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Utilizing Moringa and Sunflower Oils as Sustainable Additives for Reduced Viscosity and Lower Working Temperatures in Asphalt Binder

Uso de aceites de moringa y girasol como aditivos sostenibles para reducir la viscosidad y las temperaturas de trabajo en el ligante asfáltico

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Abstract

In efforts to mitigate greenhouse gas (GHG) emissions, the use of vegetable oils as additives in asphalt binders has gained significant interest. However, the limited availability of vegetable oil in certain regions poses challenges, as its use must not compromise critical applications like food production. To encourage the adoption of vegetable oils in asphalt pavements, it is crucial to diversify their use across different regions, such as Brazil, which offers favorable conditions for cultivating various oilseeds like soy, palm, cotton, sunflower, and moringa. This study aims to evaluate the effectiveness of incorporating moringa and sunflower oils at concentrations of 1%, 1.5%, 2%, and 2.5% by weight of the asphalt binder in reducing binder viscosity and achieving lower working temperatures during mixture preparation and compaction. An analysis of variance (ANOVA) was conducted to assess the significance of the data. The results demonstrate that incorporating vegetable oils reduced asphalt binder viscosity, mixing temperature, and compaction temperature. Moringa oil showed the most substantial reductions, with a decrease of 18.3°C in compaction

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temperature and 13.8°C in mixing temperature at a 2.5% concentration. Additionally, the use of vegetable oils effectively reduced the binder's susceptibility to aging while maintaining its resistance to permanent deformation. An optimal content of 2% vegetable oil was identified for pavement applications. This research highlights the potential of vegetable oils, particularly moringa and sunflower oils, as sustainable and effective additives for modifying asphalt binders, contributing to reduced environmental impact and promoting more sustainable infrastructure practices. Through blending charts, it was found that incorporating 2.5% of these vegetable oils allows the use of approximately 43% RAP in recycled asphalt mixtures.

Keywords: ANOVA; Bio-oils; Greenhouse Gases; Rheology; Sustainability.

Resumen

En los esfuerzos por mitigar las emisiones de gases de efecto invernadero (GEI), el uso de aceites vegetales como aditivos en ligantes asfálticos ha despertado un interés significativo. Sin embargo, la disponibilidad limitada de aceites vegetales en ciertas regiones representa un desafío, ya que su utilización no debe comprometer aplicaciones críticas como la producción de alimentos. Para fomentar la adopción de aceites vegetales en pavimentos asfálticos, es fundamental diversificar su uso en distintas regiones, como Brasil, que ofrece condiciones favorables para el cultivo de diversas oleaginosas, tales como soya, palma, algodón, girasol y moringa. Este estudio tiene como objetivo evaluar la efectividad de la incorporación de aceites de moringa y girasol en concentraciones del 1%, 1.5%, 2% y 2.5% en peso del ligante asfáltico, con el fin de reducir su viscosidad y alcanzar temperaturas de trabajo más bajas durante la preparación y compactación de mezclas. Se realizó un análisis de varianza (ANOVA) para evaluar la significancia estadística de los datos. Los resultados demostraron que la adición de aceites vegetales redujo la viscosidad del ligante, así como las temperaturas de mezclado y compactación. El aceite de moringa presentó las reducciones más significativas, con una disminución de 18.3 °C en la temperatura de compactación y de 13.8 °C en la temperatura de mezclado, en la concentración del 2.5%. Además, el uso de aceites vegetales fue eficaz para reducir la susceptibilidad al envejecimiento del ligante, manteniendo su resistencia a la deformación permanente. Se identificó un contenido óptimo del 2% de aceite vegetal para aplicaciones en pavimentos. Esta investigación destaca el potencial de los aceites vegetales, en particular los de moringa y girasol, como aditivos sostenibles y eficaces para la modificación de ligantes asfálticos, contribuyendo a la reducción del impacto ambiental y fomentando prácticas de infraestructura más sostenibles. A través de diagramas de combinación, se determinó que la incorporación del 2.5% de estos aceites vegetales permite el uso de aproximadamente un 43% de RAP en mezclas asfálticas recicladas.

Palabras clave: Economía circular; sostenibilidad; resistencia a la tracción; diseño factorial; resistencia a la compresión; índice de circularidad de materiales.

1. Introduction

The research on enhancing asphalt pavement through the addition of various types of oils has been extensively studied due to its significant impact on the country's economy and overall development [1-3]. The surface layer of asphalt mixtures in flexible pavements is widely used due to its viscoelastic properties, ability to provide a smooth surface, and effective adhesion to aggregates during the mixing process [4]. However, the construction of asphalt pavements relies heavily on non-renewable resources [5]. The availability and sustainable allocation of natural resources, responsible extraction of raw materials, environmental impacts of human activities, and increasing pollution are major concerns across various socioeconomic sectors [6]. Consequently, the paving industry is being urged to adopt more sustainable practices, recognizing the environmental impacts associated with the production and construction processes of its products [7].

The Intergovernmental Panel on Climate Change (IPCC), the United Nations body responsible for assessing climate change science, recently released a report in March 2023 highlighting promising advancements in low carbon technologies. While countries are making more ambitious commitments to reduce emissions and support communities in adapting to climate change, current efforts are still insufficient to limit global warming to 1.5°C above pre-industrial levels, the threshold deemed necessary to avoid the worst impacts of climate change. Climate adaptation measures are also fragmented, often overlooking vulnerable communities. Furthermore, continued global warming may trigger irreversible changes in ecosystems worldwide, posing catastrophic consequences for both human populations and biodiversity that depend on them [8]. Given these challenges, the pursuit of sustainable technologies becomes increasingly critical.

The utilization of vegetable oils as modifiers for asphalt binders has garnered considerable attention in recent years, primarily due to their renewable nature [9]. These oils present sustainable alternatives as recycling agents (RAs) [10], contributing to the reduction of greenhouse gas (GHG) emissions associated with paving processes. They can be employed as modifiers (up to 10%), extenders (25% to 75%), or substitutes (100%) in relation to the total weight of the asphalt binder [11]. The incorporation of vegetable oils in binders leads to a decrease in viscosity, resulting in lower mixing and compaction temperatures during the production of asphalt mixtures [12]. This development has paved the way for the adoption of Warm Mix Asphalt (WMA), which is manufactured at intermediate temperatures between Hot Mix Asphalt (HMA) and Cold Mix Asphalt, typically ranging from 100°C to 150°C. Numerous studies [13–23] have substantiated the efficacy of incorporating bio-oils in reducing binder viscosity.

Moringa Oleifera Lam, a perennial tree legume well-suited to arid and semi-arid conditions, is widely distributed in several countries, including India, Egypt, the Philippines, Thailand, Malaysia, and Brazil [24]. Introduced in Brazil in the 1950s, it has gained prominence within the region known as the "drought polygon" in the country's northeast. Moringa seeds have shown potential for water treatment due to their coagulant properties, making them suitable for domestic use [25, 26]. Given its renewable and biodegradable nature, Moringa Oleifera Lam oil can serve as an environmentally friendly additive. Its antioxidant characteristics and surface-active properties make it a promising viscosity reducer for binders. The presence of oleic acid contributes to its oxidative resistance, enhancing mixture performance [27]. Moringa oil holds merit as its multiple applications are not primarily intended for large-scale human consumption, unlike soybean oil in Brazil.

Sunflower oil, ranked fourth globally among vegetable oils, has potential for pavement applications. With a 9.5% share in production and total supply, its global supply is expected to reach 22.90 million tons in 2022-2023, mainly led by Russia, the European Union, and Ukraine. In Brazil, sunflower grain production has grown significantly, reaching 64.1 thousand tons in 2022-2023. Sunflower oil primarily consists of triacylglycerols (98-99%) with a high content of unsaturated acids (~83%) but low levels of linolenic acid (≤0.2%) [28]. While the use of vegetable oils as additives has shown technical feasibility, their availability should be assessed to ensure it doesn't impact other essential uses, such as food. This study focuses on incorporating Moringa and Sunflower oils (at various concentrations) as viscosity reducers in asphalt binders to lower working temperatures (mixing and compaction) of asphalt mixtures. Statistical analysis of variance (ANOVA) was employed to determine data significance.

2. Experimental Program

This section outlines the materials and tests utilized in the research, adhering to the guidelines established by the American Association of State Highway and Transportation Officials (AASHTO) and the American Society for Testing and Materials (ASTM).

2.1. Materials

The sunflower oil used in this study was obtained from the local market, while the moringa oil was provided by the UFS (Federal University of Sergipe). Moringa oil, known for its suitability in arid and semi-arid climates, was selected for this research considering the climate conditions in Brazil. Both oils exhibited a density of 0.92 g/cm³ and rotational viscosity of 45.3 cP at 40°C for moringa oil and 31.8 cP for sunflower oil. These viscosity values decrease with increasing temperature, as observed by Almeida [29] and Canciam [30]. The asphalt binder used in this study was a 50/70 penetration grade commonly used in Brazil.

2.2. Methods

2.2.1. Mixing procedure

The process of modifying the asphalt binder followed established methodologies that incorporated vegetable oils as additives [6, 31]. To ensure thorough homogenization of the binder and oil, a mechanical mixer operating at a constant temperature of 135°C and 600 rpm was employed for a duration of 20 minutes. The oils were gradually introduced during the initial 5 minutes. Based on recommendations from the literature [12, 21–23] for viscosity reduction in 50/70 penetration virgin asphalt binder, four different concentrations of 1.0%, 1.5%, 2.0%, and 2.5% by weight of binder were utilized. It is worth noting that Moringa oil exhibits a high content of oleic acid (>70%) [32, 33], whereas sunflower oil contains approximately 62% [34], indicating relatively low unsaturation levels. Oils rich in oleic acid demonstrate improved stability against oxidation, both at ambient and high temperatures, during the mixing process. However, it is crucial to consider that oils have the potential to decrease the consistency of the binder, which may impact its quality and the performance of asphalt mixtures. Therefore, it is essential to determine appropriate proportions based on the concentration of the oil and the consistency of the base asphalt binder [35].

2.2.2. Experimental tests

A comprehensive set of experiments was conducted to assess the characteristics of the asphalt binders, and the findings are summarized in Table 1.

The assessment of binder performance was conducted in accordance with the SUPERPAVE guidelines. The rheological properties were evaluated using the Oscillatory Rheometer Discovery HR-1 hybrid rheometer. A master curve was generated to illustrate the rheological behavior of the asphalt binder at a specific temperature (25°C), enabling a comprehensive understanding of its characteristics across a broad range of frequencies or durations of load application. The differentiation of temperature and loading frequency effects was accomplished through temperature horizontal displacement curves and master curves. It is assumed that temperature, time, and frequency exert an equal influence on the rheological properties [42].

2.3. Statistical analysis

The gathered data underwent ANOVA statistical analysis to ascertain their significance. This analysis evaluates whether there exists a statistically substantial disparity amid the observed means and determines the influence of variables on the model. The F value (Fcalc) was computed using Equation 1 and juxtaposed against the tabulated F value (Ftab). If the calculated F value surpasses the tabulated F value, it indicates a noteworthy divergence among the observed means; contrarily, if it is lower, there is no considerable difference. A significance level of 5% was adopted for this test, representing the probability of accurately accepting or rejecting the hypothesis.

$$F_{calc} = \frac{MS}{MS\ error} \quad (1)$$

Where: MS is Mean Square, and MS error is the Mean Square Error.

3. Results and Discussions

The asphalt binder samples without any additives were denoted as AB, whereas the samples with the incorporation of sunflower oil and moringa oil were identified as SO and MO, respectively. The precise quantity of vegetable oil added to each binder sample was indicated following the respective acronym. To ensure the accuracy and consistency of the findings, all experiments were carried out in duplicate.

3.1. Physical analysis

Table 2 illustrates the physical characteristics of the specimens. The outcomes indicate that the penetration value of the pure asphalt binder escalates with an increasing concentration of vegetable oils. This observation aligns with prior research [21, 22, 31, 43] and can be attributed to the viscosity-lowering effect exerted by vegetable oils. Among the samples, solely those with a 1% oil content complied with the prescribed range of 50-70 penetration according to the ASTM D5M [36] standard. A similar trend manifested in the softening point examination, wherein greater proportions of oil in the binder resulted in diminished consistency, as evidenced by lower softening point values. The samples with a 2.5% oil content demonstrated a reduction of up to 9.38% in softening point relative to the pure asphalt binder.

The viscosity results obtained in this investigation are consistent with the findings reported by Melo Neto et al. [31], indicating a decrease in viscosity with increasing vegetable oil content, particularly at lower temperatures during testing. The aging process led to an increase in viscosity for all tested samples. However, the incorporation of moringa and sunflower oils demonstrated a softening effect, resulting in lower viscosity values. Bio-oils are renowned for their ability to rejuvenate asphalt binders [44].

Table 1. Test characteristics of asphalt binder

Test	Standard Test Method
Penetration	ASTM D5M [36]
Softening Point	ASTM D36 [37]
Rotational Viscosity	ASTM D4402 [38]
Performance Grade (PG)	ASTM D6373 [39]
Thin Film Oven Test (RTFOT)	ASTM D2872 [40]
Multiple Stress Creep and Recovery (MSCR)	ASTM D7405 [41]

Table 2. Physical characterization of asphalt binders

Tests Before RTFOT												
Results												
		AB	SO (1%)	SO (1.5%)	SO (2%)	SO (2.5%)	MO (1%)	MO (1.5%)	MO (2%)	MO (2.5%)	Limits	Standard
Penetration 0.1 mm (100g, 5s at 25ºC)		52	64	77	87	94	69	77	84	94	50 to 70	ASTM D5M [37]
Softening Point (ºC)		48.0	47.5	45	45	43.5	46.5	45.5	44.5	43.5	NA	ASTM D36 [38]
Rotational Viscosity (cP)	135 °C	402.5	307.5	277.5	272.5	250.0	275.0	257.5	237.5	227.5	≥274	ASTM D4402 [39]
	150°C	203	163	152	149	139	149	144	134	132	≥112	
	177 °C	70.5	53.5	50.0	50.5	47.0	47.5	45.5	42.5	42.5	57 to 285	
Tests After RTFOT												
Binder mass variation (%)		0.08	0.07	0.05	0.09	0.05	0.07	0.08	0.05	0.06	≤ 0.5	ASTM D2872 [40]
Penetration 0.1 mm (100g, 5s at 25ºC)		35	49	53	55	61	60	68	73	81	NA	ASTM D5M [36]
Softening Point (ºC)		52.0	50.0	48.0	47.0	47.0	49.25	48.0	48.0	46.5	NA	ASTM D36 [37]
Increase in Softening Point (°C)		4.00	2.50	3.00	2.00	3.50	2.75	2.50	3.50	3.00	≤8	ASTM D36 [37]
Retained Penetration (%)		67	77	69	63	65	87	89	86	86	≥55	ASTM D5M [36]
Rotational Viscosity (cP)	135 °C	542.0	440.0	415.0	405.0	355.0	425.0	382.5	350.0	292.5	NA	ASTM D4402 [38]
	150°C	203.0	163.0	152.0	149.0	139.0	149.0	144.0	134.0	132.0	NA	
	177 °C	86.0	69.5	66.5	67.0	59.0	65.5	59.5	53.5	48.0	NA	

NA = Not Applicable.

Notably, the addition of sunflower oil yielded lower viscosity values both before and after aging, with more pronounced effects observed at oil concentrations of 2% and 2.5%.

Among the samples, only the AB, SO (1%), SO (1.5%), and MO (1%) specimens exhibited viscosity values within the specifications of the standard at a temperature of 135°C. All samples displayed values above the limit at 150°C and below the limit at 177°C, further confirming the viscosity-reducing impact of the evaluated oils. The retained penetration, which measures the binder's susceptibility to aging, was expressed as a percentage ratio between the penetration values before and after the Rolling Thin Film Oven Test (RTFOT). Higher values indicate lower sensitivity of the binder to aging. In this study, all samples exhibited retained penetration values surpassing the specified limit of 55%. Notably, samples containing moringa oil displayed higher retained penetration values compared to those incorporating sunflower oil, suggesting that moringa oil contributed to reducing the binder's susceptibility to aging.

The mass loss results presented in Table 2 revealed that volatilization had a more significant impact than oxidation on the binder. The pure binder exhibited a higher loss of lighter fractions compared to the mass gain resulting from oxidation during the aging process. However, the incorporation of moringa and sunflower oils led to smaller mass losses, indicating their ability to mitigate the imbalance between volatilization and oxidation in the binder. The reduction in asphalt binder viscosity contributes to the observed decrease in working temperatures, including mixing and compaction.

Table 3. Binder working temperatures

Samples	Temperature of Compaction (°C)	Reduction (°C)	Temperature of Mixing (°C)	Reduction (°C)
AB	140.8	NA	154.8	NA
SO (1%)	133.3	7.5	148.8	6.0
SO (1.5%)	130.0	10.8	146.4	8.4
SO (2%)	129.3	11.5	146.0	8.8
SO (2.5%)	126.0	14.8	143.5	11.3
MO (1%)	129.8	11.0	146.3	8.5
MO (1.5%)	127.5	13.3	145.0	9.8
MO (2%)	124.5	16.3	142.0	12.8
MO (2.5%)	122.5	18.3	141.0	13.8

NA = Not Applicable.

The rotational viscosity data guided the determination of the design temperature for asphalt mixture production, which should be 0.17±0.02 Pa.s [31]. The working temperatures were determined through interpolation and are detailed in Table 3.

The incorporation of sunflower oil led to a notable reduction in working temperatures, with a decrease of 11.3°C for mixing and 14.8°C for compaction at the maximum oil content of 2.5%. The utilization of moringa oil resulted in even greater reductions, with decreases of 13.8°C for mixing and 18.3°C for compaction at the 2.5% oil content. Warm asphalt mixtures typically operate at temperatures below 150°C [45]. All samples containing added oils exhibited reduced viscosity and working temperatures below 150°C, indicating the potential use of these oils as additives for warm mixtures. However, it is crucial to conduct advanced rheological analyses to evaluate the binder's performance when incorporating these oils, a topic that will be discussed in the following section. To statistically evaluate the results, an ANOVA test was conducted, and the corresponding parameters are presented in Table 4.

The ANOVA test demonstrated the significance of the RTFOT aging process as well as the type and content of oil as influential variables. The p-values obtained for all tested temperatures and tests were below the 5% significance level, with Fcalc values surpassing Ftab. These results indicate that the calculated F values fall within the rejection region of the null hypothesis, indicating a statistically significant difference between the observed means. Consequently, it can be inferred that the aging process (RTFOT) has a substantial effect on increasing the consistency and viscosity of the asphalt binder. Furthermore, the incorporation of oils (moringa and sunflower) in their respective effective amounts (1%, 1.5%, 2%, 2.5%) significantly contributes to reducing the consistency and viscosity of the binder.

3.2. Rheological properties

The rheological test results are presented in Table 5, showcasing the acquired data. Complex modulus (G*) is a crucial rheological parameter that characterizes the viscoelastic behavior of asphalt binder, reflecting its ability to resist deformation under applied stress. This parameter is typically assessed at various temperatures and frequencies to evaluate binder performance under different conditions. The Performance Grade (PG) system is used to classify asphalt binders based on their expected performance at different temperatures, providing information on both high-temperature and low-temperature properties. The PG system assigns temperature grades, such as PG 64-22, where the first number represents the high-temperature performance grade and the second number represents the low-temperature performance grade. Rheological properties, including complex modulus (G*), play a significant role in determining the performance grade. In this study, the evaluation of the binder's low-temperature properties was not conducted due to the prevailing climatic conditions in Brazil, where temperatures average around 25°C, eliminating the need for low-temperature analysis. However, based on the data presented in Table 5, it can be stated that the samples exhibit potential for field application based on their physical properties. The continuous PG values were consistent across all samples containing oil and similar to those of the pure binder, indicating that the addition of oil did not adversely affect the high-temperature performance.

Table 4. Statistical parameters for penetration tests, softening point, and rotational viscosity

Penetration						
Aging (RTFOT)		**Score**	**Amount**	**Average**	**Variance**	
Not aged		9	698	77.55556	197.7778	
Aged		9	535	59.44444	186.5278	
Type/content of vegetable oil		**Score**	**Amount**	**Average**	**Variance**	
AB		2	87	43.5	144.5	
SO (1%)		2	113	56.5	112.5	
SO (1.5%)		2	130	65	288	
SO (2%)		2	142	71	512	
SO (2.5%)		2	155	77.5	544.5	
MO (1%)		2	129	64.5	40.5	
MO (1.5%)		2	145	72.5	40.5	
MO (2%)		2	157	78.5	60.5	
MO (2.5%)		2	175	87.5	84.5	
Variation source	**SQ**	**gl**	**MQ**	**F_{calc}**	**p-value**	**F_{tab}**
Aging (RTFOT)	1476.056	1	1476.056	33.59975	0.000407	5.317655
Type/content of vegetable oil	2723	8	340.375	7.748024	0.004488	3.438101
Error	351.4444	8	43.93056	NA	NA	NA
Total	4550.5	17	NA	NA	NA	NA

Softening point						
Aging (RTFOT)		**Score**	**Amount**	**Average**	**Variance**	
Not aged		9	409	45.44444	2.590278	
Aged		9	435.75	48.41667	3.03125	
Type/content of vegetable oil		**Score**	**Amount**	**Average**	**Variance**	
AB		2	100	50	8	
SO (1%)		2	97.5	48.75	3.125	
SO (1.5%)		2	93	46.5	4.5	
SO (2%)		2	92	46	2	
SO (2.5%)		2	90.5	45.25	6.125	
MO (1%)		2	95.75	47.875	3.78125	
MO (1.5%)		2	93.5	46.75	3.125	
MO (2%)		2	92.5	46.25	6.125	
MO (2.5%)		2	90	45	4.5	
Variation source	**SQ**	**gl**	**MQ**	**F_{calc}**	**p-value**	**F_{tab}**
Aging (RTFOT)	39.75347	1	39.75347	208.1636	5.21×10^{-7}	5.317655
Type/content of vegetable oil	43.44444	8	5.430556	28.43636	4.29×10^{-5}	3.438101
Error	1.527778	8	0.190972	NA	NA	NA
Total	84.72569	17	NA	NA	NA	NA

Rotational Viscosity (135°C)						
Aging (RTFOT)		**Score**	**Amount**	**Average**	**Variance**	
Not aged		9	2507.5	278.6111	2725.174	
Aged		9	3607	400.7778	4877.632	
Type/content of vegetable oil		**Score**	**Amount**	**Average**	**Variance**	
AB		2	944.5	472.25	9730.125	
SO (1%)		2	747.5	373.75	8778.125	
SO (1.5%)		2	692.5	346.25	9453.125	
SO (2%)		2	677.5	338.75	8778.125	
SO (2.5%)		2	605	302.5	5512.5	
MO (1%)		2	700	350	11250	
MO (1.5%)		2	640	320	7812.5	
MO (2%)		2	587.5	293.75	6328.125	
MO (2.5%)		2	520	260	2112.5	
Variation source	**SQ**	**gl**	**MQ**	**F_{calc}**	**p-value**	**F_{tab}**
Aging (RTFOT)	67161.13	1	67161.13	207.1276	5.31×10^{-7}	5.317655
Type/content of vegetable oil	58228.44	8	7278.556	22.44736	0.000104	3.438101
Error	2594	8	324.25	NA	NA	NA
Total	127983.6	17	NA	NA	NA	NA

Table 4 (Continuation). Statistical parameters for penetration tests, softening point, and rotational viscosity

Rotational Viscosity (150°C)						
Aging (RTFOT)		**Score**	**Amount**	**Average**	**Variance**	
Not aged		9	1365	151.6667	462	
Aged		9	1847	205.2222	803.4444	
Type/content of vegetable oil		**Score**	**Amount**	**Average**	**Variance**	
AB		2	465	232.5	1740.5	
SO (1%)		2	384	192	1682	
SO (1.5%)		2	362	181	1682	
SO (2%)		2	356	178	1682	
SO (2.5%)		2	326	163	1152	
MO (1%)		2	366	183	2312	
MO (1.5%)		2	342	171	1458	
MO (2%)		2	318	159	1250	
MO (2.5%)		2	293	146.5	420.5	
Variation source	**SQ**	**gl**	**MQ**	**F_{calc}**	**p-value**	**F_{tab}**
Aging (RTFOT)	12906.89	1	12906.89	218.7093	4.3×10^{-7}	5.317655
Type/content of vegetable oil	9651.444	8	1206.431	20.44316	0.000148	3.438101
Error	472.1111	8	59.01389	NA	NA	NA
Total	23030.44	17	NA	NA	NA	NA
Rotational Viscosity (177°C)						
Aging (RTFOT)		**Score**	**Amount**	**Average**	**Variance**	
Not aged		9	449.5	49.94444	72.59028	
Aged		9	574.5	63.83333	117.875	
Type/content of vegetable oil		**Score**	**Amount**	**Average**	**Variance**	
AB		2	156.5	78.25	120.125	
SO (1%)		2	123	61.5	128	
SO (1.5%)		2	116.5	58.25	136.125	
SO (2%)		2	117.5	58.75	136.125	
SO (2.5%)		2	106	53	72	
MO (1%)		2	113	56.5	162	
MO (1.5%)		2	105	52.5	98	
MO (2%)		2	96	48	60.5	
MO (2.5%)		2	90.5	45.25	15.125	
Variation source	**SQ**	**gl**	**MQ**	**F_{calc}**	**p-value**	**F_{tab}**
Aging (RTFOT)	868.0556	1	868.0556	115.848	4.89×10^{-6}	5.317655
Type/content of vegetable oil	1463.778	8	182.9722	24.41891	7.62×10^{-5}	3.438101
Error	59.94444	8	7.493056	NA	NA	NA
Total	2391.778	17	NA	NA	NA	NA

NA = Not Applicable.

This finding is noteworthy because the viscosity decreased, resulting in lower working temperatures during mixing and compaction, without compromising the rheological performance of the binder. The obtained continuous PG value of 61°C when incorporating 3% fatty acid from soybean oil sludge into the same type of asphalt binder, as reported by Melo Neto et al. [31], aligns with our findings. The reduction in the G* parameter observed in the viscosity test corresponds to the loss of rigidity caused by increased oil content, consistent with previous reports [5, 6, 20–23, 31]. Additionally, the inclusion of vegetable oils led to a decrease in the aging index (AI) for all tested samples. The AI, calculated as the ratio of G*/sinδ after and before RTFOT, provides insight into the binder's susceptibility to aging. The observed data support the inference that moringa and sunflower oils effectively reduce the binder's susceptibility to aging [46].

Non-recoverable compliance (Jnr) is a rheological parameter that measures the irreversible deformation or flow of asphalt binder under a constant stress. It quantifies the binder's ability to return to its original shape after deformation, with lower Jnr values indicating better resistance to permanent deformation. The Multiple Stress Creep Recovery (MSCR) test is used to determine the percentage of binder recovery (R%), which assesses the binder's ability to regain its original shape after being subjected to stress and subsequent relaxation. The MSCR test in this study was conducted at the maximum performance grade (PG) temperature for each binder.

The results indicate that the addition of oils to the binder did not have a significant impact on the R% values. This outcome was expected since oils are not intended to enhance elastic recovery, which is typically achieved through the use of polymers as modifiers. However, the incorporation of the two additives led to a reduction in Jnr at both stress levels. Lower Jnr values indicate improved resistance to permanent deformation, while higher Jnr values suggest greater susceptibility. Comparing the samples with the two oils, they exhibited similar Jnr values, which was a consistent finding throughout the study. The Jnrdiff parameter represents the difference between Jnr values at the two stress levels (0.1 kPa and 3.2 kPa). Jnrdiff values exceeding 75% indicate sensitivity of the binder to changes in stress level. The SO (2%) and MO (1.5%) samples exhibited Jnrdiff values below 75%, indicating their susceptibility to stress level variations. According to the AASHTO M 320 standard, which classifies binders based on Jnr at 3.2 kPa, all samples were deemed suitable for standard traffic (S). However, the SO (1%) and MO (1%) samples met the requirements for heavy traffic (H), with Jnr at 3.2 kPa falling between 1 kPa-1 and 2 kPa-1. The statistical significance of the rheological data was determined through the ANOVA test, the results of which are presented in Table 6. This test confirms the significance of the observed differences in the rheological properties between the tested samples.

In the PG test, the variable "aging" was found to be significant with a p-value of 0.005045, which is below the predetermined significance level.

Table 5. Results of rheological tests on the shear rheometer

Tests Before RTFOT										
		AB	SO (1%)	SO (1.5%)	SO (2%)	SO (2.5%)	MO (1%)	MO (1.5%)	MO (2%)	MO (2.5%)
Complex modulus (G*)	46°C	21.2	18.5	16.3	13.7	12.5	17.6	15.4	12.4	10.6
	52°C	8.13	7.24	6.39	5.44	4.99	6.85	6.00	4.90	4.28
	58°C	3.33	3.02	2.68	2.30	2.14	2.84	2.51	2.07	1.83
	64°C	1.46	1.34	1.20	1.04	NA	1.25	1.13	NA	NA
Continuous PG (°C)		65	65	65	65	61	65	65	62	61
Tests After RTFOT										
Complex modulus (G*)	46°C	40.8	29.4	25.4	22.9	21.2	30.9	26.6	22.9	19.0
	52°C	15.6	11.3	9.94	9.03	8.44	12.0	10.4	9.04	7.51
	58°C	6.31	4.75	4.14	3.77	3.58	4.90	4.33	3.82	3.18
	64°C	2.73	NA	NA	NA	NA	NA	NA	NA	NA
Continuous PG (°C)		65	61	61	61	61	61	61	61	61
AI	46°C	1.94	1.60	1.57	1.68	1.71	1.77	1.74	1.85	1.81
	52°C	1.92	1.57	1.56	1.66	1.70	1.76	1.74	1.85	1.76
	58°C	1.91	1.58	1.55	1.64	1.68	1.73	1.73	1.85	1.74
	64°C	1.88	NA	NA	NA	NA	NA	NA	NA	NA
MSCR	Jnr 0.1 kPa	2.89	1.14	1.68	1.21	2.46	1.39	1.24	2.03	1.93
	Jnr 3.2 kPa	3.87	1.70	2.20	2.29	2.79	1.83	2.30	2.65	3.00
	R% 0.1 kPa	17.45	26.18	16.56	41.69	7.92	22.97	39.77	15.33	21.95
	R% 3.2 kPa	1.71	4.16	1.88	3.94	1.97	6.04	3.57	2.21	1.94
	Jnr_{diff}	33.52	49.33	30.70	89.89	13.38	31.69	85.39	30.59	54.95

NA = Not Applicable, PG = Performance grade, AI = Aging Index, RTFOT = Rolling Thin Film Oven, MSCR = Multiple Stress Creep and Recovery, R% = Average recovery.

Table 6. ANOVA test parameters for PG and MSCR rheological assays

Performance Grade Continuous (Continuous PG)						
Aging (RTFOT)		**Score**	**Amount**	**Average**	**Variance**	
Not aged		9	576.3	64.03333	3.45	
Aged		9	555.8	61.75556	1.817778	
Type/content of vegetable oil		**Score**	**Amount**	**Average**	**Variance**	
AB		2	130.8	65.4	0.02	
SO (1%)		2	127	63.5	7.22	
SO (1.5%)		2	126.7	63.35	7.605	
SO (2%)		2	126.2	63.1	7.22	
SO (2.5%)		2	122.8	61.4	0.18	
MO (1%)		2	126.9	63.45	6.845	
MO (1.5%)		2	126.5	63.25	6.845	
MO (2%)		2	122.9	61.45	0.045	
MO (2.5%)		2	122.3	61.15	0.125	
Variation source	**SQ**	**gl**	**MQ**	**F_{calc}**	**p-value**	**F_{tab}**
Aging (RTFOT)	23.34722	1	23.34722	14.64031	0.005045	5.317655
Type/content of vegetable oil	29.38444	8	3.673056	2.303257	0.129605	3.438101
Error	12.75778	8	1.594722	NA	NA	NA
Total	65.48944	17	NA	NA	NA	NA

NA = Not Applicable.

This result supports the findings presented in Table 5, indicating that the continuous PG values for all modified asphalt binder samples remained the same after undergoing the Rolling Thin Film Oven Test (RTFOT). This implies that the aging process did not significantly affect the high-temperature performance of the binders.

However, the variable "type/content of vegetable oil" was not found to be significant, with a p-value of 0.129605, which exceeds the 5% significance level. This suggests that the type and content of vegetable oil did not have a statistically significant impact on the high-temperature performance of the binders.

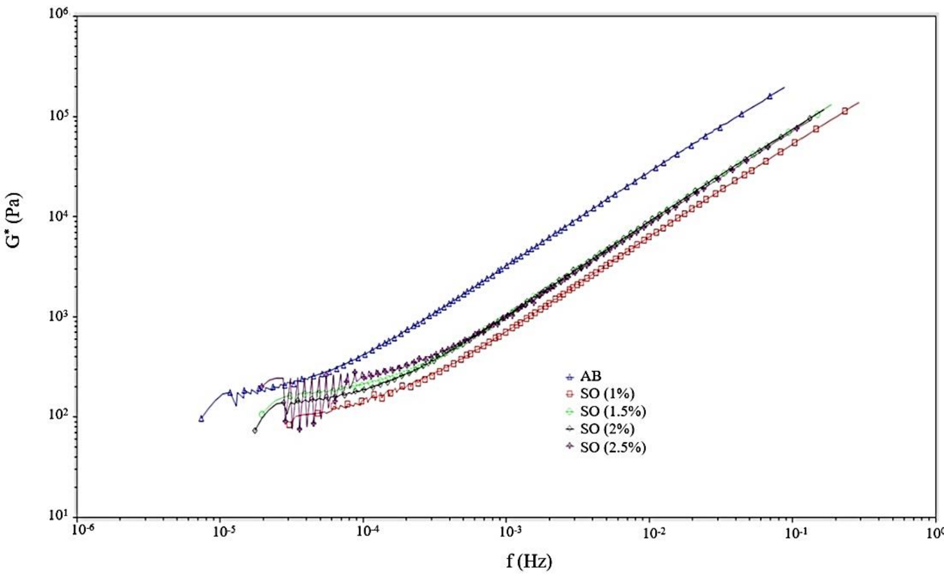
In the MSCR test, both variables, "Jnr" and "aging," were found to be significant, with p-values of 8.31×10^{-5} and 0.000323, respectively. This means that there is a statistically significant difference between the means of the observed samples for these variables. The rejection of the null hypothesis and acceptance of the alternative hypothesis indicates that both Jnr and aging have a significant influence on the resistance to permanent deformation of the binders. Overall, these statistical findings provide valuable insights into the effects of aging, type/content of vegetable oil, and rheological parameters on the performance of the asphalt binders.

Figure 1 illustrates the "Master Curves" generated for the specimens at frequencies ranging from 0.1 to 100 rad/s and temperatures ranging from 46 °C to 76 °C. These graphs show the relationship between the complex modulus (G*) values and the frequency (f) for the specimens incorporating moringa and sunflower oil in this study. Analyzing the master curve for sunflower oil (Figure 1(a)), it can be observed that the curves start to overlap after adding 1.5% or more of the oil. This indicates that the effectiveness of the additive in reducing the workability temperature of the binder is achieved at this level or higher, which is consistent with the decrease in viscosity. The decrease in the G* parameter further confirms the loss of rigidity, which can be attributed to the decrease in viscosity. Similarly, moringa oil also leads to a reduction in the G* parameter (Figure 1(b)). It is interesting to note that the behavior of the binder is very similar for the 2% and 2.5% content levels of moringa oil. However, there are some oscillations observed at the beginning of the curve for the 2.5% content level, indicating that it is more susceptible to thermal variations. These master curves provide valuable information about the rheological behavior of the asphalt binders with the addition of moringa and sunflower oil. They demonstrate the effectiveness of the additives in reducing the workability temperature and increasing the flexibility of the binder.

3.3. 3.3 Recycling rate for asphalt mixture recycled with binder modified with moringa oil and sunflower oil

To assess the potential of vegetable oils as recycling agents for use in recycled asphalt mixtures, a Reclaimed Asphalt (RA) binder was obtained from the Reclaimed Asphalt Pavement (RAP). The RAP was collected from milling operations carried out as part of the Integrated Revitalization Program of Federal Highway BR 230, between kilometers 35.7 and 42 in the Northeastern region of Brazil. The milled material composing the RAP is derived from a Hot Mix Asphalt (HMA). The highway had an average daily traffic volume of approximately 19,685 vehicles/day, with a Single Axle Load Equivalency (ESALs) of 9.37x107. The service life of the milled pavement was 10 years, with a milling depth of 5 cm.

(a)



(b)

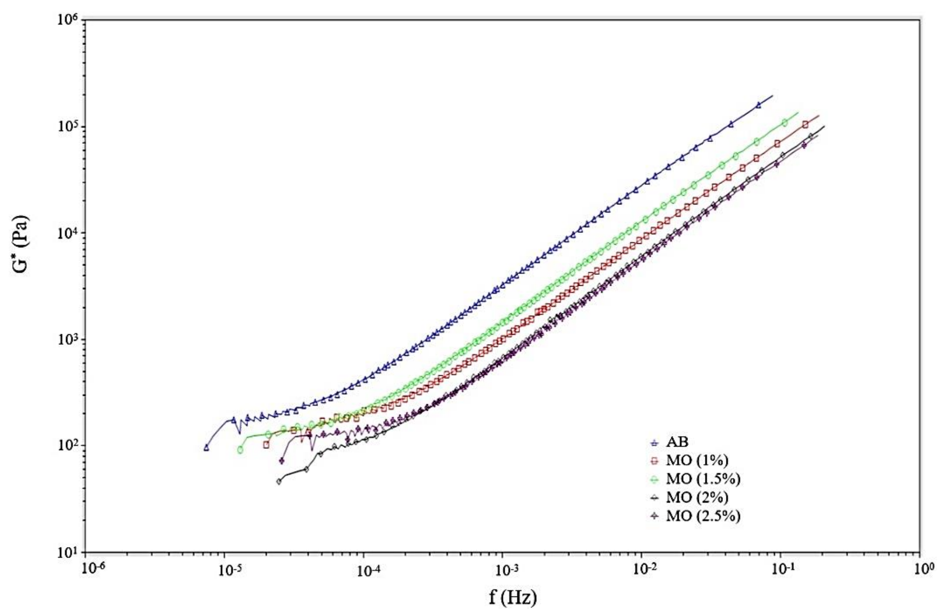


Figure 1. Master curves of asphalt binders with vegetable oils: (a) sunflower oil (SO), (b) moringa oil (MO).

The Rotarex centrifugation method, employing trichloroethylene as the solvent, was utilized in accordance with the guidelines stipulated in ASTM D2172 [47] and DNIT 158 [48] to extract the binder from the RAP aggregate matrix. This approach also facilitated the determination of the presence of aged binder in the RAP, which was quantified at 5.5%. The recovery process was executed utilizing a Rotavapor® R300 Buchi, featuring a V-300 vacuum pump and an I-300 interface, as per the protocols prescribed by ASTM D2172 [47].

The RAP binder solution in trichloroethylene was introduced into the distillation flask, which rotated within a heated water bath at 60°C until the trichloroethylene evaporated, as indicated by the reduction in bubbling, leaving only the RAP binder in the vessel. Subsequently, the RAP binder underwent placement in a vacuum oven at 80°C for 5 hours to eliminate any remaining solvent. Following this, the RAP binder underwent physical and rheological evaluation, conforming to the methods and standards employed for the characterization of binder samples containing vegetable oils. Table 7 provides the data resulting from the RAP characterization.

The asphalt binder exhibited a high PG temperature of 82°C. The substantial stiffness displayed by the RAP indicates the necessity of employing a recycling agent for the asphalt binder (AB) to enhance the performance of recycled asphalt mixtures against fatigue cracking. The elevated viscosity values and low penetration value align with the existing literature [5, 11, 19], confirming the high level of stiffness in the binder present in the RAP. The low viscosity of binders modified with moringa and sunflower oils underscores the potential of these modifiers to facilitate increased recycling rates, as indicated by the Binder Mix Design Chart in SUPERPAVE, where the allowable percentage of RAP in asphalt mixtures is inversely proportional to the PG value of the asphalt binder.

As the Performance Grades (PGs) of the modified binders and the reclaimed asphalt binder (RA) are known, the Binder Mix Design Chart B in the SUPERPAVE method was employed to simulate the RAP content to be added in the recycled asphalt mix design process. The target for all mixtures was to achieve a maximum critical PG temperature of 70°C, a value commonly obtained for asphalt binders modified with elastomers such as the PMA 55/75 modified with Styrene-Butadiene-Styrene (SBS) polymer, widely used in Brazil. The resulting charts from this evaluation are presented in Figure 2.

Based on the information presented in Figure 2, it substantiates the potential of vegetable oils as recycling agents for recycled asphalt mixtures. The incorporation of up to 2% sunflower oil and up to 1.5% moringa oil did not result in any significant alteration in the RAP content (29.41%) to be included in the recycled mixture, when compared to the neat asphalt binder (AB). This outcome was expected, as samples containing 1%, 1.5%, and 2% showed equivalent PG values. However, the introduction of 2.5% of both oils into the asphalt binder led to an increase in the RAP content to be added to the recycled asphalt mixture, raising it from 29.41% to 42.86% RAP. In essence, the reduction of 4 PG degrees for samples with 2.5% of oils considerably raised the RAP content to be added. The sample incorporating 2% moringa oil allowed for an intermediate recycling rate compared to the other oil contents tested.

This particular sample enabled the addition of 40% RAP, indicating a 36% increment in RAP content in comparison to the reference sample (AB).

Table 7. Characterization of physical and rheological properties of RA binder

Softening Point Variation (ºC)		68.00
Penetration 0.1 mm (100g. 5s to 25ºC)		29.00
Rotational Viscosity (cP)	135 °C	16476.00
	142 °C	9520.00
	150 °C	6676.00
	165 °C	2483.00
	177 °C	1215.00
PG (°C)		82.00
MSCR	Jnr at 0.1 kPa	1.82
	Jnr at 3.2 kPa	2.30
	Jnr_{diff}	26.57

Jnr = Non-recoverable compliance, PG = Performance Grade, MSCR = Multiple Stress Creep Recover.

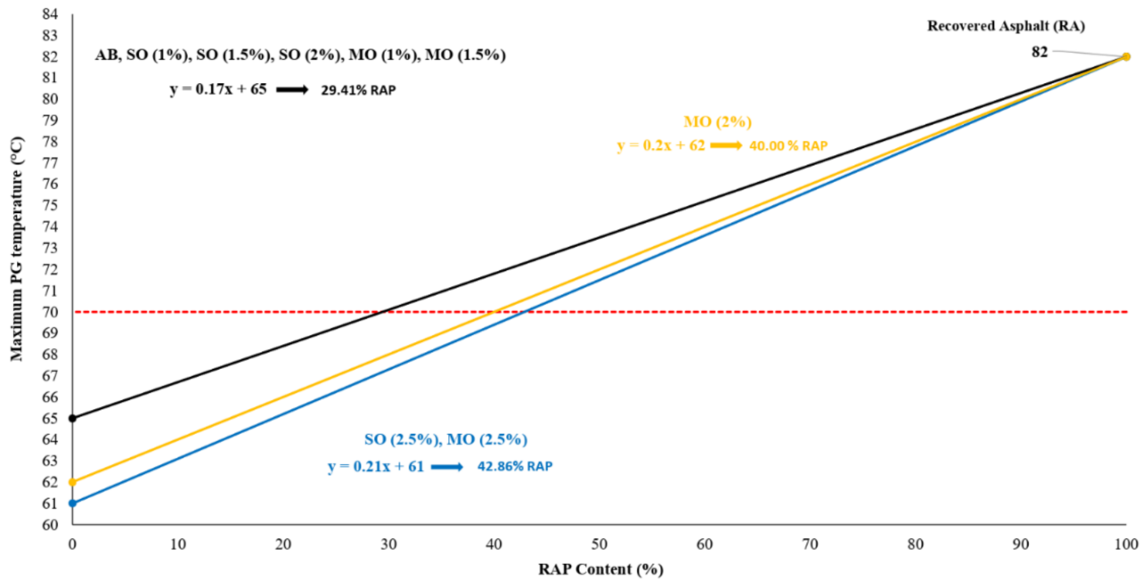


Figure 2. Blending chart for binder samples modified with moringa and sunflower oil.

It's important to note that the data used for chart construction was based on the continuous PG temperature to provide a more accurate representation of the binder's maximum performance. Melo Neto et al. [11, 19] relied on PG data rather than continuous PG, a choice that may overestimate the binder, as in some cases, the failure temperature, for instance, was 57°C while the binder aligned with a PG of 52°C. For this reason, continuous PG values were preferred in this study. These findings underscore the necessity for binders with low viscosity and PG to deliver recycled mixtures with a high RAP content (>30%).

4. Conclusions

Here are the conclusions drawn from the experimental analysis of the current study:

(1) Both moringa oil and sunflower oil were effective in reducing the viscosity of the asphalt binder, resulting in lower compaction and mixing temperatures. Moringa oil exhibited the highest reductions, with a decrease of 18.3°C in compaction temperature and 13.8°C in mixing temperature at a 2.5% concentration.

(2) The incorporation of moringa and sunflower oils did not have a significant impact on the continuous Performance Grade (PG) of the binder, which remained at 61°C after the Rolling Thin Film Oven Test (RTFOT).

(3) The asphalt binders containing vegetable oils met the requirements for use in standard traffic (S), and the 1% concentration samples also fulfilled the criteria for heavy traffic (H).

(4) The master curves generated for the modified binders indicated a decrease in binder rigidity due to the reduction in viscosity caused by the addition of oils.

(5) Based on the results, it is feasible to incorporate 2% additives into the binder as it did not compromise its rheological performance while leading to significant reductions in working temperatures.

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(6) The utilization of vegetable oil contents below 2% had no impact on the quantity of RAP to be incorporated into the recycled mixture.

(7) Incorporating 2.5% moringa and sunflower oil into the virgin asphalt binder enables the utilization of approximately 43% RAP for a PG working temperature of 70°C.

Additional research is advised to assess the mechanical performance of warm and recycled asphalt mixtures utilizing binders enhanced with moringa and sunflower oil. Furthermore, it is crucial to evaluate the production process's costs and environmental implications comprehensively, considering technical, environmental, and economic aspects.

Autor Contributions

G. G. da Silva: Conceptualization, Methodology, Investigation, Data Curation, Writing - Original Draft, Writing - Review & Editing. O. de M. Melo Neto: Methodology, Investigation, Data Curation, Writing - Original Draft. A. E. de F. L. Lucena: Conceptualization, Supervision, Project administration. T. M. R. P. de Moraes: Data Curation, Writing - Original Draft. D. B. Costa: Data Curation, Writing - Original Draft. T. M. de Sousa: Writing - Original Draft. B. B. de Souza: Writing - Original Draft. F. do S. de S. Carvalho: Data Curation, Writing - Original Draft. M. Y. Abdulwahid: Data Curation, Writing - Original Draft.

Conflicts of Interest

The authors have no relevant financial or non-financial interests to disclose.

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