybean oil sludge, the viscosity increased with higher additive percentages. The study aimed to establish soybean oil sludge as a potential viscosity reducer, and the test results indicate that adding up to 5% raw sludge ensures this reduction. However, samples with dry sludge suggest that values lower than 1% can achieve this viscosity reduction. Given that raw sludge contains a notable moisture content, the effective added amount of soybean oil sludge is lower than the actual quantity employed.

According to Silva et al. (2022), as viscosity decreases relative to the pure binder, the action of the modifying agent will consequently influence compaction and mixing temperatures, which are linked to the workability of the binder.

4.2. Performance Grade (PG)

The results presented in Figures 2 and 3 allowed us to ascertain that following the addition of crude soybean oil sludge (in natura) and dried sludge, as well as when subjected to higher temperatures, there is increased development of stiffness in the asphalt binder. For lower temperatures, these findings indicate that incorporating the modifier contributes to reducing the stiffness of the binders. Furthermore, it's noteworthy that for both crude and dried sludge, there was an increase in the G*/Senδ parameter with short-term aging, albeit consistently maintaining values lower than those of the pure sample. This phenomenon arises from the samples becoming stiffer after RTFO, with parameters around twice as high as those obtained before the procedure, a behavior also observed in the studies by Portugal (2016) and Melo Neto et al. (2022b).

It's worth noting that in the case of adding dried sludge, the fact that this parameter is lower than that of the pure binder diverges from the values obtained in the viscosity tests. This suggests that there may have been incomplete homogeneity in the mixture, leading to non-representative results, or that this modifying agent may behave as a viscosity reducer under low temperatures.

An increase was observed regarding the PG values for the binders modified with both crude and dried sludge, as presented in Figures 4 and 6. This increase could be attributed to the high moisture content present in the material (approximately 40%). As the amount of sludge added to the asphalt binder increases, the water content also increases, which could have oxidized the asphalt binder and increased its stiffness.

4.3. Multiple Stress Creep Recovery (MSCR)

Regarding the samples of modified binder derived from crude sludge, based on the Jnr values presented in Figure 8, it can be observed that when applying stress levels of 0.1 kPa and 3.2 kPa, the binders modified with 3%SS and 5%SS exhibited the lowest values of non-recoverable compliance compared to the other modified binders. These values were also lower than those obtained for the binder without additives. The reduction of this parameter indicates a less fluid binder and, thus, a lower susceptibility to permanent deformation, which contradicts the rotational viscosity test results indicating a reduction in material stiffness. The samples 7%SS and 9%SS had Jnr values at 3.2 kPa above the specified norm, rendering them unsuitable for use, according to AASHTO M320 (2021). High Jnr values at 3.2 kPa indicate a more fluid asphalt binder and, consequently, a binder more susceptible to permanent deformation.

As for the differential Jnr values (Jnr diff), it can be observed that for all tested samples, the Jnr diff values were below the limit of 75%, indicating that the samples are suitable for use at this temperature range.

Regarding the percentage of recovery shown in Figure 9, as mentioned by Mendonça et al. (2022), the Multiple Stress Creep Recovery (MSCR) percentage can identify and quantify the effect of the additive in the binder. Based on this, it is noted that the additions of 3%SS, 7%SS, and 9%SS significantly influence the elastic characteristics of the binder under high traffic levels.

As for the binders modified with dry sludge, according to Figure 10, it was observed that for additions of 1%, 3%, and 5%, the Jnr values at 3.2 kPa tend to increase. This indicates that adding material at these percentages to the pure binder increases its stiffness and reduces its susceptibility to permanent deformation.

However, it is important to note that these results are for a temperature of 64°C above the RTFO-aged PG temperature obtained for these samples (58°C). This might have led to oxidation and increased consistency. These inconsistent values could result from a lack of chemical interaction between the asphalt binder and the dry soybean oil sludge. This absence of interaction prevents a homogeneous mixture from forming, rendering the samples unrepresentative and invalidating the data or the material's use in pavement applications.

In terms of the percentages of 7%DSS and 9%DSS, a decrease in the value of Jnr at 3.2 kPa was observed, a behavior unexpected due to the stiffness gain observed in rotational viscosity and PG tests for these additions. Concerning their recovery percentages, these samples are classified as inadequate due to having values significantly above 75%, as shown in Figure 11.

5. FINAL CONSIDERATIONS

Based on the obtained results, it can be concluded that using soybean oil sludge as a viscosity-reducing agent is not viable. The modified samples exhibited higher stiffness than the pure binder. The PG temperatures before the aging process were higher than the reference values, but after aging, the found values were equal to or lower than the reference values. Thus, the modifier was not effective in reducing the PG temperature.

In the case of raw soybean oil sludge, adding this modifier reduced susceptibility to permanent deformation at the 9% level. Concerning this material, it was noted that the high moisture content present in the soybean oil sludge could have contributed to the oxidation process of the asphalt binder during modification and short-term aging, resulting in increased stiffness.

Such uncertainties led to analyzing the binder modified with dried soybean oil sludge. The results showed that additions of up to 5% provided viscosity values equivalent to those of the pure binder. In comparison, percentages above 7% increased the stiffness of the asphalt binder. Like the raw sludge, the dried sludge did not reduce the PG temperature, rendering it unsuitable as a rejuvenating agent for recycled asphalt mixtures.

Furthermore, removing moisture from the soybean oil sludge samples increased the materials' sensitivity to elevated stress levels, making the modified binder more susceptible to deformation under increased load, as demonstrated in the MSCR test. Thus, the hypothesis that removing moisture from the sludge would improve its performance in the asphalt binder was disregarded.

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