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TECHNICAL PAPER

Analysis of the chemical, physical, and rheological properties of asphalt binder modified with pequi oil for potential application in asphalt mixtures

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Abstract

The increasing demand for sustainable paving technologies has driven research into bio-based additives capable of reducing energy consumption and improving asphalt mixture performance. This study evaluated the chemical, empirical physical, and rheological behavior of asphalt binders modified with pequi oil (Caryocar brasiliense), a native Brazilian bio-oil, aiming to assess its potential as an additive for asphalt mixtures. Petroleum asphalt cement with a 50/70 penetration grade (PAC 50/70) was modified with 1, 2, and 3% pequi oil by weight. The characterization included chemical analysis, penetration, softening point, rotational viscosity, and advanced rheological tests before and after short-term aging. The chemical analysis revealed the presence of ester and carbonyl functional groups, indicating good compatibility and absence of significant oxidation after aging. Increasing pequi oil content led to a progressive decrease in viscosity and softening point, confirming its plasticizing effect. Rheological tests showed that each 1% increment of oil reduced the Performance Grade (PG) by approximately 2 °C. The modified binders exhibited reduced stiffness (G*) and higher phase angles (δ), reflecting enhanced flexibility but reduced elastic recovery. Rheological tests showed Jnr > 4.0 and negligible recovery, classifying the binders as unsuitable for heavy traffic but effective for moderate conditions. LAS results indicated that fatigue life decreased with higher oil contents, with BFF (Binder Fatigue Factor) values transitioning from "intermediate" to "inferior" at 3% addition. The blending analysis predicted that binders with 1–3% pequi oil can accommodate 35–52% RAP for a target PG of 70 °C. Overall, pequi oil demonstrated technical potential as a sustainable asphalt modifier, reducing production temperature and energy demand while enhancing pavement circularity, both through material circularity via the incorporation of RAP (Reclaimed Asphalt Pavement) and systemic circularity through the integration of bio-based supply chains.

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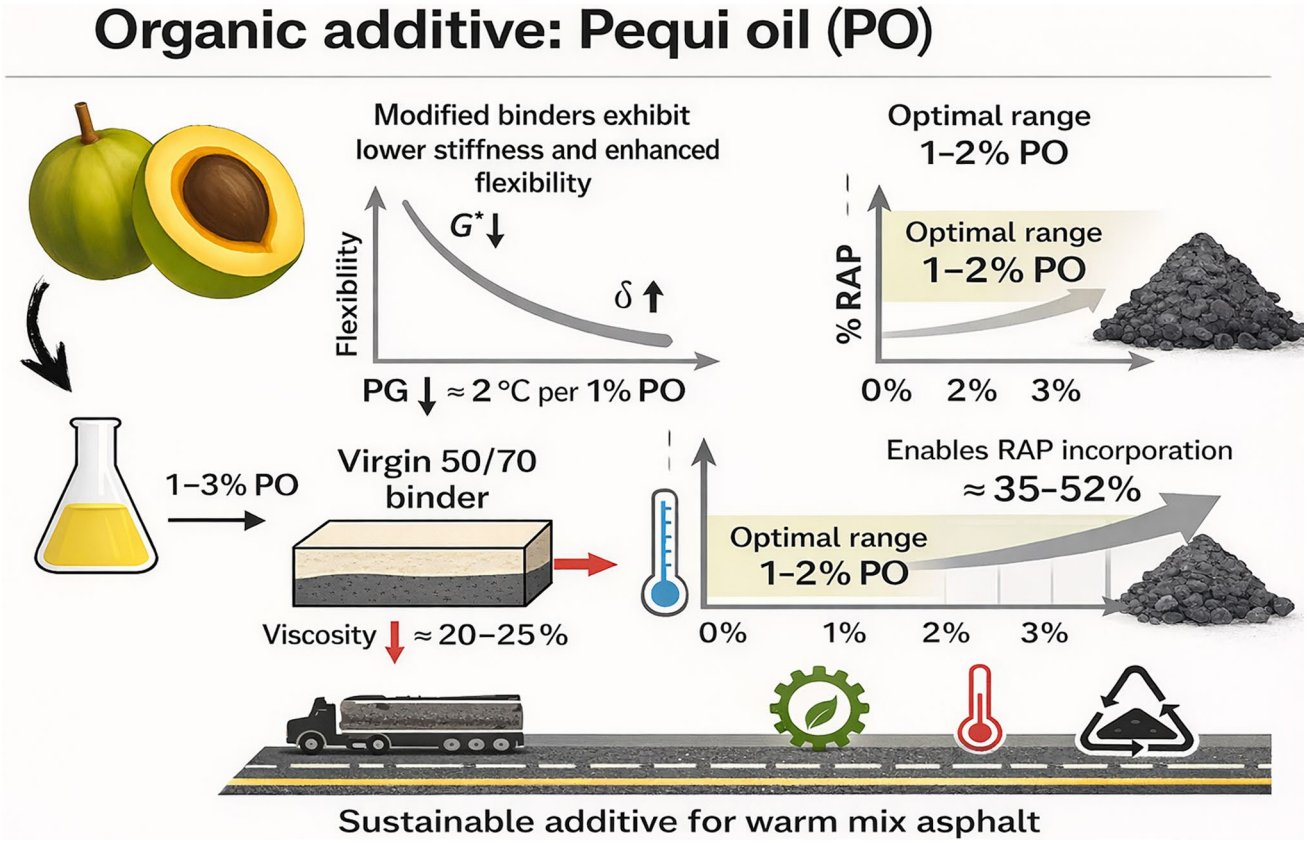
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Graphical abstract



Keywords Warm asphalt mix · Vegetable oil · Bioeconomy · Sustainability · Circular economy · Rejuvenation

Abbreviations

AASHTO	American association of state highway and transportation officials
ASTM	American society for testing and materials
BFF	Binder fatigue factor
CBUQ	Hot-mix asphalt concrete
CMA	Cold-mix asphalt
CNT	National confederation of transport
DNIT	National department of transport infrastructure
DSR	Dynamic shear rheometer
FFL	Fatigue factor of the binder
FTIR	Fourier transform infrared spectroscopy
HMA	Hot mix asphalt
Jnr	Non-recoverable creep compliance
LAS	Linear amplitude sweep
LEP	Pavement engineering laboratory
PG	Performance grade
PO	Pequi oil
RAP	Reclaimed asphalt pavement
RTFO	Rolling thin film oven
TSI	Thermal susceptibility index
WMA	Warm mix asphalt

List of symbols

R	Multiple stress creep and recovery
PAC	Petroleum asphalt cement
ρ	Density (g/cm^3)
η	Dynamic viscosity (cP)
T	Temperature (°C)
G*	Complex shear modulus (kPa)
δ	Phase angle (°)
Jnr	Non-recoverable creep compliance (kPa^{-1})
Jnr diff	Percent variation of Jnr between applied stresses (%)
$R_{0.1}$	Elastic recovery at 0.1 kPa stress (%)
Nf	Fatigue life (number of cycles to failure)
C	Integrity parameter of the binder during fatigue damage evolution
PG_{max}	Maximum performance grade temperature (°C)
PG_{min}	Minimum performance grade temperature (°C)
t	Mixing time (min)

rpm	Rotational speed (revolutions per minute)
TSI	Thermal susceptibility index (dimensionless)
σ	Stress (kPa or MPa)
ε	Strain (%)
f	Frequency (Hz)
ΔT	Temperature difference (°C)

Introduction

The Brazilian highway network, the fourth largest in the world, extends over 1.7 million kilometers, of which 65.8 thousand were paved in 2022 [1]. Asphalt pavements, which predominate on paved roads, are viscoelastic composites sensitive to temperature and loading, whose properties deteriorate due to the oxidative aging of the asphalt binder [2].

There are two main processes for producing asphalt mixtures: cold mix asphalt (CMA) and hot mix asphalt (HMA). CMAs, which have lower cost and are applicable at lower temperatures, are typically used in low-traffic pavements. HMAs, such as the conventional hot-mix asphalt concrete (CBUQ), exhibit superior mechanical performance and are recommended for high-traffic roads [3–5].

The production of hot mix asphalts (HMAs) requires high energy consumption and results in greater emission of pollutants, in addition to accelerating the oxidative aging of the binder due to the elevated temperatures during mixing and compaction. Warm mix asphalts (WMAs) have emerged as a promising alternative to mitigate these effects, allowing a temperature reduction of 20–30 °C during production and application, thereby improving energy efficiency and reducing emissions [6–9].

Studies indicate that the use of WMAs can reduce CO_2 emissions by 17 to 37% and energy consumption by significant proportions when compared to conventional mixtures [10, 11]. In addition to environmental benefits, these mixtures enable construction under more favorable thermal conditions and shorten the time required for traffic release, representing both technical and operational advantages.

In Brazil, the use of organic additives for the production of warm mix asphalts (WMAs) has been extensively studied. Various vegetable oils, such as soybean, corn, moringa, sunflower, babassu, coconut, and linseed oils, have been employed to reduce viscosity and application temperature, in addition to acting as natural antioxidants [12–16].

Beyond the technical benefits, the adoption of bio-based modifiers represents a strategic shift toward systemic sustainability in infrastructure governance. Such innovations are essential for global energy and environmental transitions, promoting efficient resource use and social well-being [17, 18]. Within the circular economy framework, the use of regional bioresources, such as pequi oil, contributes to decarbonization pathways by reducing the carbon footprint of pavement construction and fostering equitable rural development in the Brazilian Cerrado region.

Furthermore, the valorization of local non-edible oils aligns with the fair allocation of resources and strengthens regional supply chains, addressing challenges in infrastructure resource governance [19]. This approach not only enhances pavement circularity through improved compatibility with recycled materials but also situates laboratory-scale material modifications within broader macro-level sustainability transitions [17]. However, scaling up the use of these additives requires overcoming governance challenges, including land-use competition and the establishment of certification frameworks to ensure resilient and sustainable supply chains.

According to the Brazilian Institute of Geography and Statistics [20], national pequi production reached R$ 65.75 million in 2023, representing an increase compared to previous years. Minas Gerais is the main producing state, accounting for 51.8% of the total volume, followed by Tocantins (39.7%), with Ceará also standing out in the Northeast region. The Brazilian Agricultural Research Corporation [21] reports that the oil extraction yield from the fruit pulp ranges from 30 to 40%, and can reach up to 75% with modern cold-pressing techniques. The oil is rich in unsaturated fatty acids, carotenoids, vitamin A, and phenolic compounds, which provide relevant antioxidant properties for industrial applications [22, 23].

In this context, this study investigated the effects of adding pequi oil on the physical, chemical, and rheological properties of asphalt binder, evaluating its potential as a warm-mix and antioxidant additive for asphalt mixtures. Thus, this research aims to contribute to the development of sustainable paving technologies by incorporating a renewable and regionally available product, with the objective of reducing the environmental impact of road infrastructure.

Materials and methods

This section describes the materials and procedures employed during the experimental phase of the research, following recognized technical standards, including those established by the American Society for Testing and Materials (ASTM) and the American Association of State Highway and Transportation Officials (AASHTO). Figure 1 presents the flowchart of the activities carried out in this study.

Materials

The asphalt binder used in this study was a petroleum asphalt cement with a penetration grade of 50/70 (PAC 50/70),

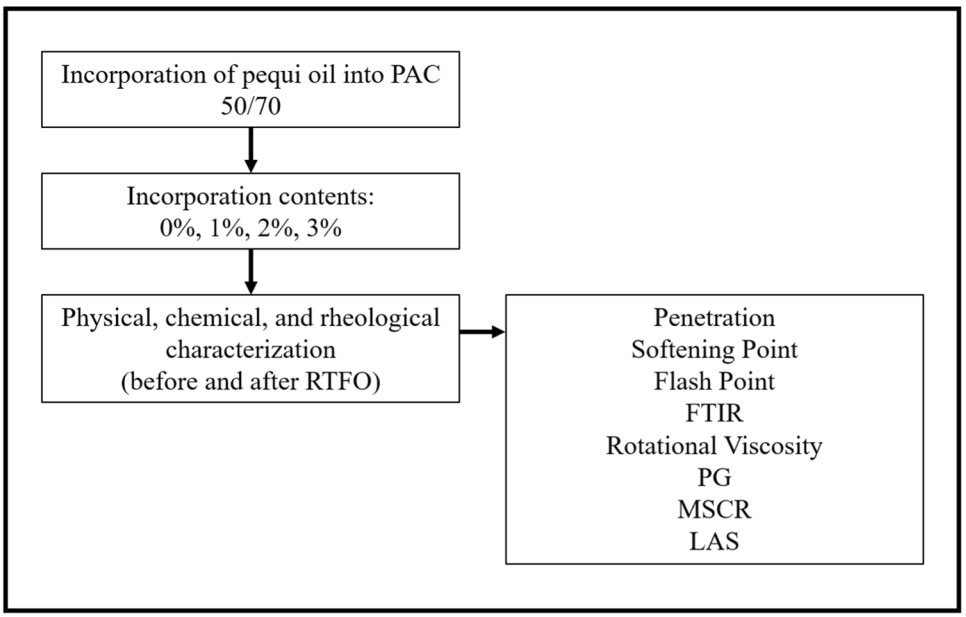


Fig. 1 Flowchart of the activities carried out in the study

Table 1 Summary of the physicochemical properties and fatty acid profile of pequi oil

Property	Value	References
Acidity index (mg KOH/g)	3.86	Colares et al. [24]
Iodine index (g I_2/100 g)	70 to 80	Torres Silva et al. [25]
Saponification index (mg KOH/g)	0.1 to 0.3	Colares et al. [24]
Fatty acid profile (%)		
Oleic acid (C18:1)	51 to 55	Miranda-Vilela et al. [26]; Roll et al. [27]
Palmitic acid (C16:0)	35 to 46	Miranda-Vilela et al. [26]; Roll et al. [27]
Others (Linoleic, Stearic)	<10	Miranda-Vilela et al. [26]; Roll et al. [27]

supplied by Construtora Rocha Cavalcante Ltda., headquartered in Campina Grande, Paraíba, Brazil. This binder is classified according to the Brazilian regulatory framework established by ANP Resolution No. 19/2005, which defines the technical specifications for petroleum asphalt cements in the national territory. The selection of this binder is justified by its widespread use in paving works throughout the Northeastern region of Brazil.

The pequi oil used in this study was a commercial virgin oil (Aroma D'Minas, Brazil), obtained by cold pressing to preserve its chemical integrity and natural antioxidant properties, with pulp extraction yields ranging from 30 to 40%. To ensure experimental consistency, a single batch of oil was used for all samples. Its use was based on the practicality of acquisition, the satisfactory productivity achieved through extraction methods, and its availability in the national market. In addition, it is naturally rich in bioactive compounds, notably oleic and palmitic fatty acids, as well as vitamins A and E, present in the forms of carotenoids and tocopherols, respectively. Table 1 describes the physicochemical properties and the fatty acid profile of pequi oil.

The pequi oil exhibited a density of 0.887 g/cm3 and viscosities of 62.5, 27.5, and 22.5 cP at temperatures of 25, 70, and 140 °C, respectively. The volatile content obtained was 0.27%. It was observed that the density of pequi oil is comparable to that of other vegetable oils, such as soybean, corn, sunflower, cottonseed, olive, babassu, and canola oils; however, its viscosity is slightly higher [28, 29].

Methods

The incorporation of pequi oil into the asphalt binder was carried out according to the procedures described by Neto et al. [30] and Melo Neto et al. [14]. Initially, the virgin binder was heated to 135 °C using an integrated digital controller to avoid localized overheating or premature oxidative aging. Subsequently, pequi oil was added at concentrations of 1%, 2%, and 3%, separately, under agitation at 410 revolutions per minute (rpm) for 20 min. The addition was carried out slowly and fractionally during the first 5 min. After mixing, the samples were manually stirred to ensure the removal of any trapped air bubbles (degassing) and were

then immediately sealed in hermetic containers to minimize oxygen exposure prior to rheological testing. Homogeneity was verified through consistent triplicate results in the penetration and softening point tests, ensuring the reliability of the modification protocol. The selected contents (1% to 3%) were based on previous studies using other vegetable oils, such as soybean, sunflower, babassu, and moringa oils, in which these concentration ranges proved effective in reducing viscosity and providing antioxidant properties without compromising the stability and cohesion of the binder [13, 15, 31]. This range is widely recognized in the literature as representing a balance between technical performance and practical feasibility in warm-mix asphalt production. After the mixing and resting process, complete homogenization of the modified binder samples was observed, indicating good compatibility between the asphalt binder and the pequi oil. This behavior is consistent with previous studies employing other vegetable oils as modifiers [29, 32].

After the preparation of the modified asphalt binders, they were subjected to a series of chemical, empirical, and rheological characterization tests, performed in duplicate to ensure the reproducibility and reliability of the results. The duplicate measurements presented minimal variability between replicates, remaining within the expected precision range of the respective test standards.

The chemical characterization was carried out through Fourier Transform Infrared Spectroscopy (FTIR), using a Bruker Vertex 70 spectrometer equipped with an Attenuated Total Reflectance (ATR) probe, operating in the range of 4000 to 500 cm^{-1}. This technique enabled the identification of changes in chemical composition and the evaluation of the oxidation degree of the binders after the addition of pequi oil and the aging processes.

The empirical characterization included penetration (ASTM D5M [33]) and softening point (ASTM D36M [34]) tests. The penetration test was conducted according to ASTM D5M [33], applying a total load of 100 g for five seconds at a controlled temperature of 25 °C. Three determinations were performed at different points on each sample, maintaining a minimum spacing of 10 mm between penetrations. The softening point test followed ASTM D36M [34], with heating at a rate of (5 ± 0.5) °C/min, determining the temperature corresponding to the fall of the metal sphere onto the lower plate. Both tests were conducted in duplicate, and repetitions were carried out whenever the difference between determinations exceeded 1 °C.

The rheological characterization was performed using a Dynamic Shear Rheometer (DSR, model DHR-1, TA Instruments) at the Pavement Engineering Laboratory of the Federal University of Campina Grande. The tests included:

(i) Rotational viscosity (ASTM D4402 [35]), measured between 135 and 177 °C under rotational speeds of 20, 50, and 100 rpm;
(ii) Performance Grade (PG) determination, according to ASTM D6373 [36], using controlled strain amplitudes of 12% for unaged binders and 10% for short-term aged samples, with temperature increments of 6 °C;
(iii) Multiple Stress Creep and Recovery (MSCR) test, following AASHTO M332 [37], performed at 64 °C under stress levels of 0.1 kPa and 3.2 kPa; and.
(iv) Linear Amplitude Sweep (LAS) test, according to AASHTO T391 [38], applied to short-term aged samples at 19 °C under a constant frequency of 10 Hz and strain amplitudes ranging from 0.1% to 30%. Master curves were obtained between 5 and 65 °C for frequencies ranging from 0.01 to 25 Hz.

Finally, the binders were subjected to short-term aging using the Rolling Thin Film Oven (RTFO) method, according to ASTM D2872 [39]. This procedure consists of continuously rotating 35 g of binder in glass bottles at 163 °C for 85 min under a constant air flow. The RTFO test allowed the evaluation of thermal stability, mass loss, and changes in the physical and rheological properties of the binders, simulating the effects of mixing and field application.

Results and discussions

This section presents and discusses the chemical, physical, and rheological results obtained in the experimental phase for both the unmodified and the pequi-oil-modified asphalt binders, before and after short-term aging simulation using the RTFO procedure.

Chemical analysis

Figure 2a shows the FTIR test results for the unmodified binder and the binders containing 1, 2, and 3% pequi oil, both before and after the RTFO procedure. Figure 2b presents a magnified view of the spectral region near the peak at 1740 cm^{-1}.

The obtained spectra reveal important information regarding the composition of the neat and pequi-oil-modified binders. For the spectra of the binders (before and after RTFO), a similar profile was observed among the samples, indicating that the addition of pequi oil did not cause significant structural changes in the asphalt matrix. No peaks with sufficient intensity were detected to indicate a high degree of oxidation resulting from the thermal treatment, particularly in the regions between 1760 and 1700 cm^{-1},

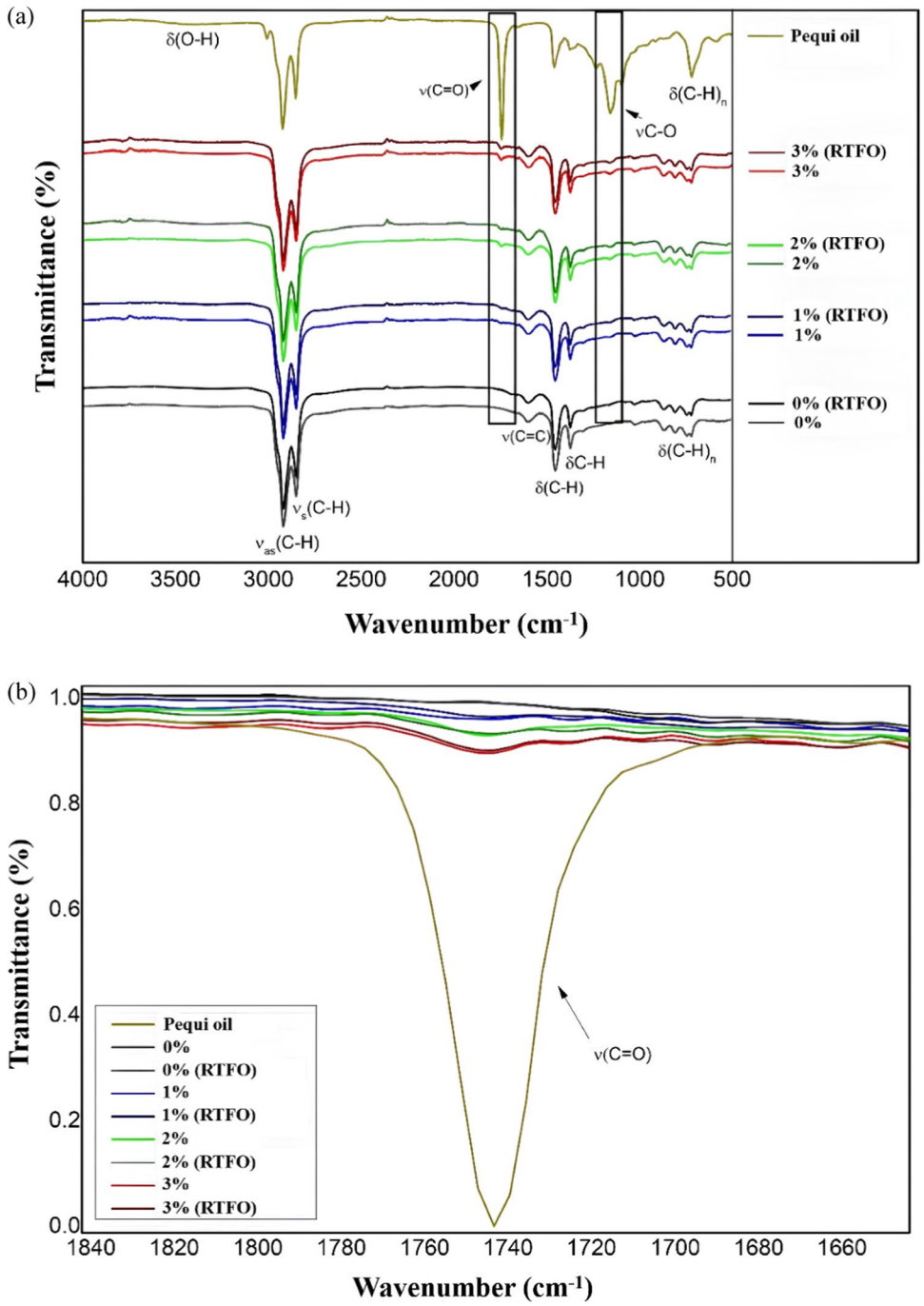


Fig. 2 **a** FTIR spectra of asphalt binders modified with pequi oil; **b** magnified spectral region highlighting the ester carbonyl band (~1740 cm^{-1}) associated with triglycerides in pequi oil and its progressive contribution to the modified binders

corresponding to carbonyl (C=O) stretching, or between 1250 and 1050 cm^{-1}, related to C–O stretching. Likewise, no signals indicative of oxygenated functional groups were identified within the 3100–3600 cm^{-1} range, associated with hydroxyl (O–H) vibrations, suggesting low susceptibility to thermal oxidation after aging.

Regarding the binder characterization, the main signals indicative of its composition were observed in regions typical of hydrocarbon compounds, with prominent high-intensity peaks between 3000 and 2800 cm^{-1} corresponding to the stretching (axial deformation) of saturated aliphatic groups. In this range, peaks were identified at 2951 cm^{-1} and 2867 cm^{-1}, related to the asymmetric and symmetric stretching of methyl groups (–CH_3), and at 2919 cm^{-1} and 2850 cm^{-1}, associated with the respective stretching of methylene groups (–CH_2–), in addition to a peak at 2890 cm^{-1} attributed to methine groups (–CH–). Lower-intensity peaks between 2000 and 1700 cm^{-1} indicate harmonic bands of substituted aromatic compounds, as well as small amounts of carbonyl (C=O) groups. The presence of

peaks around 1600 cm^{-1} and 1475–1455 cm^{-1} confirms the existence of unsaturations (C=C) and angular deformations of methylene groups, respectively, consistent with the axial deformations previously observed. The peak at 1375 cm^{-1}, corresponding to the angular deformation of methyl groups ($–CH_3$), and the one around 720 cm^{-1}, associated with rocking vibrations of long aliphatic chains, confirm the organic nature of the binder, composed of saturated, unsaturated, and aromatic chains.

The FTIR spectrum of pequi oil exhibited peaks similar to those of the asphalt binder concerning carbon–hydrogen bonds, as these species are common in organic structures. Peaks were identified between 2950 and 2870 cm^{-1}, associated with methyl and methylene groups (asymmetric and symmetric stretching), and at 1460 and 1375 cm^{-1}, corresponding to the angular deformations of the same groups. The peak at 1740 cm^{-1}, characteristic of carbonyl (C=O) stretching, together with the peak at 1162 cm^{-1} (C–O stretching), indicates the presence of ester groups typical of triglycerides and fatty acids. The slight band between 3100 and 3600 cm^{-1} suggests a low content of free fatty acids, consistent with natural vegetable oils. Finally, the peak at 719 cm^{-1} confirms the presence of long aliphatic chains in the oil molecules. In Fig. 2 (b), the slight increase in the intensity of the peak at 1740 cm^{-1} is consistent with the progressive incorporation of pequi oil into the asphalt binder, indicating effective modifier integration. After RTFO aging, only minor variations in band intensities were observed, suggesting that the oil remained stable and present in similar proportions. The progressive increase of the band near 1740 cm^{-1} corresponds to the carbonyl stretching vibration of ester groups present in the triglyceride structure of pequi oil. Although the increase in peak intensity is relatively subtle in the plotted spectra, this behavior is expected because the oil contents used in the modification (1–3%) represent a small fraction of the total binder mass. Consequently, the FTIR response reflects the superposition of the asphalt matrix spectrum with the ester carbonyl contribution from the bio-oil rather than the formation of new chemical bonds. This spectral behavior indicates physical incorporation of the modifier into the maltene phase of the binder rather than chemical reaction with the asphalt components.

The obtained results are consistent with previous studies that employed other vegetable oils as asphalt binder modifiers. Investigations using babassu oil [15, 29] identified similar bands at 1743 and 1164 cm^{-1}, associated with the presence of esters, and reported comparable spectral behavior, in which increasing the oil content intensified the carbonyl bands without substantially altering the hydrocarbon structure of the asphalt binder. Similarly, studies with soybean and corn oils [40–42] revealed strong C=O (~1740 cm^{-1}) and C–H (~2920 and 2850 cm^{-1}) bands, responsible for reducing viscosity and improving the binder's workability, characteristics also observed in the present study.

The absence of a significant increase in the intensity of the bands associated with S=O (1030–1100 cm^{-1}) and O–H (3100–3600 cm^{-1}) after thermal aging indicates that pequi oil helped limit binder oxidation, possibly due to its composition rich in unsaturated fatty acids, carotenoids, and tocopherols—compounds known for their antioxidant properties. This behavior is similar to that reported for fatty acid residue from soybean oil [14], which exhibited lower formation of oxygenated compounds and a pronounced antioxidant effect. Therefore, pequi oil shows potential to delay the oxidative aging of asphalt binders, contributing to greater durability and thermal stability.

Overall, the incorporation of pequi oil resulted in a spectroscopic behavior consistent with that observed in binders modified with other vegetable oils, confirming the presence of ester groups and the preservation of the binder's chemical structure. The stability of band intensities after aging and the absence of significant oxidation signs reinforce the efficiency and chemical compatibility of pequi oil as an organic additive, further highlighting its potential contribution to the development of warm mix asphalt technologies.

Empirical physical characterization

Table 2 presents a summary of the physical test results obtained in this study, before and after the RTFO procedure, for the different incorporated pequi oil contents.

The incorporation of pequi oil into the asphalt binder produced clear effects on the material's consistency and thermal behavior, in accordance with what has been reported in the literature for other vegetable oils used as modifiers or rejuvenators of binders. The penetration results obtained before aging (RTFO) show a progressive increase with the addition of oil, rising from 51 dmm in the base binder (0%) to 57 dmm (1%), 62 dmm (2%), and 68 dmm (3%). This increase in penetration indicates a reduction in stiffness and

Table 2 Summary of the physical characterization results of the modified binder

Results		0% Pequi	1% Pequi	2% Pequi	3% Pequi
Penetration	before RTFO (0.1 mm)	51.00	57.00	62.00	68.00
	after RTFO (0.1 mm)	37.80	41.20	49.60	50.20
	Retained (%)	0.74	0.72	0.81	0.73
Softening point	before RTFO (0.1 mm)	46.00	45.00	43.00	41.00
	after RTFO (0.1 mm)	50.00	49.00	47.00	45.00
Thermal susceptibility index (TSI)		− 2.18	− 2.24	− 2.67	− 3.08

a plasticizing effect of pequi oil, meaning greater molecular mobility and lower binder consistency. A similar behavior has been observed for other vegetable oils, such as babassu, sunflower, moringa, soybean, and corn, where higher oil content systematically results in softer binders with greater penetration, confirming the typical softening effect of fatty-based modifiers.

After the RTFO procedure, all binders exhibited reduced penetration values, as expected due to thermal hardening and the loss of light fractions during accelerated aging. However, even after RTFO, the binders modified with pequi oil maintained higher penetration values than the aged base binder: while the binder without oil showed 37.8 dmm, the samples containing 2% and 3% pequi retained 49.6 and 50.2 dmm, respectively. This finding is relevant because it indicates that, although thermal aging induces hardening, pequi oil mitigates the loss of workability. A similar aging-mitigation effect has been reported for babassu oil and for fatty acid residues derived from soybean oil sludge, both of which also reduced post-aging stiffness and were interpreted as rejuvenating agents capable of restoring part of the flexibility of oxidized binders.

The retained penetration (ratio between penetration after RTFO and before RTFO) remained equal to or greater than 0.72 for all pequi oil contents and reached 0.81 at the 2% level. These values correspond to penetration retention rates of 72% or higher, exceeding the minimum threshold of 55% established by ANP [43] as the reference for short-term aged binders. They are also comparable to the high retained penetration values reported for binders modified with corn and moringa oils, which likewise exhibited lower sensitivity to short-term oxidative hardening. The 2% pequi oil content stood out for presenting the highest retained penetration among the formulations evaluated, suggesting that this specific dosage promoted the best preservation of consistency after accelerated thermal aging. This result indicates that 2% may represent an optimal balance point between initial flexibilization and resistance to the loss of flexibility during thermal oxidation.

The softening point results obtained before RTFO reinforce the plasticizing effect of pequi oil. A continuous reduction in softening point was observed with increasing oil content, decreasing from 46 °C (neat binder) to 45 °C (1%), 43 °C (2%), and 41 °C (3%). This type of behavior has also been reported in the literature for binders modified with vegetable oils such as babassu, canola, sunflower, copaiba, linseed, moringa, soybean, and corn oils [12–15, 30, 40–42, 44], where the incorporation of fatty compounds reduces the binder's thermal stiffness and consequently lowers the temperature at which the material begins to flow. From a practical standpoint, this initial reduction in softening point is associated with a decrease in in-service viscosity and thus enables the production and application of asphalt mixtures at lower temperatures, a desirable characteristic in warm mix technologies.

After the RTFO procedure, a systematic increase in softening point was observed for all samples, reaching 50 °C in the binder without oil and values of 49 °C (1%), 47 °C (2%), and 45 °C (3%) for the binders modified with pequi oil. This behavior, an increase in softening point after aging, is typical of oxidative hardening and is widely reported in the literature for both neat and vegetable oil–modified binders. Nevertheless, even after accelerated aging, the binders containing pequi oil remained less stiff at high temperatures than the base binder without oil, as they exhibited post-RTFO softening points lower than that of the reference binder. This result aligns with findings for binders modified with babassu and other unsaturated oils (such as sunflower and moringa), which also retained lower relative stiffness after aging, indicating a potential to mitigate oxidative hardening [13, 15].

The combined interpretation of penetration and softening point allows the discussion of thermal susceptibility. The Thermal Susceptibility Index (TSI), as determined by Eq. (1), became progressively more negative with increasing pequi oil content, decreasing from −2.18 in the base binder to −2.24 (1%), −2.67 (2%), and −3.08 (3%). A more negative TSI indicates greater sensitivity to temperature variations: the binder becomes more flexible at low temperatures, potentially reducing the risk of thermal cracking, but also more prone to permanent deformation at high temperatures (e.g., rutting). The same trade-off has been reported for mixtures modified with higher contents of babassu oil, in which increased flexibility was accompanied by reduced resistance to plastic flow under elevated service temperatures, especially above 60–70 °C and under heavy traffic. Similar results were also observed for sunflower and moringa oils when used at concentrations above 2%, where the mixture exhibited improved workability and compactability but required attention regarding permanent deformation under hot climates and heavy traffic conditions.

$$TSI = \frac{(500)\,(\log PEN) + (20)\,(T°C)\,1951}{120\,(50)\,(logPEN) + (T°C)} \quad (1)$$

where: PEN = penetration at 25 °C, 100 g, and 5 s; (T°C) = softening point.

In the context of pequi oil, the results suggest an intermediate and technically attractive behavior: at 1% and 2% contents, binder flexibilization is observed, with a moderate reduction in softening point and high retained penetration after RTFO, without an extreme decrease in softening point that would immediately compromise stability at high temperatures. At the 3% content, on the other hand, the

binder becomes softer (68 dmm before RTFO and 50.2 dmm after RTFO) and exhibits the lowest softening point among the evaluated formulations (41 °C before and 45 °C after RTFO), indicating greater susceptibility to permanent deformation when exposed to elevated service temperatures or continuous heavy traffic.

This same pattern, satisfactory performance at lower dosages and the need for caution at higher ones, has been reported for soybean/corn and babassu oils [15, 40–42], which transition from beneficial additives (improving workability and reducing mixing temperature) to potentially critical softening agents when used at higher concentrations.

In summary, the results indicate that pequi oil acts as an organic modifier with both plasticizing and rejuvenating characteristics, promoting increased penetration, a reduction in softening point, and greater consistency retention after accelerated thermal aging. The observed behavior aligns with that reported for other vegetable oils used in asphalt binder modification, confirming that pequi oil can reduce the initial stiffness of the binder and mitigate the hardening induced by RTFO. From an application standpoint, dosages up to 2% appear to provide a favorable balance between flexibility, workability, and thermal resistance, whereas higher dosages (3%) may require additional measures to control permanent deformation in service, such as combining with elastomeric modifiers or limiting its use to pavements subjected to lower traffic demand and/or milder climates.

Rheological assessment

Table 3 presents a summary of the rheological results obtained in this study, before and after the RTFO procedure, for the different pequi oil dosages incorporated. The results of rotational viscosity corroborate the Thermal Susceptibility Index (TSI) values, showing that the binder becomes progressively more fluid as the oil content increases. The reduction in viscosity is more pronounced at 135 °C, which is beneficial in terms of workability, pumping, and asphalt mixture application. This behavior is consistent with that observed in studies involving vegetable oils such as soybean, corn, moringa, sunflower, and babassu [13, 15, 40–42], in which viscosity decreased by approximately 20–30% for dosages up to 3%, allowing mixture production at temperatures lower than those typically required for conventional hot mix asphalt (WMA). A similar viscosity-reducing effect was also reported for binders modified with beeswax [45], particularly above its melting point (~60 °C), where the wax behaves as a lubricant, reducing shear resistance and improving flowability. However, unlike vegetable oils, wax also increases the softening point and the thermal stability of the binder in service, indicating that pequi oil behaves more similarly to liquid bio-oils than to wax-based modifiers in its mode of action.

The mixing and compaction temperatures calculated for each pequi oil content indicate that higher dosages allow lower energy consumption during plant production, as mixing and compaction can occur at temperatures up to 5 °C lower than those required for the pure binder. A similar

Table 3 Rheological characterization results of the modified binder

Results			0% Pequi	1% Pequi	2% Pequi	3% Pequi
Rotational viscosity	Before RTFO	135 °C	402	378	329	313
		142 °C	286	270	238	228
		150 °C	204	190	172	162
		165 °C	114	107	98	92
		177 °C	75	71	66	63
	After RTFO	135 °C	564	511	453	446
		142 °C	393	359	321	313
		150 °C	272	249	224	218
		165 °C	147	136	123	120
		177 °C	95	89	81	79
Working temperatures		Mixing Temperature (°C)	155	156	152	150
		Compaction Temperature (°C))	142	144	139	137
Performance grade (PG)		Before RTFO (°C)	64	64	58	58
		After RTFO (°C)	64	58	58	58
True grade		Before RTFO (°C)	66.7	64.9	62.4	60.9
		After RTFO (°C)	64.9	63.1	61.5	59.3
Non-recoverable creep compliance (Jnr)	Temperature of 64 °C	100 Pa	3.88	5.09	6.12	7.44
		3200 Pa	4.08	5.13	6.52	8.29
Average recovery (%)		100 Pa	0.81	0.00	0.39	2.32
		3200 Pa	0.39	0.21	0.00	0.00
Jnrdiff (%)			5.06	0.75	6.54	11.41

trend was observed for binders modified with soybean, corn, moringa, and sunflower oils [13, 40, 42], whose mixing and compaction temperatures were reduced by up to 8–10 °C without compromising workability. In the case of babassu oil, even greater reductions were reported, allowing the binder to fit within the typical range of warm mix asphalt (< 150 °C). For beeswax, although a reduction in viscosity during mixing is also observed, its thermoplastic effect results in increased stiffness as the pavement cools, providing greater resistance to permanent deformation [45]. It should be noted, however, that the temperature reduction observed in the present study results solely from viscosity decrease, making it necessary to further evaluate the effect of lubricity to confirm the efficiency of pequi oil as a temperature-reducing agent in warm mix asphalts.

It was observed that the addition of 1% pequi oil does not alter the PG classification of the modified binder, while the contents of 2% and 3% result in a one-grade reduction in PG. This reduction is accompanied by a decrease of approximately 2 °C in the PG temperature for each 1% of oil added, a behavior consistent with that reported for binders modified with babassu, soybean, and corn oils [15, 40, 42], in which the incorporation of fatty compounds leads to a decrease in the continuous PG due to the reduction of the G*/sin δ parameter. Conversely, binders modified with beeswax tend to increase the PG by up to one grade because of the formation of a crystalline network that enhances resistance at high temperatures [45], showing that pequi oil follows the opposite trend, acting as a thermal softening agent. In practical terms, this reduction indicates that asphalt mixtures produced with pequi oil tend to exhibit lower stiffness and greater thermal susceptibility, a behavior that may be advantageous in colder regions but requires caution in pavements exposed to high service temperatures and heavy traffic loads.

The recovery percentage obtained from the MSCR test was practically null at all incorporation levels, indicating that the binders, even when modified with pequi oil, exhibit a predominantly viscous behavior typical of non-polymeric binders. The highest recovery value ($R_{0.1} \approx 2\%$) was observed for the 3% content, possibly due to a slight transient internal structuring. This result is similar to that reported for binders modified with copaiba, babassu, corn, and soybean oils [15, 30, 40, 42], which also showed low elastic recovery rates and increased viscous behavior with additive incorporation. In contrast, binders modified with beeswax presented recovery values above 15% at intermediate dosages, evidencing the enhanced elastic character imparted by wax crystallization upon cooling [45], a behavior opposite to that of liquid oils, including pequi oil. For all incorporation levels, Jnr values were higher than 4.0, classifying the binders as unsuitable for severe traffic according to AASHTO M332 [37]. This trend was also reported for binders containing high levels of babassu and other vegetable fatty oils [14, 15], confirming that increasing pequi oil content significantly reduces binder stiffness and its ability to resist permanent deformation at high service temperatures.

The Jnr diff parameter, which measures the percentage variation of Jnr with increasing applied stress, exhibited values consistent with the observed viscoelastic behavior. The 1% content showed the lowest Jnr diff (0.75%), suggesting a more linear and predictable response under different stress levels, whereas the 3% content presented values exceeding 10%, indicating high sensitivity to loading. Similar results were reported for binders containing high babassu oil contents and soybean fatty acid residue [14, 15], where increasing modifier dosage also led to greater load sensitivity and reduced stability under heavy traffic. In contrast, the addition of beeswax decreases Jnr diff and enhances elastic recovery, making the binder more stable under cyclic loads, a desirable yet distinct effect compared with that produced by liquid oils [45].

Figure 3a presents the Master Curves of the Dynamic Shear Modulus (G*) as a function of reduced frequency for the studied binders. It is observed that G* increases with reduced frequency, a typical behavior of viscoelastic materials, reflecting higher stiffness at low temperatures (high frequencies) and lower stiffness at high temperatures (low frequencies). The neat binder exhibited the highest G* values across almost the entire frequency range, whereas increasing the pequi oil content led to systematic reductions, particularly from 2% incorporation onward. This trend is analogous to that reported in studies with babassu, soybean, corn, and moringa oils [13, 15, 40–42], where the addition of unsaturated oils reduced the shear modulus and increased viscous behavior. Conversely, studies with beeswax have shown an increase in G* at elevated temperatures due to the formation of microcrystals that stiffen the binder [45], once again demonstrating that pequi oil behaves as a purely non-crystalline plasticizer. The reduction in G* implies greater flexibility and potential improvement in low-temperature performance but also decreased resistance to permanent deformation under service conditions.

The phase angle (δ), shown in Fig. 3b, represents the proportion between viscous (δ approaching 90°) and elastic (δ approaching 0°) behaviors. At low frequencies (high temperatures), the curves overlap, indicating a predominantly viscous behavior for all binders, with δ ranging between 85 and 90°. At high frequencies (low temperatures), the neat binder exhibited lower δ values, indicating a greater elastic component. As the pequi oil content increased, particularly from 2% onward, a systematic increase in δ was observed, reflecting a reduction in elasticity and predominance of viscous behavior. This trend contrasts with that reported

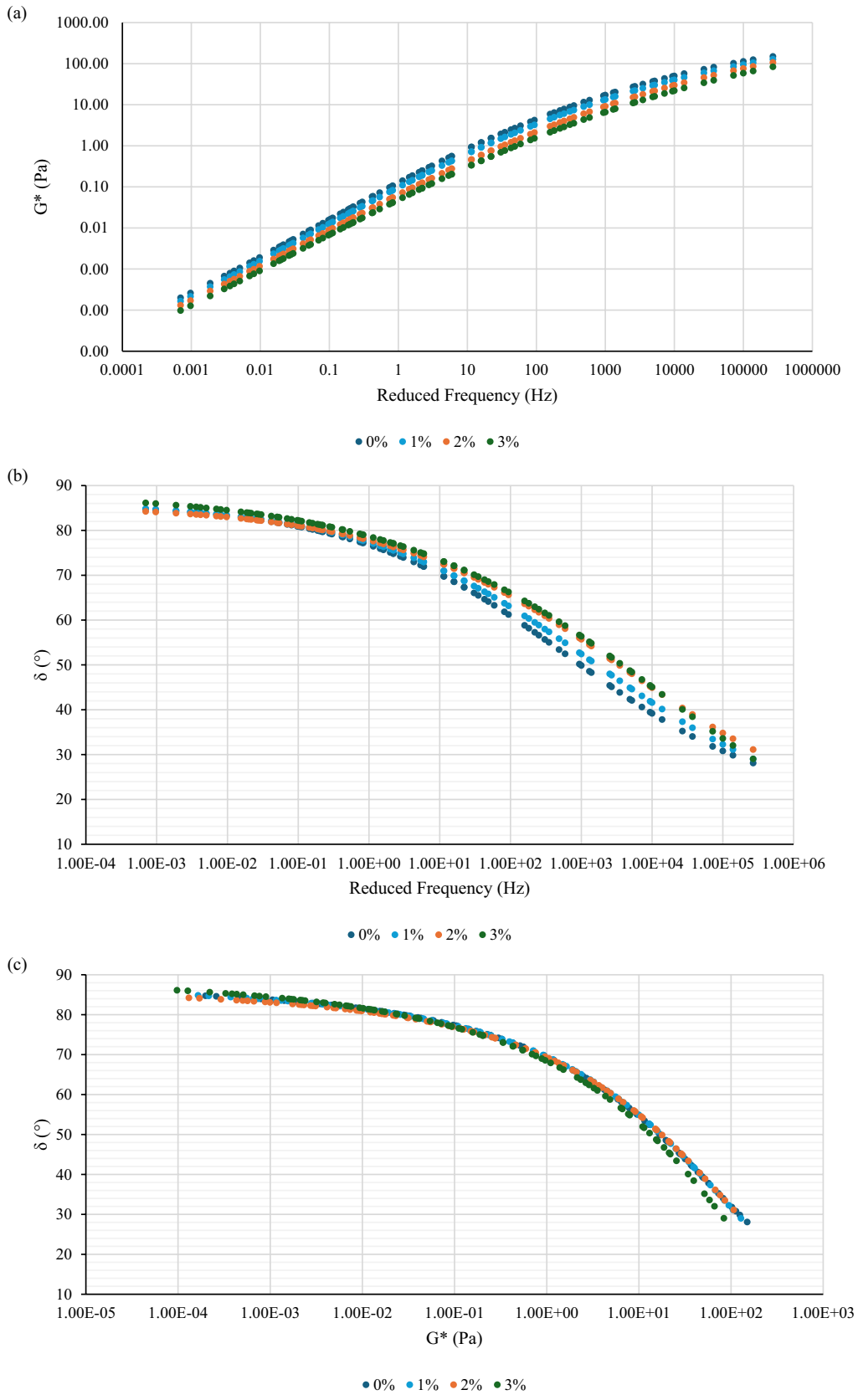


Fig. 3 **a** Master curve of dynamic shear modulus versus reduced frequency, **b** Master curve of phase angle versus reduced frequency, **c** Black curve of phase angle versus dynamic shear modulus

for binders modified with beeswax, which exhibit lower δ values due to the higher elastic fraction after solidification, reinforcing that pequi oil acts as a plasticizing rather than a structuring modifier [45].

Figure 3c presents the Black Curves, which relate δ and |G*| and are used to assess the homogeneity and thermorheological behavior of the modified binders. The obtained curves exhibited a continuous and smooth trend for all samples, indicating that the modification with pequi oil did not compromise the structural uniformity of the binder and preserved its thermorheologically simple behavior. Similar results were reported for binders modified with babassu and moringa oils [13, 15], as well as with beeswax [45], where the thermorheologically simple behavior was maintained up to moderate contents, demonstrating that both fatty and waxy additives can disperse stably within the bituminous matrix.

Figure 4 presents the fatigue results obtained from the LAS test. Figure 4a shows the Wöhler Curves, where the neat binder exhibited the highest number of cycles to failure for all strain levels analyzed. Increasing the pequi oil content systematically reduced fatigue life, a behavior also reported for binders modified with babassu and sunflower and moringa oils [13, 15]. In contrast, binders containing beeswax displayed the opposite trend, showing increased fatigue life due to the reinforcement of the elastic structure after cooling, which highlights the distinction between crystalline and liquid modifiers [45].

Figure 4b shows the Integrity versus Damage Curve obtained from the LAS test. The neat binder exhibited the highest initial degradation rate, whereas binders with higher oil contents showed greater residual integrity during damage accumulation. The 3% blend demonstrated the best performance in this regard, suggesting that although it had a lower initial fatigue life (lower N_f), it better resisted damage propagation once initiated. The crossover behavior, in which the 3% blend outperformed the 1% blend at higher damage levels, is explained by the increase in viscous dissipation provided by higher oil concentrations, which enhances damage tolerance even at lower stiffness levels. This behavior is consistent with that reported for binders modified with unsaturated bio-oils, such as babassu and corn [15, 40], where the presence of fatty compounds contributed to greater toughness under accumulated damage conditions.

Figure 4c presents the stress–strain curves obtained from the LAS test. All binders reached their peak stress between 8 and 10% strain, indicating that failure onset occurs at relatively high deformation levels. The binder with 1% oil showed the highest peak stress, suggesting that small amounts of oil may enhance initial deformation resistance. Conversely, the 3% binder exhibited the lowest peak stress and residual strength, revealing a loss of structural cohesion due to excessive softening. Similar results were reported for binders modified with high contents of babassu and soybean oils [15, 42], where increased fluidity reduced resistance to repeated loading, although it improved workability and low-temperature performance.

Figure 5 presents the classification of the Binder Fatigue Factor (BFF). The BFF, calculated from the pseudo strain energy (Eq. 2) as proposed by Underwood [46] and Hintz and Bahia [47], showed a decreasing trend with increasing pequi oil content. This reduction indicates lower fatigue resistance in the modified binders, particularly at high strain amplitudes, which is consistent with the typical behavior of binders plasticized by bio-oils.

$$BFF = \frac{(\log(N_{f1.25\%}) + \log(N_{f2.5\%}))}{2} x\,(\log(0.025) - log\,(0.0125)) \quad (2)$$

where: $N_{f1.25\%}$ is the fatigue life of the binder at 1.25% strain; $N_{f2.5\%}$ is the fatigue life of the binder at 2.5% strain.

In comparison, the results reported by Melo Neto et al. [15] for binders modified with babassu oil show that incorporating this additive also reduces the BFF at low strain amplitudes (1.25–2.5%) but tends to increase it at higher strain levels (15–25%). This behavior was attributed to the ability of babassu oil to enhance the flexibility and energy dissipation capacity of aged binders, acting as an effective rejuvenator and improving fatigue performance under large deformations. This difference can be explained by the distinct chemical and structural characteristics of each oil. Babassu oil contains a higher proportion of saturated fatty acids, primarily lauric and myristic acids, which promote stronger cohesion between phases and reduce brittleness under cyclic loading. In contrast, pequi oil, composed mainly of unsaturated fatty acids such as oleic and palmitic acids, acts primarily as a plasticizer, reducing stiffness but also diminishing the binder's ability to store and dissipate elastic energy. Consequently, pequi-modified binders exhibit a more viscous and less resilient behavior, resulting in lower BFF values within the strain ranges typical of repeated loading.

According to the classification proposed by Nascimento (2015), which defines BFF > 1.57 as "excellent," between 1.31 and 1.48 as "intermediate," and < 1.31 as "inferior," the binders modified with pequi oil exhibited a clear transition from intermediate performance (0%–2%) to inferior performance (3%). This continuous decrease contrasts with the behavior observed for binders modified with babassu oil, in which contents between 3 and 7% remained predominantly within the intermediate range, showing improvement at higher strain levels and indicating a better balance between flexibility and strength.

Therefore, although pequi oil demonstrates potential as a sustainable modifying agent, its effects on fatigue resistance

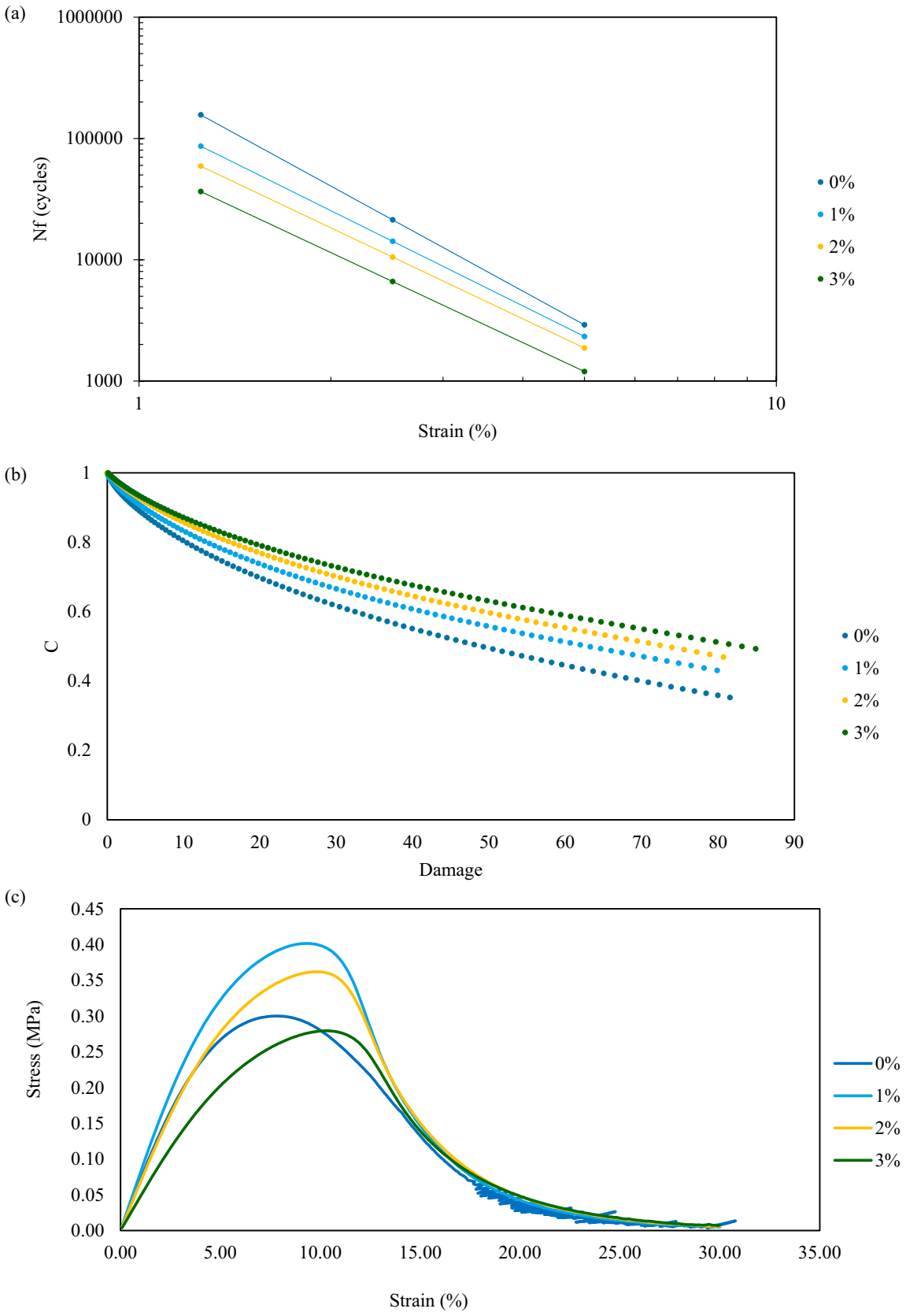


Fig. 4 **a** Wöhler curves for fatigue obtained from the LAS test, **b** Integrity versus damage curve obtained from the LAS test, **c** Stress–strain curves obtained from the LAS test

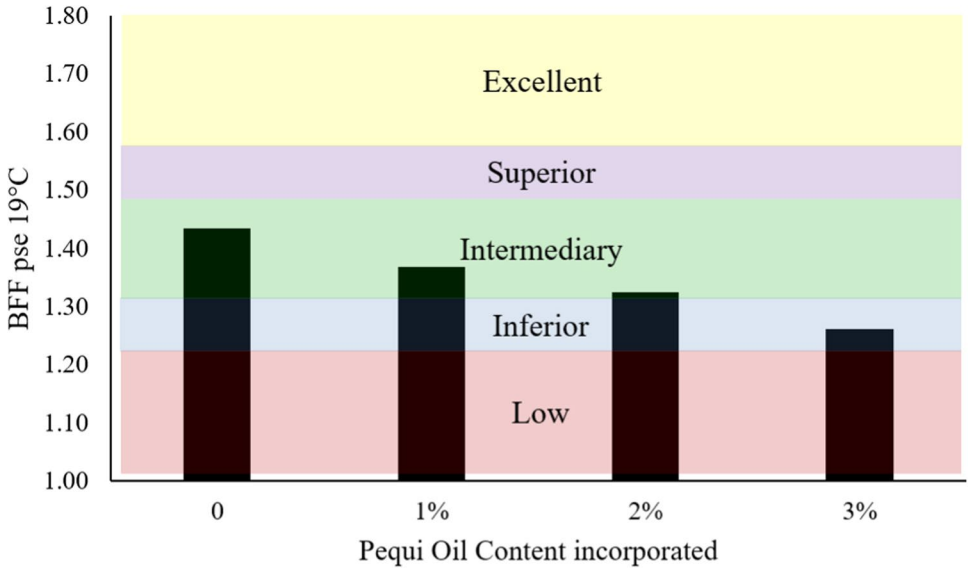


Fig. 5 Binder fatigue factor (BFF) for the modified binders

indicate that increasing the additive content tends to compromise the balance between flexibility and toughness. This trend suggests that, while babassu oil acts as a balanced rejuvenator, pequi oil behaves as a pure plasticizer, making it advisable to limit its use to contents up to 2% to prevent significant loss in fatigue performance.

It is important to highlight, however, that pequi oil was not evaluated in this study using previously aged binders, a condition under which rejuvenating effects could become more evident. As observed for babassu oil, which demonstrated a significant ability to restore viscoelastic properties and reduce the stiffness of aged binders, pequi oil, due to its composition rich in unsaturated fatty acids such as oleic and palmitic acids, may exhibit similar behavior when applied to oxidatively degraded binders. This hypothesis reinforces the need for future research focused on assessing its performance as a rejuvenating agent in recycled mixtures containing RAP, where its plasticizing action could help reestablish rheological and fatigue properties without compromising the binder's structural cohesion.

Overall, the rheological results demonstrate that pequi oil acts as both a plasticizing and rejuvenating additive, reducing the binder's viscosity, G* modulus, and PG rating while mitigating post-RTFO hardening. Similar to other bio-oils such as babassu, soybean, corn, moringa, and sunflower, pequi oil enables lower mixing and compaction temperatures, contributing to the sustainability of asphalt paving. However, increasing the oil content is associated with a reduction in rutting and fatigue resistance, limiting its application in heavy-traffic pavements. Therefore, pequi oil contents up to 2% appear to offer the best balance between flexibility, durability, and mechanical performance, making them technically suitable for warm-mix asphalt applications under moderate traffic and tropical climate conditions.

Mechanistic discussion of the bio-modifier interaction

The observed reduction in Performance Grade (PG) and the plasticizing effect of pequi oil can be explained through the colloidal structure of asphalt binder. According to the Yen–Mullins model [48], asphalt is a complex system in which asphaltene nanoaggregates are dispersed within a maltene matrix. The addition of pequi oil, which is rich in triglycerides and unsaturated fatty acids, acts by solvating the maltene phase and reducing molecular friction between asphaltene clusters [49]. The ester and carbonyl groups identified in the FTIR analysis suggest that the bio-oil interacts with the polar fractions of the binder, promoting a more fluid rheological behavior.

Regarding fatigue performance (LAS), the apparent contradiction between the reduction in N_f and the improvement in integrity (C) indicates a shift in the damage evolution mechanism. Pequi oil appears to increase the viscous dissipation capacity of the binder, promoting micro-scale crack blunting. This process limits rapid crack propagation by dissipating energy through viscous flow, even though the overall stiffness of the system is reduced. Such a mechanism is consistent with the behavior of bio-based fluxing agents that restore molecular network mobility in aged asphalt binders.

Comparative analysis with other vegetable oils

Table 4 presents the rheological data related to the maximum Performance Grade (PG) temperature, both before and after short-term aging, as well as the Jnr values at 3.2 kPa^{-1} obtained for asphalt binders modified with different

Table 4 Rheological data and costs of asphalt binders modified with different vegetable oils*Source:* Adapted from Melo Neto et al. [15]

Samples	PG	PG - After RTFO	Jnr at 3.2 kPa	PG based on climate and traffic	References
PEN 50/70	64	64	4.08	PG 64 (S)-XX	NA
99% PEN 50/70 and 1% vegetable oil					
PEN 50/70 + 1%BO	64	64	4.43	PG 64 (S)-XX	Melo Neto et al. [15]
PEN 50/70 + 1%SO	64	64	2.15	PG 64 (S)-XX	Silva et al. [13]
PEN 50/70 + 1%MO	64	64	2.03	PG 64 (S)-XX	Silva et al. [13]
PEN 50/70 + 1%CPO	64	64	2.10	PG 64 (S)-XX	Neto et al. [30]
PEN 50/70 + 1%CO	58	58	2.30	PG 58 (S)-XX	Mendonça et al. [50]
PEN 50/70 + 1%LO	64	64	3.20	PG 64 (S)-XX	Silva et al. [12]
PEN 50/70 + 1%COR	64	64	3.63	PG 64 (S)-XX	Portugal et al. [40]
PEN 50/70 + 1%SOY	64	64	3.52	PG 64 (S)-XX	Portugal et al. [42]
PEN 50/70 + 1%PE	64	58	5.13	PG 64 (S)-XX	NA
98% PEN 50/70 and 2% vegetable oil					
PEN 50/70 + 2%BO	64	58	2.77	PG 58 (S)-XX	Melo Neto et al. [15]
PEN 50/70 + 2%SO	64	58	2.29	PG 58 (S)-XX	Silva et al. [13]
PEN 50/70 + 2%MO	58	58	2.65	PG 58 (S)-XX	Silva et al. [13]
PEN 50/70 + 2%CPO	64	64	4.00	PG 64 (S)-XX	Neto et al. [30]
PEN 50/70 + 2%CO	58	58	2.80	PG 58 (S)-XX	Mendonça et al. [50]
PEN 50/70 + 2%LO	58	58	3.50	PG 58 (S)-XX	Silva et al. [12]
PEN 50/70 + 2%COR	64	64	4.05	PG 64 (S)-XX	Portugal et al. [40]
PEN 50/70 + 2%SOY	64	64	4.13	PG 64 (S)-XX	Portugal et al. [42]
PEN 50/70 + 2%PE	58	58	6.52	PG 58 (S)-XX	NA
97% PEN 50/70 and 3% vegetable oil					
PEN 50/70 + 3%BO	58	58	3.52	PG 58 (S)-XX	Melo Neto et al. [15]
PEN 50/70 + 3%CPO	64	64	3.40	PG 64 (S)-XX	Neto et al. [30]
PEN 50/70 + 3%CO	58	58	3.90	PG 58 (S)-XX	Mendonça et al. [50]
PEN 50/70 + 3%LO	58	58	3.80	PG 58 (S)-XX	Silva et al. [12]
PEN 50/70 + 3%COR	64	58	3.41	PG 58 (S)-XX	Portugal et al. [40]
PEN 50/70 + 3%SOY	58	58	2.88	PG 58 (S)-XX	Portugal et al. [42]
PEN 50/70 + 3%PE	58	58	8.29	PG 58 (S)-XX	NA
Materials			*R$/kg*		
PEN 50/70			3.85		
Babassu oil (BO)			27.59		
Sunflower oil (SO)			29.5		
Moringa oil (MO)			140.5		
Copaiba oil (CPO)			116.16		
Cottonseed oil (CO)			22.65		
Linseed oil (LO)			45.80		
Corn oil (COR)			23.58		
Soybean oil (SOY)			17.00		
Pequi oil (PO)			115.00		

BO, babassu oil; CO, cottonseed oil; COR, corn oil; CPO, copaiba oil; Jnr, non-recoverable creep compliance; LO, linseed oil; MO, moringa oil; NA, not applicable; PG, performance grade; PO, pequi oil; R$, real; RTFO, rolling thin-film oven; (S), standard; SO, sunflower oil; SOY, soybean oil; XX, not determined

vegetable oils at 1, 2, and 3% contents. In addition, the table includes information on the cost per kilogram of each vegetable oil to enable a comparative analysis among the modifiers. It is important to note that the reported costs correspond solely to the average purchase price and do not account for possible variations related to transport distance. The penetration, softening point, rotational viscosity, and continuous PG data were not included, as some of the studies reviewed did not provide sufficient numerical detail for extraction and standardization. The unit costs of each vegetable oil were determined as the average of prices obtained from three different suppliers, as reported by Melo Neto et al. [15].

The results indicate that incorporating pequi oil into the 50/70 asphalt binder (CAP 50/70) reduces the maximum PG temperature even at a 1% content. At this concentration, the pequi-modified binder (PEN 50/70 + 1% PO) exhibits a PG 64 °C before RTFO but drops to PG 58 after short-term aging, whereas the reference binder (PEN 50/70) remains at

PG 64 °C both before and after RTFO. In contrast, other oils added at 1%, such as babassu (BO), sunflower (SO), moringa (MO), copaiba (CPO), cottonseed (CO), linseed (LO), corn (COR), and soybean (SOY), tend to maintain the same PG grade before and after RTFO, generally PG 64 °C or PG 58 °C, showing no immediate decrease after aging.

This suggests that pequi oil exhibits a stronger plasticizing effect even at low concentrations, reducing the binder's ability to withstand high temperatures after short-term thermal aging. In practical terms, this means that for asphalt mixtures exposed to high service temperatures, the use of pequi oil may lead to an earlier loss of stiffness compared to oils such as moringa and sunflower, which tend to preserve higher PG values at equivalent incorporation levels.

On the other hand, this decrease in PG also represents an advantage: such a reduction in stiffness at high temperatures is associated with lower susceptibility to thermal cracking at low temperatures, a desirable feature in binders that must maintain flexibility and the ability to relax internal stresses. The same rationale has been widely discussed in the literature to justify the use of vegetable oils as recovery or rejuvenating agents for aged binders, particularly in the recycling of RAP materials.

The Jnr parameter at 3.2 kPa, measured at 64 °C, indicates the binder's susceptibility to permanent deformation (rutting). The higher the Jnr value, the lower the resistance to creep. When comparing different modifiers, it is observed that pequi oil consistently produces higher Jnr values than most other vegetable oils across all evaluated concentrations.

At a 1% content, the pequi-modified binder exhibits Jnr = 5.13, while oils such as sunflower (2.15), moringa (2.03), copaiba (2.10), cottonseed (2.30), linseed (3.20), corn (3.63), soybean (3.52), and even babassu (4.43) remain below this level. At 2%, the pequi binder reaches Jnr = 6.52, a value higher than those observed for babassu (2.77), sunflower (2.29), moringa (2.65), cottonseed (2.80), linseed (3.50), corn (4.05), and soybean (4.13). At 3%, this trend becomes more pronounced, with the pequi-modified binder achieving Jnr = 8.29, exceeding babassu (3.52), copaiba (3.40), cottonseed (3.90), linseed (3.80), corn (3.41), and soybean (2.88).

These results reveal two key findings. First, among the studied oils, pequi oil is one of the most effective in reducing the binder's resistance to permanent deformation at high temperatures, particularly at 2% and 3% concentrations. In practice, this means a greater risk of rutting under heavy traffic and hot climates when the binder is modified solely with pequi oil and applied in structural pavement layers subjected to high loads. Second, this implies that for use in high-traffic roadways, pequi oil would likely need to be combined with another modifier that restores the binder's elastic response (for example, an elastomeric polymer) or be restricted to less mechanically demanding applications, such as surface courses of low-volume roads or localized maintenance and thin overlay operations.

This contrasts particularly with the performance of oils such as moringa and sunflower, which, while softening the binder (reducing PG), maintain relatively low Jnr values, suggesting a more balanced window between workability and load-bearing stability. Babassu occupies an intermediate position: it lowers PG, softens the binder, and acts as a rejuvenator, yet keeps Jnr within moderate levels up to 2–3%, explaining why it has been described as technically promising for use in recycled mixtures and asphalt restoration works. Pequi oil, in comparison, behaves as a more "pure" plasticizer, rapidly reducing structural stiffness and increasing creep compliance.

The classification of each binder in Table 4, based on PG after RTFO and the corresponding traffic level ("S" classification, standard traffic), shows that nearly all evaluated systems, including pequi, remain technically suitable for standard-traffic scenarios at their respective PG temperatures but do not qualify for heavy or very heavy traffic when evaluated using the Jnr criterion. This means that, from a regulatory standpoint, pequi oil does not render the binder unsuitable for paving applications but rather limits its use to less demanding service conditions.

Pequi oil is therefore compatible with standard-traffic conditions but less suitable, on its own, for severe-traffic environments, as it combines a PG reduction (lower thermal resistance) with a sharp increase in Jnr (greater susceptibility to permanent deformation). This limitation becomes more critical above 2%, reinforcing that formulations containing 1–2% pequi oil are more technically defensible than those with 3%, particularly in hot-climate regions.

Much of the literature supporting the use of vegetable oils, especially babassu, addresses their application in aged or hardened binders, where the goal is to restore flexibility. In such cases, the reduction in PG and increase in Jnr are viewed less as drawbacks and more as the intended rejuvenating effect for recycled pavements with high RAP content. This suggests that pequi oil may possess a similar potential as a sustainable rejuvenating agent for aged binders, rather than merely serving as a plasticizing additive in virgin binders. This hypothesis warrants further investigation, as it was not experimentally assessed in the present study.

Although Table 4 presents a rheological comparison with other bio-oils, it is essential to recognize the methodological heterogeneity among the studies, including differences in base binders and aging protocols, which limits direct numerical comparisons. Nevertheless, a multicriteria perspective highlights the strategic advantages of pequi oil. From an economic standpoint, although its market price (approximately R$115/kg) may appear higher than that of

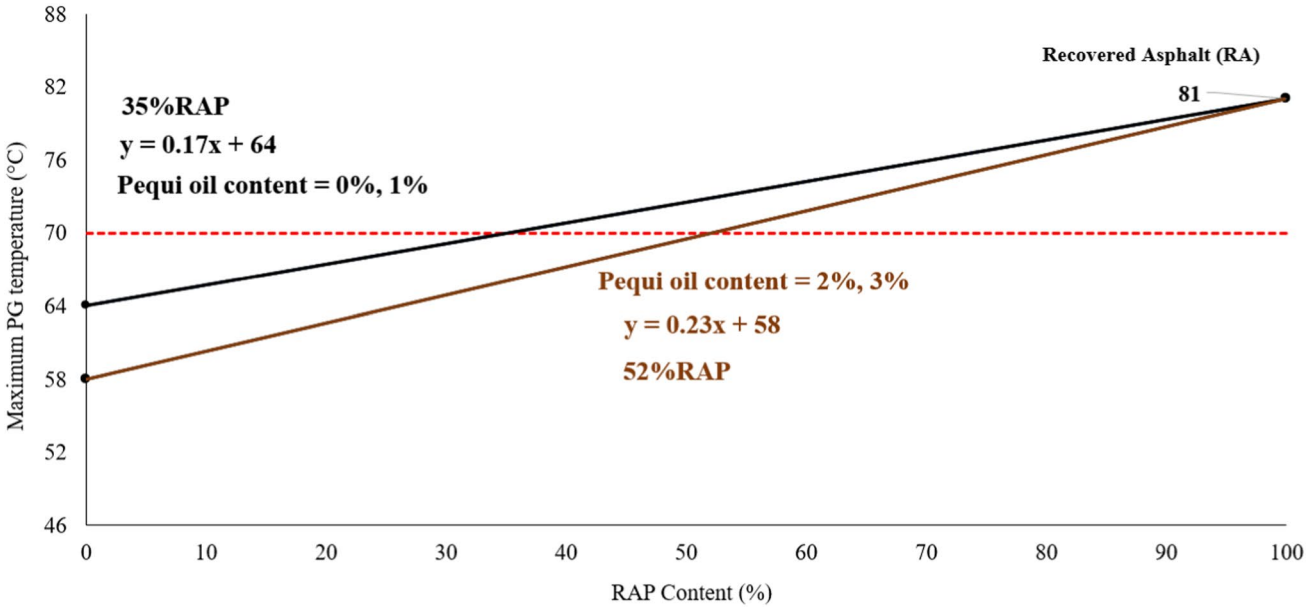


Fig. 6 Blending chart for asphalt binder samples modified with pequi oil

commodity oils such as soybean or babassu oil, this cost is highly sensitive to geographic location. In regions of the Brazilian Cerrado, such as Maranhão and Tocantins, regional availability can significantly reduce logistical costs and transport-related emissions.

From a sustainability perspective, pequi oil offers distinct benefits. First, biodiversity conservation: its valorization encourages the preservation of native Cerrado ecosystems rather than deforestation for monoculture expansion. Second, social impact: harvesting is predominantly carried out by traditional communities, supporting equitable regional development. Third, circularity: the use of non-edible or surplus bioresources aligns with the energy–water–food nexus. Consequently, the higher unit cost can be offset by the reduction of environmental externalities and by the greater socio-environmental value associated with its use compared with large-scale industrial oils.

However, when considering targeted applications, such as hot or cold recycling with high RAP content, localized maintenance works, or sustainable paving programs emphasizing the use of regionally sourced materials, the relative cost of pequi oil can be justified as a technological and environmental differentiator, particularly in regions where the fruit and its by-products are readily available.

Perspectives on the use of pequi oil in pavement engineering

Based on the comparative analysis with other vegetable oils and the verified strong softening capacity of pequi oil, this study sought to estimate its potential not only as a warm-mix asphalt modifier but also as a rejuvenating agent for recycled mixtures containing high RAP contents (>30%). To this end, a Blending Chart (Method B) was developed, using the PG of the virgin asphalt binder (either unmodified or modified) and the PG of the recovered RAP binder to estimate the optimal RAP percentage required to achieve a target design PG temperature. The procedure followed the methodology described in the NCHRP Report 452 [51] and demonstrated in the studies of Melo Neto et al. [52], Costa et al. [53], and Melo Neto et al. [15].

Figure 6 presents the estimated RAP contents corresponding to binders modified with pequi oil at 1%, 2%, and 3%, targeting a design PG temperature of 70 °C. This value was chosen considering that northeastern Brazil, particularly the state of Maranhão, where most of the national pequi production is concentrated, exhibits high surface and internal pavement temperatures due to the regional climate. For this estimation, the PG of the recovered RAP binder (81 °C) was obtained from Melo Neto et al. [29], while the PG values of the virgin binders (modified and unmodified with pequi oil) were determined in the present study.

The results indicated that the incorporation of small amounts of pequi oil (2% and 3%) enabled the use of mixtures containing more than 50% RAP without exceeding the target PG, demonstrating the oil's potential as a recycling

agent in addition to being a warm-mix modifier. This performance is comparable or even superior to that observed in studies involving other vegetable and residual oils. Torres et al. [16] achieved similar RAP contents only with 4% of residual vegetable oil, while Silva et al. [13] reported that 2% moringa oil and 2.5% of either moringa or sunflower oils allowed for 40% and 42.86% RAP incorporation, respectively, both lower than the values obtained in the present study. Furthermore, at 1% additive content, moringa and sunflower oils enabled only 29.41% RAP, whereas pequi oil reached 35%, highlighting its greater softening capacity and rejuvenating efficiency even at low dosages.

Regarding cottonseed oil, Costa et al. [53] demonstrated that its incorporation caused a marked reduction in maximum PG and an increase in fluidity, allowing RAP contents between 15 and 33% in recycled mixtures with a target PG of 64 °C. However, this viability was only achieved with oil dosages ranging from 2 to 12%, significantly higher than those required for pequi oil in this study, which achieved comparable performance with only 2–3% incorporation. This difference reinforces the strong softening capacity of pequi oil and the efficiency of its composition rich in unsaturated fatty acids (oleic and palmitic), capable of restoring flexibility and reducing stiffness in aged binders more effectively than predominantly saturated oils such as cottonseed oil. In comparison, the results from Costa et al. [53] also showed that cottonseed oil provided a good balance between workability and rheological stability, but required higher doses to reach a similar PG range. Pequi oil, although producing a more pronounced softening effect, allows for lower additive consumption and reduced indirect mixing and energy costs, provided its use is controlled to prevent losses in rutting resistance.

Complementarily, Melo Neto et al. [52] investigated the use of soybean soapstock fatty acid (SSFA) as a rejuvenating agent in recycled asphalt mixtures. The authors observed that SSFA was able to significantly reduce the maximum PG of highly aged binders (PG 98 °C) to ranges around 64 °C, enabling the use of 40% RAP with 3% and 5% SSFA, without compromising the overall rheological performance of the mixtures. This behavior demonstrates the strong restorative capacity of SSFA on stiff binders, recovering their flexibility and energy-dissipation ability under cyclic loading.

These observations align the behavior of pequi oil with that of other high-performance bio-oils, such as babassu oil [15], known for its balanced plasticizing action and its ability to restore the viscoelastic properties of aged binders, enabling RAP contents above 50%. Similar to babassu oil, pequi oil reduced the PG of the aged binder from 81 °C to approximately 70 °C, making it suitable for recycled mixtures in regions with high pavement surface and internal temperatures. The integration of Warm Mix Asphalt (WMA) technologies with high Reclaimed Asphalt Pavement (RAP) contents represents a critical frontier for sustainable infrastructure, although it requires careful rheological balancing to ensure adequate performance [9].

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However, it is important to emphasize that the strong softening capacity of pequi oil also entails greater susceptibility to permanent deformation, especially under heavy traffic and high-temperature conditions, as evidenced by the higher Jnr values compared to other vegetable oils. Therefore, the use of pequi oil as a warm-mix modifier should be carefully controlled, limited to dosages up to 2% in structural layers, or combined with complementary additives that enhance the binder's elastic behavior, such as elastomeric polymers.

Furthermore, it is imperative to recognize that these estimates are based on the linear superposition of binder properties, which may not fully capture the complex chemical interactions and aging gradients present in bio-modified systems. Studies with other bio-oils, such as corn oil, have demonstrated that binder-level predictions do not always correlate perfectly with the performance of asphalt mixtures, particularly with regard to moisture susceptibility and long-term fatigue behavior [40].

Thus, pequi oil demonstrates superior potential in terms of dosage efficiency and RAP incorporation, associated with a matrix of predominantly unsaturated fatty acids, a feature that promotes its plasticizing and rejuvenating effects, establishing it as a regionally sustainable and technically comparable alternative to the rejuvenating agents already investigated.

Conclusions

This study evaluated the chemical, empirical physical, and rheological behavior of asphalt binders modified with pequi oil, considering its potential application in asphalt mixtures. The results obtained allow the following conclusions to be drawn:

(1) The FTIR analysis showed that pequi oil induces moderate chemical changes in the binder, with the appearance of characteristic bands of ester and carbonyl groups, without indicating significant oxidation reactions after RTFO aging. This behavior confirms the compatibility between the materials and the stabilizing effect of the oil.

(2) The empirical physical characterization revealed a pronounced plasticizing effect, reflected by the increase in penetration and the reduction in softening point and rotational viscosity as the oil content increased.
(3) The rheological analyses showed a systematic reduction in stiffness (G*) and Performance Grade (PG) with increasing oil content, evidencing the plasticizing and rejuvenating action of pequi oil. At contents up to 2%, the material maintained an adequate balance between elasticity and workability, representing an optimal technical limit for use in warm-mix asphalt without significant loss of resistance to permanent deformation.
(4) The MSCR results confirmed the reduction in elastic recovery and the increase in the Jnr parameter, indicating a more viscous behavior and greater susceptibility to deformation under severe traffic. This effect is beneficial for enhancing flexibility and stress relaxation but requires strict control of the additive content to prevent loss of structural resistance at high temperatures.
(5) The master curves and fatigue analyses (LAS and BFF) demonstrated that pequi oil reduces stiffness and fatigue life at higher contents (3%) but improves residual integrity under accumulated damage—a behavior similar to that of bio-oils with high concentrations of unsaturated fatty acids. Although acting as a plasticizer, pequi oil showed potential for use as a rejuvenator in unaged binders, suggesting that tests on oxidized binders could confirm its effectiveness as a recycling agent.
(6) The feasibility analysis using the Blending Chart indicated that incorporating 1% to 3% of pequi oil allows RAP contents between 35 and 52% for mixtures with a target PG of 70 °C. This efficiency results from its strong ability to reduce stiffness at low dosages, positioning it as a promising alternative for hot-climate regions with high pequi production potential.

Overall, pequi oil demonstrated technical potential for application in sustainable asphalt mixtures, functioning both as a warm-mix additive and as a possible rejuvenator in recycled mixtures. Its use promotes the valorization of regional resources from the Cerrado and may contribute to reducing the carbon footprint of the paving sector, provided that the dosages are optimized and combined with rheological and aging-control strategies.

Study limitations and suggestions for future research

Although the results demonstrate the technical potential of pequi oil as a plasticizing additive and a possible rejuvenator for aged asphalt binders, this study presents some limitations that should be considered. The analyses were conducted using unaged binders, which restricts the direct assessment of the oil's rejuvenating behavior in previously oxidized binders. Future experiments should include different stages of aging—both short- and long-term—to more comprehensively understand the mechanisms of viscoelastic property recovery promoted by pequi oil.

Future work should adopt a phased validation approach, progressing from binder-level rheology to mixture-scale mechanical testing, such as Hamburg Wheel Tracking and Indirect Tensile Fatigue tests, and ultimately to experimental field sections, in order to verify whether the plasticizing effect observed at the binder level translates into durable pavement performance. Consequently, the claim of "superior potential" should remain limited to the chemical softening capacity of the binder under the conditions evaluated.

Future research should also explore the behavior of pequi oil in different types of binders and temperature ranges, as well as assess field performance in pavements subjected to varying climatic and traffic conditions. The integration of rheological, mechanical, and durability analyses will help consolidate knowledge regarding its long-term stability, cost-effectiveness, and overall contribution to the sustainability of Brazilian asphalt pavement technology.

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Data availability The datasets used or analyzed during the current study are available from the corresponding author upon reasonable request.

Declarations

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

Ethical approval I certify that this manuscript is original and has not been published and will not be submitted elsewhere for publication while being considered by Innovative Infrastructure Solutions. And the study is not split up into several parts to increase the number of submissions and submitted to various journals or to one journal over time. No data have been fabricated or manipulated (including images) to support your conclusions. No data, text, or theories by others are presented as if they were our own. The submission has been received explicitly from all co-authors. And authors whose names appear on the submission have contributed sufficiently to the scientific work and therefore share collective responsibility and accountability for the results.

Consent to participate We, the authors, are giving our consent for this paper to be published in your journal if found publishable.

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Human and animal rights and informed consent This article does not contain any studies with human participants or animals performed by any of the authors.

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