Efecto de la adición de ácido graso de soja en ligantes asfálticos mediante espectroscopía de infrarrojo por transformada de Fourier

Effect of the addition of soybean fatty acid on asphalt binders using Fourier-Transform Infrared Spectroscopy

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Resumen

El uso de materiales residuales como agentes rejuvenecedores para ligantes asfálticos recuperados está ganando atención por sus beneficios ambientales y económicos. El ácido graso de jabón de aceite de soja (SSFA), un subproducto del refinado de aceite de soja, fue evaluado como rejuvenecedor mediante espectroscopía de infrarrojo por transformada de Fourier (FTIR). El ligante asfáltico PG 64-XX fue modificado con SSFA en concentraciones del 1%, 3%, 5%, 6% y 7% en peso. Además, el SSFA fue incorporado en ligantes asfálticos recuperados (de Pavimento Asfáltico Reciclado) en concentraciones del 5%, 7%, 10%, 15% y 20%. Los resultados mostraron que la adición de SSFA aumentó los picos de grupos carbonílicos, indicando la restauración de fracciones ligeras pérdidas durante la vida útil del asfalto. Las muestras con mayor contenido de SSFA presentaron picos prominentes alrededor de 1745 y 1715 cm^{-1} y una reducción significativa en la rigidez. Esto demuestra el potencial del SSFA para rejuvenecer tanto ligantes asfálticos vírgenes como recuperados. El uso de SSFA se alinea con los principios de la economía circular, mejorando el rendimiento mientras se reduce el uso de materiales vírgenes y se mitigan los problemas de residuos peligrosos.

Palabras clave: residuos, sostenibilidad, material alternativo, bioeconomía, pavimentos.

Abstract

The use of residual materials as rejuvenating agents for recovered asphalt binder is gaining attention for its environmental and economic benefits. Soybean oil soapstock fatty acid (SSFA), a byproduct from soybean oil refining, was evaluated as a rejuvenator through Fourier-transform infrared spectroscopy (FTIR) analysis. PG 64-XX asphalt binder was modified with SSFA at 1%, 3%, 5%, 6%, and 7% by weight. Additionally, SSFA was incorporated into recovered asphalt binder (from Reclaimed Asphalt Pavement) at 5%, 7%, 10%, 15%, and 20%. Results showed that SSFA addition increased carbonyl group peaks, indicating the restoration of light fractions lost

during asphalt service life. Samples with higher SSFA content displayed prominent peaks around 1745 and 1715 cm⁻¹ and significant stiffness reduction. This demonstrates SSFA's potential to rejuvenate both virgin and recovered asphalt binders. Utilizing SSFA aligns with circular economy principles, enhancing performance while reducing the use of virgin materials and mitigating hazardous waste issues.

Keywords: waste, Sustainability, alternative material, bioeconomy, pavements.

1. Introduction

In view of the high costs and environmental impacts associated with the use of virgin raw materials, the utilization of recycled materials has gained increasing prominence in pavement construction [1]. Reclaimed Asphalt Pavement (RAP) is obtained from milled pavement material and can be incorporated into hot and cold mixed asphalt mixtures [2]. Research indicates that the use of different percentages of RAP in asphalt mixtures can lead to reductions in pollutant emissions and energy consumption, resulting in improved environmental conditions [1]. From analyses with the same purpose, studies have shown that the use of 20 to 50% RAP in new pavements can result in a reduction of 14 to 34% in the cost of new pavement construction [3,4].

The asphalt mixture recycling process consists initially of activating the RAP followed by its incorporation into a new mixture. This process results in a reduction in the amount of virgin binder required in the mixture. Consequently, total costs are reduced as a greater amount of RAP is used [2]. However, a high content of RAP in the composition of new pavements can lead to the emergence of defects, such as cracking, moisture damage, and even pavement fatigue before the project's anticipated lifespan [5,6]. Additionally, research has shown that mixtures containing RAP in their composition exhibit lower resistance to cracking at various temperatures due to increased stiffness [7,8].

The binder present in RAP becomes excessively oxidized over its service life. This effect weakens the pavement as a whole and can cause premature structural damage [9]. Due to its organic nature, asphalt binder naturally oxidizes as it ages due to reactions with atmospheric oxygen [10,11]. During asphalt aging, there is also a reduction in the aromatic content, which is responsible for binder flexibility. The reduction in aromatic content can result in the embrittlement of asphaltenes, leading to their accumulation [12]. Consequently, aging leads to a reduction in mechanical properties and pavement performance as it contributes to binder embrittlement due to increased association forces, resulting in an increase in the volumetric fraction of colloids in the aged asphalt [12,13].

Studies have proposed the use of rejuvenating agents from various sources with the aim of restoring the aged binder to the necessary properties for its reuse [14,15]. Rejuvenators are chemical or biological substances capable of balancing the SARA fractions (saturates, aromatics, resins, and asphaltenes) of aged asphalt. Although the source of the rejuvenating agent may vary, its use results in significant improvements in the performance of the aged binder [12,16,17]. Studies [9,18,19] show that the ability to revitalize the properties of the binder depends primarily on the origin of the rejuvenating agent. The results point to a greater effectiveness in revitalizing aged binder through the use of organically sourced rejuvenators, surpassing those of petroleum origin.

In this regard, bio-oils are capable of compensating for the loss of asphalt components during oxidative aging, acting as a rejuvenating agent [20,21]. In this context, the possibility of using soybean oil soapstock fatty acid arises. Soybean oil soapstock is a residue from the soybean oil refining process [18,19,22]. During this refining process, in the neutralization stage, an aqueous solution of alkalis (sodium hydroxide or sodium carbonate) is added, removing free fatty acids from the oil and other impurities such as proteins, oxidized fatty acids, and by-products of glyceride decomposition.

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Several studies have already demonstrated the physical and rheological effectiveness of using soybean oil soapstock fatty acid in asphalt binder [18,19]. However, there is a gap regarding the analysis of the material addition in RAP-recovered asphalt binder using Fourier-transform infrared spectroscopy (FTIR) to verify the increase in carbonyl and aromatic groups present in the asphalt binder. Therefore, the present research aims to investigate the chemical alterations resulting from the addition of soybean oil soapstock fatty acid in virgin and recovered asphalt binder through Fourier-Transform Infrared Spectroscopy (FTIR) analysis.

2. Materials and Methods

2.1. Materials

2.1.1. Asphalt binder

Due to its widespread use in Brazil, the PG 64-XX asphalt binder with a penetration index of 50/70 was chosen for the research.

2.1.2. Soybean oil soapstock fatty acid (SSFA)

As a modifier agent for both pure and recovered binders, the fatty acid derived from soybean oil soapstock was used. Table 1 presents the characterization of the soapstock used for the acidulation process. The obtaining of the fatty acid occurred analogously to the study by de Medeiros Melo Neto et al. [19] described in Figure 1. The obtained fatty acid presented the following characteristics: total fatty acid content of 90%, moisture content of 2.44%, and pH of 6.52.

Table 1. Characterization of soybean oil soapstock.

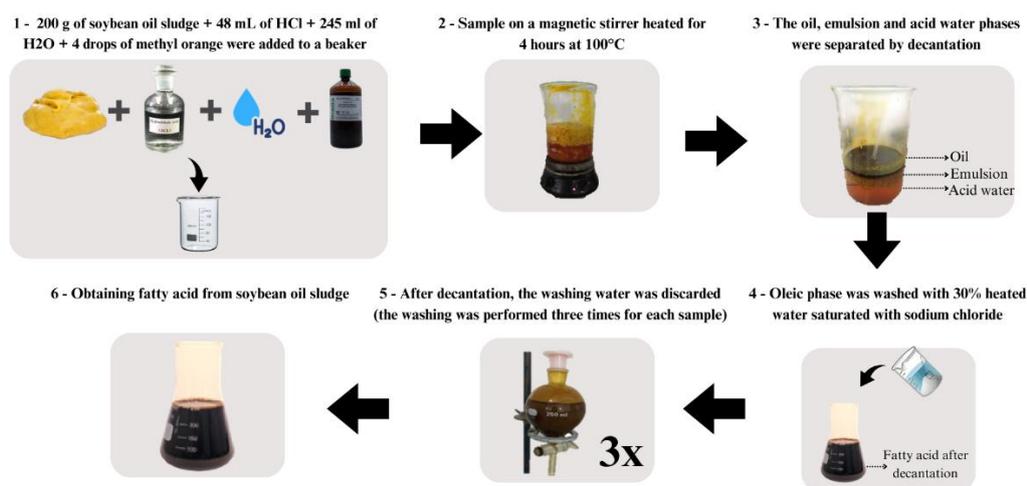| Analysis	Standard	Results
Total fatty acid content	[23] AOCS G3-53	41.60%
Neutral oil content	[24] AOCS G5-40	12.50%
Moisture and volatile content	[25] AOCS Ca 2c-25	42.00%
pH (25°C)	[26] AOCS G 7-56	9.96

Figure 1. Process of soybean oil soapstock acidulation.

2.1.3. Recovered Asphalt Binder

The Reclaimed Asphalt Pavement (RAP) used for binder extraction was obtained from asphalt milling located on highway BR 230 Km 15. The asphalt binder extraction was carried out according to the methodology of de Medeiros Melo Neto et al. [18]. The binder extraction was performed using an electric centrifuge, model Rotarex [27]. Trichloroethylene was used as the solvent [27] and [28]). The extraction process involved preheating the samples in an oven at 110°C for 1 hour, followed by weighing the material directly on the centrifuge plate. Then, 200 ml of solvent was added to the material, and centrifugation was initiated for 15 minutes. This addition process was repeated 3 times. After the process, it was found that the binder content present in the RAP was 5.49%. For the binder recovery process, the material was placed in a rotary evaporator at a temperature of 60°C under a vacuum of 0.035 mmHg for 2 hours. Then, the material was placed in a vacuum oven at 60 mmHg and 70°C for 6 hours. Afterward, the sample was kept in a vacuum oven at 60 mmHg and 85°C for 2 hours. Finally, it was returned to the vacuum oven at a temperature of 100°C for 2 hours.

2.2. Methods

2.2.1. Modification of the asphalt binder

For the modification of the PG 64-XX asphalt binders, the following percentages of soybean oil soapstock fatty acid were used: 1%, 3%, 5%, 6%, and 7%. In the case of the recovered asphalt binder, percentages of 5%, 7%, 10%, 15%, and 20% of fatty acid were used. For the agitation process, a mechanical stirrer, Fisatom 722D model, was used, analogous to the studies by Mendonça et al. [29], Souza Neto et al. [30], de Medeiros Melo Neto et al. [31], and Carvalho et al. [32]. The binder was preheated in an oven at 130°C for 90 minutes until it reached the ideal consistency for mechanical agitation. Then, the material was transferred to the metallic cylinder of the equipment and subjected to continuous mechanical agitation at 600 rpm. Subsequently, the fatty acid was added in the predetermined proportions by weight of the binder. Finally, the stirrer was adjusted to 700 rpm for 20 minutes. It was observed that the homogeneity of the samples was maintained after the modification process.

2.2.2. Fourier-transform infrared spectroscopy (FTIR)

The analysis of the chemical composition of asphalt binders is often performed using the SARA method, which, although effective, can be complex and expensive for separating binder phases. Due to these limitations, several researchers [33] have turned to Fourier-transform infrared spectroscopy (FTIR) to identify SARA fractions in different types of asphalt binders. This technique offers advantages such as speed, reduced cost, and simplified sample handling. The ATR-FTIR measurements were performed at room temperature using a Bruker spectrometer, model FTIR VERTEX 70, following ASTM E1252 standard [34], at the Laboratory of Multifunctional Materials and Nanocomposites at the Federal University of Rio Grande do Norte. Two samples of each binder (pure and recovered) modified by the addition of soybean oil soapstock fatty acid were used, placed directly on the diamond crystal with the aid of a metal spatula. Spectra were collected in the wavenumber range of 4000 to 500 cm^{-1} with 16 scans per analysis at a resolution of 4 cm^{-1}.

2.2.3. Performance Grade (PG)

All samples subjected to FTIR testing were evaluated for stiffness characteristics through the Performance Grade (PG) test standardized by ASTM D6373 [35]. The maximum binder performance was determined by the temperature corresponding to the value of the parameter G*/sinδ greater than 1.0 kPa (for virgin binder) and greater than 2.2 kPa (for recovered asphalt

binder). Continuous PG determination was carried out in 1°C increments over a temperature range of 46°C to 100°C. The test was conducted on a Dynamic Shear Rheometer (DSR). The data obtained in this test are the average of two samples for each composition analyzed. The purpose of this test was to verify the relationship between the addition of SSFA and the reduction in stiffness and increase in carbonyl stretching to demonstrate the material's potential as a rejuvenating agent.

3. Results and Discussion

FTIR analysis is used to identify chemical functional groups within a medium, making it possible to describe chemical changes caused by the addition of rejuvenators and aging of the material under analysis [36]. The intensity of the peaks depends on the concentration of the bonds or functional groups [37]. Spectral peaks corresponding to carbonyl (with a peak at around 1700 cm^{-1}) and sulfoxide functional groups (with a peak at around 1030 cm^{-1}) are widely recognized as relevant when asphalt ages [36,38].

In Figure 2, FTIR spectra for the recovered binder samples added with SSFA (a) and asphalt binder added with SSFA (b) are available. Regarding the characterization of the binders, the main indicative signals of their composition are present in regions common to hydrocarbon compounds, characterized by high-intensity peaks, corresponding to stretches (or axial deformations) for saturated aliphatic groups between 3000 and 2800 cm^{-1}. In this range, peaks were found at 2951 cm^{-1} and 2867 cm^{-1} for asymmetric and symmetric stretching in methyl groups (_CH_3), and peaks at 2919 cm^{-1} and 2850 cm^{-1} for asymmetric and symmetric stretching in methylene groups (CH_2), along with a peak at 2890 cm^{-1} for stretching of methine groups (CH), partially obscured by the 291 cm^{-1} peak.
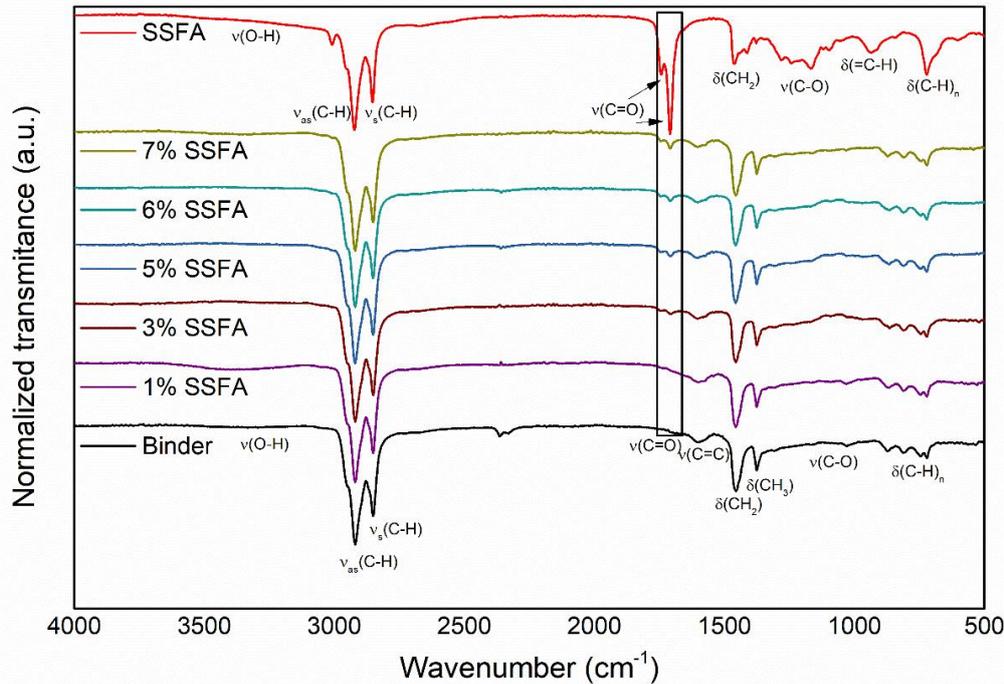
(a)
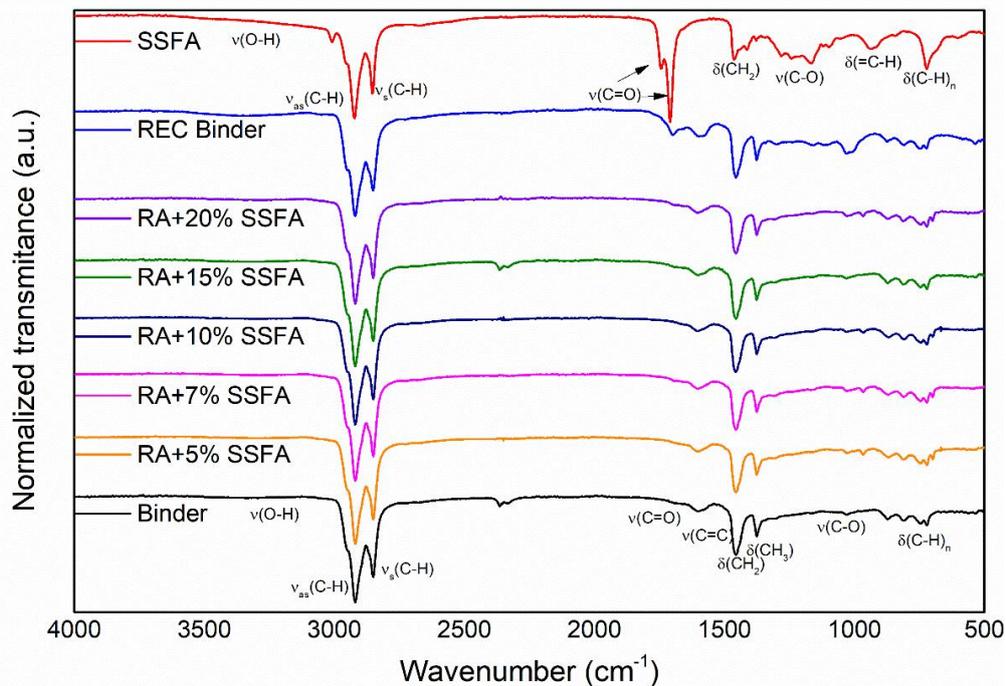
(b)

Figure 2. FTIR spectra for the asphalt binder samples modified with SSFA: **(a)** recovered binder, **(b)** virgin binder.

Peaks with low intensity were found between 2000 and 1700 cm^{-1}, indicating harmonic bands for substituted aromatics, as well as small peaks for carbonyl (C=O) groups in small quantities between 1760 and 1700 cm^{-1}. The presence of a peak around 1600 cm^{-1} is indicative of unsaturations (C=C) in rings, along with peaks around 1478 cm^{-1}, the latter usually obscured by the peak of angular deformation of methylene groups (CH$_2$) at 1455 cm^{-1}. The appearance of the latter is in line with the peaks of this group for axial deformation (2921 and 2850 cm^{-1}), while the peak at 1373 cm^{-1}, referring to angular deformation in methyl groups (CH$_3$), corresponds to peaks of axial deformation at 2952 and 2869 cm^{-1}. Finally, the peak around 720 cm^{-1} corresponds to rocking vibration for saturated chains with more than 4 carbons, (CH$_2$)$_n$, with n$\geq$4, confirming the organic characteristic of the binder with the presence of saturated, unsaturated, and aromatic groups.

In Figure 2, several peaks with relevant intensity were found to indicate a degree of oxidation, such as in regions between 1760 and 1700 cm^{-1}, corresponding to carbonyl (C=O) stretching, or between 1250 and 1000 cm^{-1}, corresponding to C-O stretching groups, especially around 1030 cm^{-1}. Another region where an indicative signal of oxygenated functions was found was between 3200 and 3600 cm^{-1}, related to the vibration of O-H groups (hydroxyls). The increase in intensity of these peaks relative to the intensities of the corresponding ones in the spectra of aged samples, such as the recovered binder. Regarding the spectrum of SSFA, the main peak that distinguishes it from the peaks found in the binder spectra is around 1745 and 1715 cm^{-1}, corresponding to carbonyl groups related to carboxylic acids, resulting from the acidification of the soapstock. These peaks are present in the samples containing SSFA added to the binders, proportionally to the amounts of SSFA in these samples.

Table 2 presents the continuous PG data of the virgin asphalt binder samples and recovered from RAP before and after modification with varying SSFA contents. A greater reduction in values was observed as the incorporated SSFA content increased. Consequently, the highest tested contents, for both binders, showed the highest percentage reduction, being 7% for the virgin binder and 20% for the recovered binder. This allows for the assessment of the softening potential of the additive on the asphalt binder, both in the virgin and aged conditions. However, it is noticeable that the reductions were more pronounced in the virgin asphalt binder than in the recovered asphalt binder,

indicating that aged materials tend to exhibit greater difficulty in softening due to the loss of lighter fractions during the oxidative processes of pavement life.

Table 2. Continuous PG of the asphalt binders.

Continuous PG (°C) | Percentage Reduction (%) - Virgin Binder					
Binder	1%SSFA	3%SSFA	5%SSFA	6%SSFA	7%SSFA
67°C | (0.00%)	65°C | (2.98%)	61°C | (8.96%)	57°C | (14.92%)	55°C | (17.91%)	53°C | (20.90%)
Continuous PG (°C) | Percentage Reduction (%) - Recovered Binder					
Recovered Binder	RA+5%SSFA	RA+7%SSFA	RA+10%SSFA	RA+15%SSFA	RA+20%SSFA
98°C | (0.00%)	97°C | (1.02%)	96°C | (2.04%)	93°C | (5.10%)	92°C | (6.13%)	88°C | (10.20%)

Previous studies [29,39–43] have observed the same trend of reduced stiffness of the asphalt binder with the addition of virgin vegetable oils, residual oils, and residual engine oil. However, vegetable oils tend to exhibit a more significant reduction [44]. Therefore, the softening potential observed in this test aligns with the findings in the FTIR test, where peaks of carbonyl groups related to carboxylic acids are present in the binder samples containing SSFA, proportionally to the amounts of SSFA in these samples. In other words, samples with higher SSFA contents showed greater reduction in stiffness and higher peaks in the spectra around 1745 and 1715 cm^{-1}.

4. Conclusions

This study aimed to evaluate the influence of adding soybean oil soapstock fatty acid (SSFA) as a rejuvenating agent for recyclable mixtures through FTIR testing. Based on the results obtained, the following conclusions were reached:

(i) The addition of SSFA to virgin and recovered RAP asphalt binders resulted in increased peaks in the spectra of carbonyl groups related to carboxylic acids, indicating the potential to restore the loss of light fractions lost by the asphalt binder during its service life in the field.

(ii) The stiffness of the asphalt binders decreased as more SSFA was added to the binder samples.

(iii) Samples showing the highest peaks in the spectra around 1745 and 1715 cm^{-1} were those with higher SSFA content, which consequently exhibited greater stiffness reduction for both tested asphalt binders.

(iv) Samples 7% SSFA and RA+20% SSFA were the least rigid and showed higher peaks in the carbonyl group spectra, converging as the least consistent for virgin and recovered binders, respectively.

Therefore, the FTIR test allowed verifying the feasibility of using SSFA as a rejuvenating agent for asphalt mixtures, where until now, it had only been verified through physical, rheological, and mechanical tests. The use of materials of residual origin, coupled with gains in durability and performance, is associated with the principles of circular economy. This favors the reduction of consumption of virgin raw materials and the storage/treatment of hazardous waste.

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Conflict of Interests

No potential competing interest was reported by the authors.

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