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The Influence of a New Food Waste Bio-Oil (FWBO) Rejuvenating Agent on Cracking Susceptibility of Aged Binder and RAP

Ahmed Abdalla *, Ahmed Faheem * and Berk Ayranci

Department of Civil and Environmental Engineering, College of Engineering Temple University, 1947 N 12th St, Philadelphia, PA 19122, USA; berk.ayranci@temple.edu
* Correspondence: tuh37876@temple.edu (A.A.); afaheem@temple.edu (A.F.)

Abstract: This research aims to deliver a new promising asphalt binder rejuvenator developed from food waste to mitigate the effects of aging on the asphalt. The research relied on three different binders: two unmodified PG 64–22 binders and a Polymer Modified (PMA) PG 76–22 binder. Moreover, a field-extracted RAP binder is utilized in evaluating the rejuvenator's efficiency. For this study, the proposed food waste bio-oil (FWBO) is compared against two market-available rejuvenators. The experimental program relied on aging control binder samples for each asphalt type with no rejuvenation using the Rolling Thin Film Oven (RTFO) test, followed by the Pressure Aging Vessel (PAV) test for 20 hours to create an Artificial RAP (ARAP) binder. Then, ARAP and RAP binders were blended with 5% by their weight with one of the two on-market rejuvenators (#1, #2) or the proposed FWBO rejuvenator. Testing results reveal that low-temperature relaxation was significantly improved for all the investigated samples after an additional PAV aging cycle, as Delta Tc values increased compared to the control binders. Further, samples' master curves were used to calculate the Glover–Rowe (G-R) parameter, crossover frequency, and modulus (ω_c, G_c*). The results clearly showed the ability of the FWBO to reduce the aging rate and improve the rheological properties of RAP binders. Further, the Fourier Transform Infrared Spectroscopy (FTIR) test showed that the new FWBO rejuvenator reduces the oxidation levels of the aged RAP binders, as suggested by the carbonyl index.

Keywords: asphalt binder; rejuvenators; food waste bio-oil (FWBO); aging; oxidation; FTIR; softeners; vegetable-based oil (VBO); petroleum-based oil (PBO); asphalt binder performance

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1. Introduction

Applying reclaimed asphalt pavement (RAP) and recycled asphalt shingles (RAS) in new pavement construction can have significant environmental and economic advantages. Studies have shown that the reuse of RAP saves approximately USD 2.5 billion annually, avoiding the need for vast landfill space and preserving natural resources [1–5]. State departments of transportation (DOTs) limit the amount of RAP to be utilized in a ton of asphalt mixture due to the potential adverse effects of RAP on the performance of the resulting pavement. However, most DOTs allow up to 20% RAP with the asphalt mixtures used [6].

There are three different environmental benefits of reusing RAP. First, it reduces road construction materials waste by incorporating them into new construction projects [7]. Second, it reduces energy consumption and emissions caused by asphalt binder production. Asphalt binder production causes the highest energy consumption and emissions among the pavement materials, such as aggregates. Using RAP, the required binder decreases, which eventually results in less energy consumption and fewer emissions [7,8]. Third, the landfill space required for the disposal of RAP is reduced [9,10]. Zaumanis et al. [8] compared the costs of conventional asphalt mixtures to those of asphalt mixtures containing

RAP. Different RAP (0–100%) material percentages were considered in their study. The authors found that using 50% RAP materials decreased the construction cost by almost 29% of its original cost.

The aged bitumen in RAP loses some of the light components of maltenes during aging, which changes the balance between the asphaltene and the maltene portions. Mogawer et al. [11] showed that aged bitumen has higher stiffness and lower workability than unaged bitumen. Low workability prevents the achievement of proper compaction in the field, leading to premature failure. Thus, replacing some of the bitumen in new pavement construction with RAP bitumen disturbs the colloidal balance of the overall blend. Many studies have researched the chemical performance of aged asphalt binder and RAP. It has been proved that aging increases the carbonyl and sulfoxide contents in bitumen and makes it more susceptible to forming aromatic conjugates [12–15]

Many researchers use recycling agents (also referred to as rejuvenators). Using rejuvenators can enhance the chemical and rheological performance of aged bitumen in RAP. Bio-oil additives are utilized in the asphalt binder industry as asphalt modifiers (<10% asphalt replacement), asphalt extenders (25–75% asphalt replacement), and direct alternative binders (100% asphalt replacement) [16]. Bio-oils sources can be categorized into three categories. The first category is agricultural or forest production wastes that include but are not limited to crop residues (such as maize straw), wood wastes (such as sawdust and bark), and urban organic wastes (such as microalgae). The second category is animal wastes that include but are not limited to swine manure bovine feces. Finally, the third category is post-consume oil wastes that include but are not limited to cooking oil/residues, waste auto engine oil/residues, cottonseed, and soybean oil residues [17,18]. Many studies have indicated that most of these materials could restore the balance lost after aging between asphaltene maltenes and aromatics by using different contents [18–24].

Yan et al. [25] used the surface free energy (SFE) method and molecular dynamics (MD) simulation to study the effect of waste cooking oil (WCO) on the adhesion of asphalt and aggregate from the macroscopic and nanoscopic levels, respectively. The authors concluded that WCO could restore the ratio of asphalt components and thus increase the SFE of WCO rejuvenated asphalt, in which the Lifshitz–van der Waals interaction dominates, and the Lewis acid interaction also increases. Furthermore, researches have shown that Waste Engine Oil (WEO) can reduce the viscosity of aged asphalt, such that, when it is used as a rejuvenator in aged asphalt, the softening point and viscosity of aged asphalt decreases as its content increases [26,27].

Zhang et al. [28] evaluated a bio-based rejuvenator derived from waste wood as an asphalt recycling agent. The study showed that using the wood waste rejuvenator can increase the viscous components and reduce the stiffness of the aged asphalt binder. The high content of light compounds in the bio-oil balanced the chemical compounds of the aged asphalt. As a result, the rutting resistance, fatigue resistance, and low-temperature crack resistance of the aged asphalt binder were significantly restored by the bio-oil. Xinxin et al. [29] proposed a waste vegetable-based oil (VBO) as an asphalt binder rejuvenator. The researchers concluded that the optimum dosage to be used is 13.4 wt%. Compared with virgin asphalt, the rutting property of rejuvenated asphalt and the workability were found to be slightly poorer. On the other hand, the fatigue and low-temperature properties have been significantly enhanced.

Pahlavan et al. [19] examined the merits of coliquefying high protein algae with high-lipid swine manure to form a bio-oil containing a high concentration of nitrogen-containing fused aromatics to intercalate into oxidized asphaltene nanoaggregates. It was found that high-lipid swine manure bio-oil can rejuvenate the aged asphalt found in the reclaimed asphalt pavement by restoring the aged asphalt binder's original chemical balance and molecular conformation. Elkashef et al. [30] introduced soybean oil-derived material to be utilized as a potential asphalt binder rejuvenator. The results suggested that this material is a viable candidate as a rejuvenator. Using the soybean oil-derived rejuvenator at a low dosage improved the fatigue and low-temperature properties of aged asphalt binder. In addition, it led to a notable decrease in the complex shear modulus accompanied by an increase in the phase angle, which is an indication of reversing the effect of aging.

This paper presents a new Food Waste Bio-Oil (FWBO) rejuvenator. The proposed rejuvenator is investigated to study its effect on aged asphalt binder, Artificial RAP (ARAP), and RAP's rheological and cracking performance. In addition, the proposed FWBO is compared to two different market rejuvenators. Rejuvenator #1 is a vegetable-based oil (VBO) and the other, rejuvenator #2, is a petroleum-based oil (PBO).

2. Materials and Methods

2.1. Experimental Design Overview

The experimental program is presented in Figure 1. The program relies on three different binders: Two unmodified PG 64–22 binders and a Polymer Modified (PMA) PG 76–22 binder. These binders are used to create artificial RAP (ARAP) for full testing program with two market available rejuvenators and the proposed FWBO. The ARAP production process is stated in the following section. In addition, an extracted RAP binder is used to confirm the results of the ARAP with rejuvenators. The RAP binder was extracted and recovered from the RAP mixes per AASHTO T-164. Table 1 lists the high, intermediate, and low temperature Performance Grade (PG) for the investigated binders and RAP.

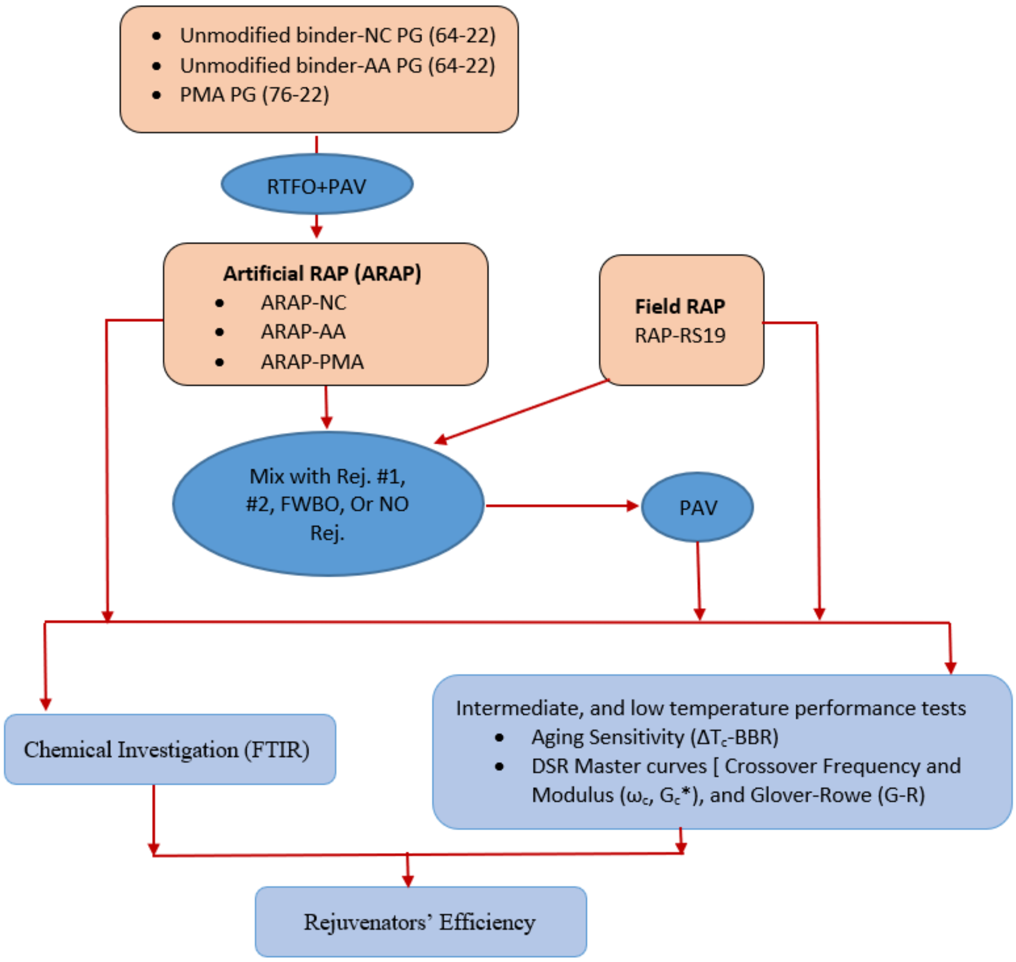


Figure 1. Experimental program.

Table 1. Continuous performance grade (PG) for the investigated asphalt binders and RAP.

	High Temperature			Low Temperature (PAV 20 h)		
Tested Binder	**Original [1]**	**RTFO [2]**	**Inter. Temp. [3]**	**S/MPa [4]**	**m-Slope [5]**	**Continuous PG**
NC PG 64–22	67.4	68.5	22.9	−26.7	−25.7	67–25
AA PG 64–22	69.5	70.0	24.0	−24.1	−23.3	69–23
PMA PG 76–22	79.4	81.4	23.7	−25.6	−24.90	79–24
RAP-RS19	87.3	-	23.3	−29.4	−27.5	87–27

[1] Temperature (°C) corresponds to |G*|/sin(delta) = 1.0 Kpa. [2] Temperature (°C) corresponds to |G*|/sin(delta) = 2.2 KPa. [3] Temperature (°C) corresponds to |G*|*sin(delta) = 5000 KPa. [4] Temperature (°C) corresponds to Stiffness = 300 MPa. [5] Temperature (°C) corresponds to m-value = 0.3.

Three different rejuvenators were used in this study. Two market rejuvenators, VBO and PBO, are referred to as rejuvenators #1 and #2, respectively. In addition, a new vegetable-based FWBO rejuvenator is introduced in this research.

The aging characterization of the blends and binders was quantified using Fourier-transform infrared spectroscopy (FTIR). The test was used to calculate the carbonyl functional group (C=O) increase in asphalt samples with oxidative aging. In addition, aging-dependent rheological behavior was also investigated and utilized to quantify the aging behavior of the samples. Therefore, the testing program included low-temperature performance using the bending beam rheometer (BBR) test according to AASHTO PP-42 to measure the low-temperature ductility. The Glover–Rowe (G-R) parameter, crossover frequency, and crossover modulus (ω_c and G_c*) were also evaluated for the investigated RAP binders. Finally, all samples were tested using two replicates to achieve a coefficient of variation not exceeding 10%.

In general, rejuvenator dosage is determined based on achieving one of the following three conditions: first, restoring the low-temperature grade (PGL) of the binder blend to that of the base binder, or, second, restoring the high-temperature grade (PGH) of the binder blend to that of the base binder, or third, achieving ΔT_c equal to −5 [31]. For this study, achieving ΔT_c equal to or less than −5 °C was set for choosing the rejuvenator dosage. This criterion led to using a 5% dosage for the rejuvenators.

2.2. Fabrication of Blends

For producing Artificial RAP (ARAP), a sample of the binders for each asphalt type with no rejuvenation was exposed to short-term aging using the Rolling Thin Film Oven (RTFO) test per AASHTO T 240. The RTFO aging was followed by the Pressure Aging Vessel (PAV) test for 20 h following AASHTO R 28. The testing matrix included a control "non-rejuvenated" ARAP and field RAP to serve as a baseline for comparisons. Then, ARAP and RAP were blended with 5% by their weight with one of the two on-market rejuvenators (#1, #2) or with the new proposed FWBO rejuvenator using a high shear mixer with a shear rate of 380 rpm. The blending process was completed over 30 min. Following the application of the rejuvenators, the samples were put through an additional PAV cycle. It is important to note that the un-rejuvenated ARAP and RAP binders' samples were exposed to the same mixing protocol as a control sample to avoid fabrication/conditioning bias influencing the ARAP and RAP properties. This process yielded four RAP binders (three ARAP binders and one field RAP binder) and sixteen unrejuvenated/rejuvenated samples (Three ARAP binders and one RAP with three rejuvenators and no rejuvenator).

2.3. Chemical Investigation through Infrared Spectroscopy (FTIR)

The principal mechanism of infrared spectroscopy is that a beam of infrared radiation passes through a sample and a spectrometer analyzes the transmitted radiation. The spectrum of transmitted radiation, either transmittance or absorbance, shows absorbed radiations at specific wavelengths. This absorbed radiation at a particular wavelength corresponds to the absorbance due to the vibration or rotation of a specific chemical bond when the frequency of vibration (or rotation) of the bond matches that of the applied radiation. It was reported that, due to aging, the carbonyl functional groups (C=O) increase in asphalt samples because of the oxidation reaction [12–14,32–35]. The relative increase or decrease in the previously mentioned functional group compounds in the samples is determined through the following equations proposed by Grenfell et al. [36]:

$$\text{The carbonyl Index (ICO)} = \frac{\text{Area around 1700 cm}^{-1}}{\text{Area around 1460 cm}^{-1} + \text{Area around 1375 cm}^{-1}} \quad (1)$$

The numerator in Equation (1) corresponds to the peak area due to the stretching of ketonic bonds. The denominator corresponds to the bending of C–H bonds in the spectrum of the binder sample. The relative area in a particular spectrum was considered in this case instead of the height or magnitude of the peaks. The areas around the peaks of interest were calculated by constructing a baseline from valley to valley, as shown in Figure 2. FTIR was carried out on four samples for each binder and mastic to account for repeatability, achieving a coefficient of variation (CoV) below 10%.

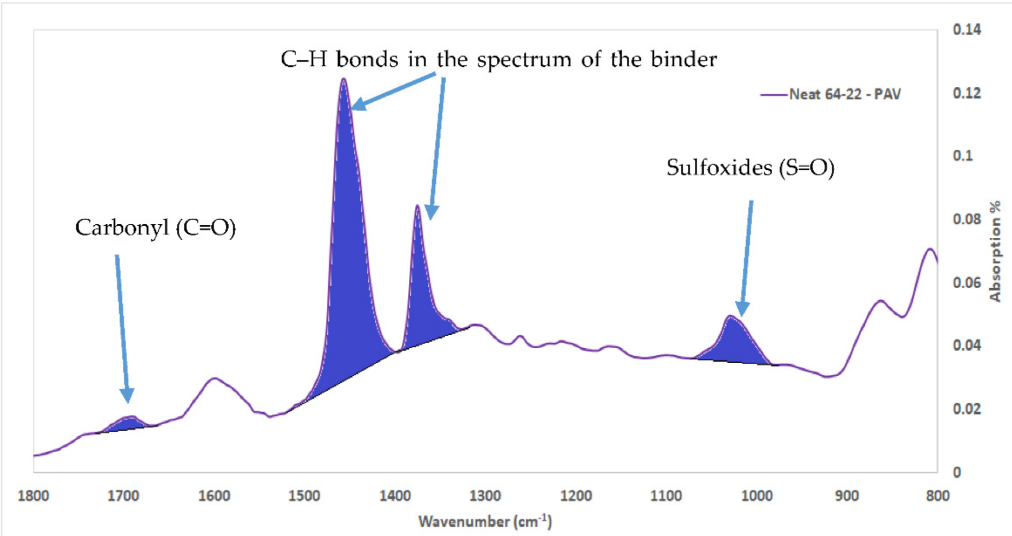


Figure 2. Illustration of determination of peak areas for the unmodified binder NC after PAV aging.

2.4. Measuring Low-Temperature Performance of Blends after Aging

The Bending Beam Rheometer (BBR) test was used to determine the impact of aging on cracking potential on asphalt binder. The critical low temperature of an asphalt binder is determined based on the values of the stiffness modulus at 60 s, S (60 s) and the slope of the stiffness modulus curve at 60 s, m (60 s). The loss in the relaxation capacity of the asphalt binder at low temperatures due to aging leads to reduced ductility and durability and an increase in non-load-associated cracking potential during the asphalt's service life. Accordingly, Anderson et al. (2011) introduced the ΔT_c parameter, which is the numerical difference between the critical low temperature corresponding to 300 MPa of Stiffness, S (60 s), and the critical temperature that corresponds to 0.30 slope of the stiffness curve, m (60 s). Pavements with a rapid aging rate are prone to thermal cracking due to poor binder thermal relaxation. Accordingly, the ΔT_c parameter is vital in measuring the ductility loss of aged asphalt, quantifying the effect of binder aging [37]. For ΔT_c, a crack warning value of −2.5 °C and a cracking limit value of −5 °C were suggested [37,38].

2.5. Glover–Rowe Parameter (G-R), Crossover Frequency, and Modulus (ω_c), (G_c)*

The Glover–Rowe (G-R) parameter is utilized to evaluate the effect of aging on the blends' ductility. Before the Strategic Highway Research Program (SHRP) was established, the ductility test (at 15 °C) was used to assess binders for susceptibility to cracking at intermediate temperature. In 2005, Glover conducted a comprehensive study on the aging of asphalt binder and, in that study, Glover presented the DSR function ($G'/(\eta'/G')$ measured at 0.005 rad/sec and 15 °C [39]. The DSR function strongly correlated with the ductility test [39]. Anderson et al. [38] conducted a field study in which the DSR function was validated as a good candidate for identifying loss of ductility that may lead to non-load associated cracking in pavements. The G-R parameter shown in Equation (2) was a reformulation of the DSR function [40]:

$$G - R = \frac{G^{*}(\cos\delta)^{2}}{\sin\delta} \tag{2}$$

where, G*—complex shear modulus (kPa); and δ—phase angle (degree).

The crossover frequency and modulus (ω_c, G_c*) are rheological parameters that quantify aging susceptibility. Crossover modulus is defined as the complex shear modulus when the phase angle equals 45°, where the storage modulus equals the loss modulus. The crossover modulus is a unique point on the material's viscoelastic spectrum, not depending on the test frequency and temperature. Increased crossover values indicate an increased viscous and elastic component of the materials. The crossover modulus is inversely related to aging. The frequency sweep tests determined the crossover modulus G_c* from the master curves of the complex shear modulus and phase angle.

Similar to the crossover modulus, the crossover frequency (ω_c) corresponds to the frequency at which the phase angle (δ) is 45°or $G' = G''$, which indicates the equal contribution of the elastic and viscous components of G*. Some rejuvenators mainly act as a softener by influencing the viscous component of asphalt. Other rejuvenators restore both the viscous and elastic performance of aged asphalt. It has been shown that nearly all so-called rejuvenators soften the aged bitumen and increase its crossover frequency, which measures bitumen's vicious behavior. However, not all rejuvenators increase the crossover modulus, which measures the extent of polydispersity. Asphalt binder's polydispersity increases with aging and is inversely related to crossover modulus [5,41]. Therefore, crossover modulus and frequency are reliable tools for evaluating rejuvenator efficiency. A concurrent increase in crossover modulus and crossover frequency is needed to show successful rejuvenation, and not only softening agent [5,41].

The crossover frequency and modulus (ω_c, G_c*) and Glover–Rowe parameter (G-R) were obtained through the DSR frequency sweep test. The DSR frequency sweep test was conducted on all blends and binders. For PG 64-22 blends/binders and field RAP, the test was conducted at 10, 22, 34, 46, 58, 64, and 70 °C, over a frequency range from 100 to 0.1 rad/s at each temperature. For PMA PG 76-22 blends and binder, the test was held at 10, 22, 34, 46, 58, 70, and 76 °C over a frequency range from 100 to 0.1 rad/s. The amplitude strain was set to 0.1% to attain linear viscoelastic range behaviors for the blends and binder tested.

3. Test Results and Discussion

3.1. Analysis of Infrared Spectroscopy (FTIR)

Table 2 shows the change in the FTIR results for the different investigated ARAP and RAP at different aging levels. Thus, FTIR monitored the main changes in the chemical functional groups between the two aging levels for the different investigated ARAP and RAP. The data shown are the average of four replicates. The spectral peak of interest was for the carbonyl peak at 1700 cm^{-1}. To analyze the carbonyl group with the presence of rejuvenators, these spectra were normalized using a reference of bending of C–H bonds in the spectrum of the binder sample. The higher the carbonyl index, the greater the oxidation level and aging, accordingly.

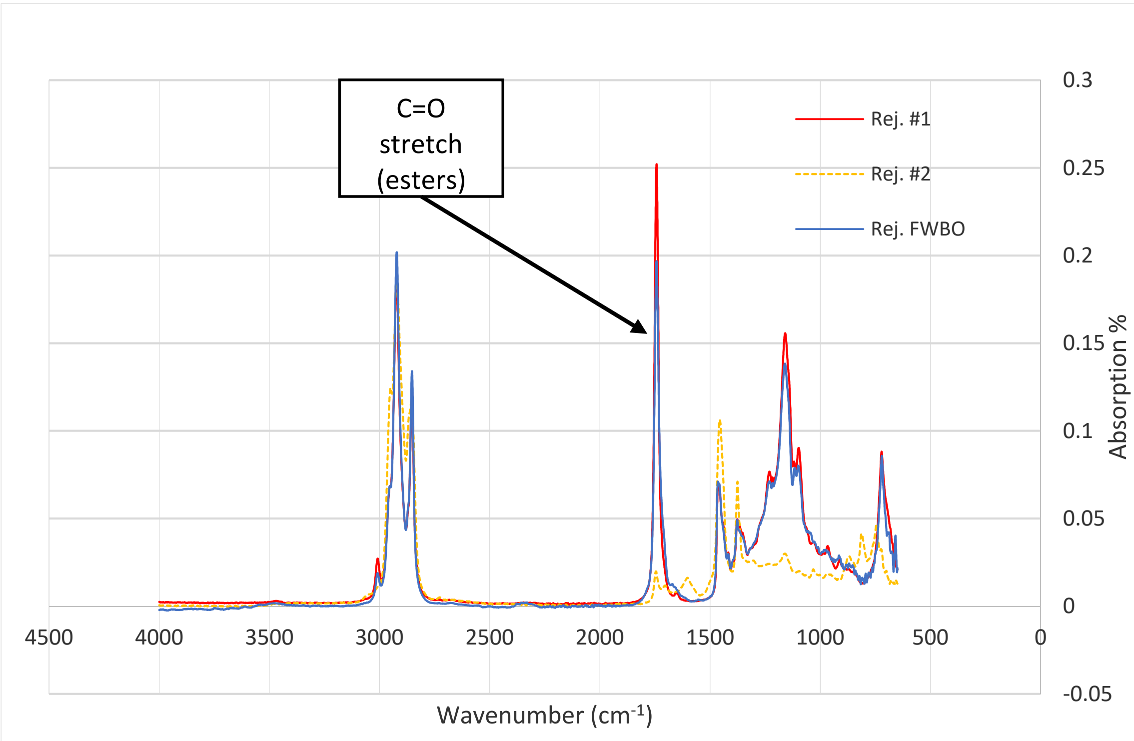
Table 2. Carbonyl index (ICO) for rejuvenated and unrejuvenated RAP binders.

Asphalt Binder /RAP	ARAP@1st PAV or Field RAP	@ 2nd PAV		RAP + Rej. #1 @ 2nd PAV		RAP + Rej. #2 @ 2nd PAV		RAP + FWBO @ 2nd PAV	
		ICO	Relative to RAP *	ICO	Relative to RAP	ICO	Relative to RAP	ICO	Relative to RAP
NC PG 64–22	0.172	0.168	−2.59%	0.157	−8.87%	0.133	−22.76%	0.169	−1.87%
AA PG 64–22	0.125	0.160	28.17%	0.145	16.12%	0.178	42.81%	0.110	−12.28%
PMA PG 76–22	0.131	0.138	4.61%	0.121	−8.28%	0.116	−11.45%	0.120	−8.51%
RAP-RS19	0.139	0.141	1.61%	0.144	3.76%	0.145	4.29%	0.139	0.00%

* RAP term includes both Artificial RAP and field extracted RAP.

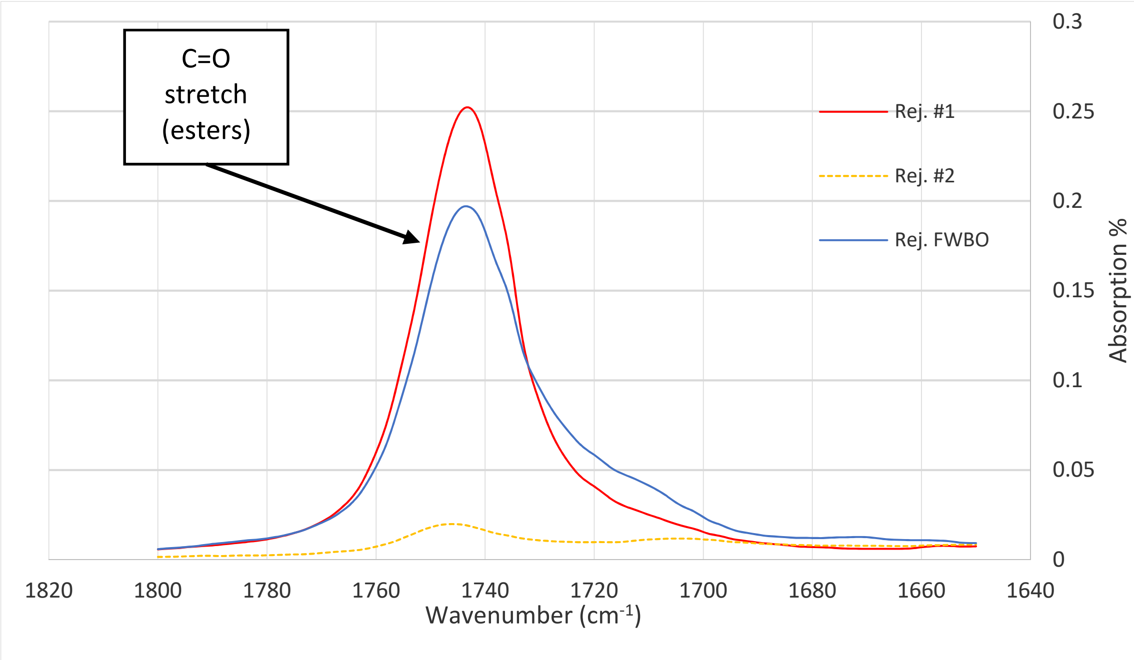
As mentioned above, the different rejuvenators are introduced to the ARAP and RAP and are then subjected to the PAV aging cycle. Figure 3 shows the influence of the different rejuvenators when blended with the investigated RAP binders. Rejuvenator #1 is a vegetable-based bio-oil (VBO), and rejuvenator #2 is a petroleum-based bio-oil (PBO). The new proposed FWBO also has a VB nature. The ester group (1735 cm^{-1}, 750 cm^{-1}) referred to in Figure 3 can clearly distinguish between the PBO and VBO. The VBO contains the ester groups, as shown on rejuvenators and rejuvenated and unrejuvenated binders.

Table 2 lists the calculated values of ICO for the different investigated RAP binders. The results show that further aging applied on both the ARAP and RAP binders caused minimal changes in the measured ICO. Only the ARAP-AA binder shows a noticeable increase in ICO by 28%.

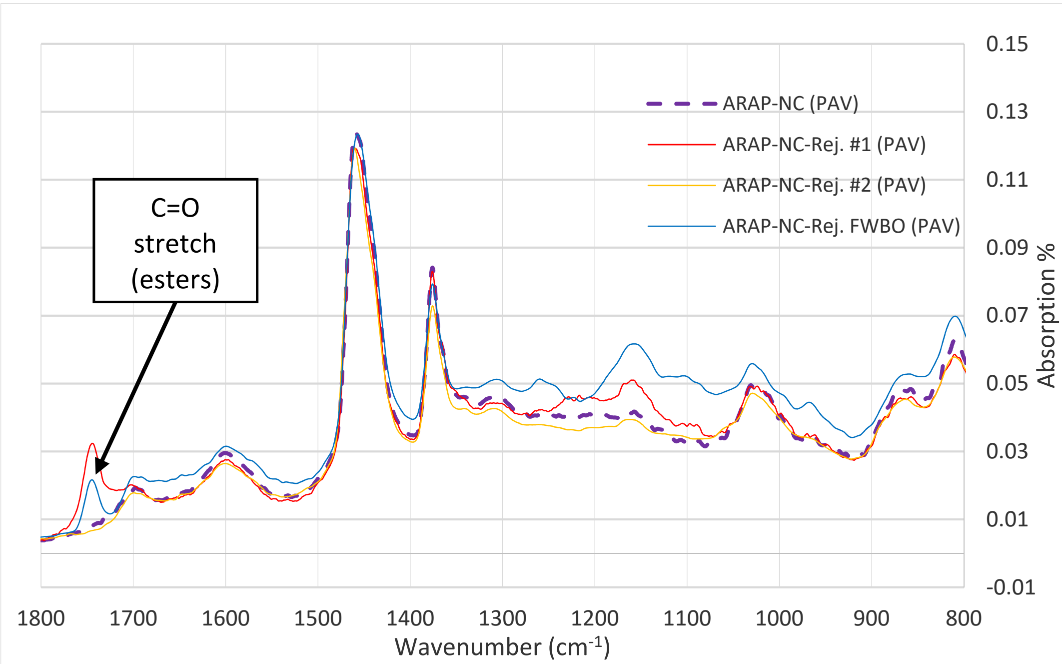


(a)

Figure 3. *Cont.*



(**b**)



(**c**)

Figure 3. Normalized FTIR for (**a**) full spectra rejuvenators used, (**b**) magnified area, (**c**) PAV-aged rejuvenated and unrejuvenated unmodified NC binder. (Symbol "#" is rejuvenator number).

Both rejuvenators #1 and #2 are found not to positively influence the aging index ICO when introduced to ARAP-AA and RAP-RS19. Rejuvenator #1 drops the ICO by 8.9% and 8.3% when introduced to the ARAP-NC and ARAP-PMA binders, respectively. Introducing rejuvenator #2 to ARAP-NC and ARAP-PMA drops the aging index ICO values by 22.8% and 11.5%, respectively.

For the FWBO rejuvenator, it is the only rejuvenator showing a consistent reduction in the ICO aging index when introduced to the different investigated ARAP/RAP. It drops the ICO values by 1.9%, 12.3%, and 8.5% when blended with the ARAP-NC, ARAP-AA, and ARAP-PMA, respectively. The new FWBO is the only rejuvenator that maintained the ICO level of the field RAP at a constant level after the second cycle of PAV.

Understandably, the results of the spectroscopy indices provide a more qualitative evaluation. This is because (1) it depends on small samples that may have issues representing the bulk material, and (2) the approximate method in calculating the areas under the appropriate peaks of the output.

Both rejuvenator types (petroleum-based and vegetable-based) present an adequate amount of maltene constituents that rebalance the composition of the aged binder at distinct levels. However, it can be concluded that the introduction of the VBO rejuvenators, especially the newly proposed FWBO, is associated with the higher recovery of PAV-aged RAP binders. This could be due to the predominance constitution of esters with saturated and unsaturated fatty acids for the VBO rejuvenators, making the vegetable-based rejuvenator behave as a nonpolar oil. On the other hand, the PBO rejuvenators are products with a predominance of polar aromatic molecules. These interpretations have been referred to by several authors [7,28–30,42–44].

In 2014, Yu et al. [45] indicated that the recovery of aged binders, by adding aromatics, was less significant than observed when organic vegetable bio-oils were employed. The argument was that the petroleum-based rejuvenator is free of carbonyl groups. Accordingly, the carbonyl index presented by the PB rejuvenated binders directly results from the aging of the asphalt binder, implying that aging is more evident in PB rejuvenated binders than in VB rejuvenated binders. Therefore, vegetable-based bio-oil rejuvenators show a better and safer alternative to petroleum-based rejuvenators, not only from a technical point of view, as the FTIR results prove, but also because of its less environmental and occupational health impacts [28,30,46].

The asphalt binder aging process is very complex and involves changes in chemical composition and rheological properties. The chemical composition change is irreversible, including oxidation, loss of volatiles, and the formation of highly polar functional groups. The rheological properties change a reversible process related to physical hardening attributed to the reorganization of the binder molecules [44]. Accordingly, cracking and ductility-related evaluation is conducted.

3.2. Thermal Cracking and Relaxation Performance Evaluation

ΔT_c is used for evaluating the age-related cracking potential at low temperatures. The lower the value of ΔT_c, the lesser the ability of the binder to relax under thermal stresses as the pavement ages. Figures 4 and 5 demonstrate the results related to low-temperature performance. Figure 4 demonstrates that all the investigated RAP binders are more susceptible to aging. The ARAP-AA is more prone to thermal cracking than the other investigated RAP binders, as its ΔT_c value drops the most by 343%. On the other hand, ARAP-NC, ARAP-PMA, and RAP-RS19 binders' ΔT_c values are found to drop by 286%, 178%, and 75%, respectively.

Figure 5 demonstrates the effect of the different rejuvenators when introduced to the different RAP binders. In general, introducing rejuvenators to the aged RAP binders increases the resistance to thermal cracking. Rejuvenator #1 is found to improve the performance of ARAP-NC and RAP-RS19 by 45% and 26%, respectively, as measured by ΔT_c. Rejuvenator #2, the PBO rejuvenator, has no significant improvement in thermal cracking resistance when introduced to ARAP-AA and RAP-RS19 but significantly increases ΔT_c for ARAP-PMA by 122%.

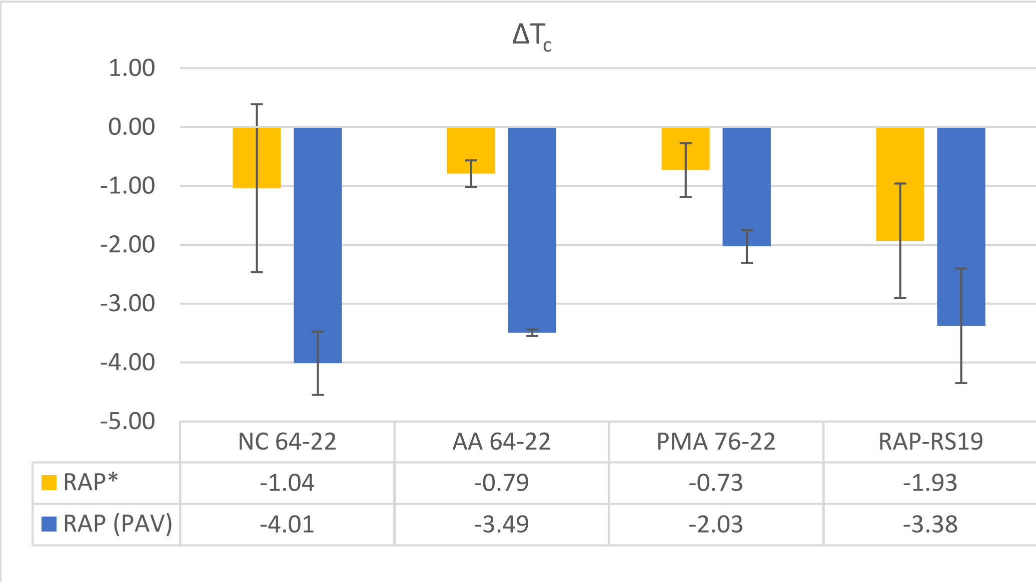


Figure 4. ΔT_c of the investigated RAP binders before and after second cycle of PAV aging. * RAP term includes both artificial RAP and field-extracted RAP.

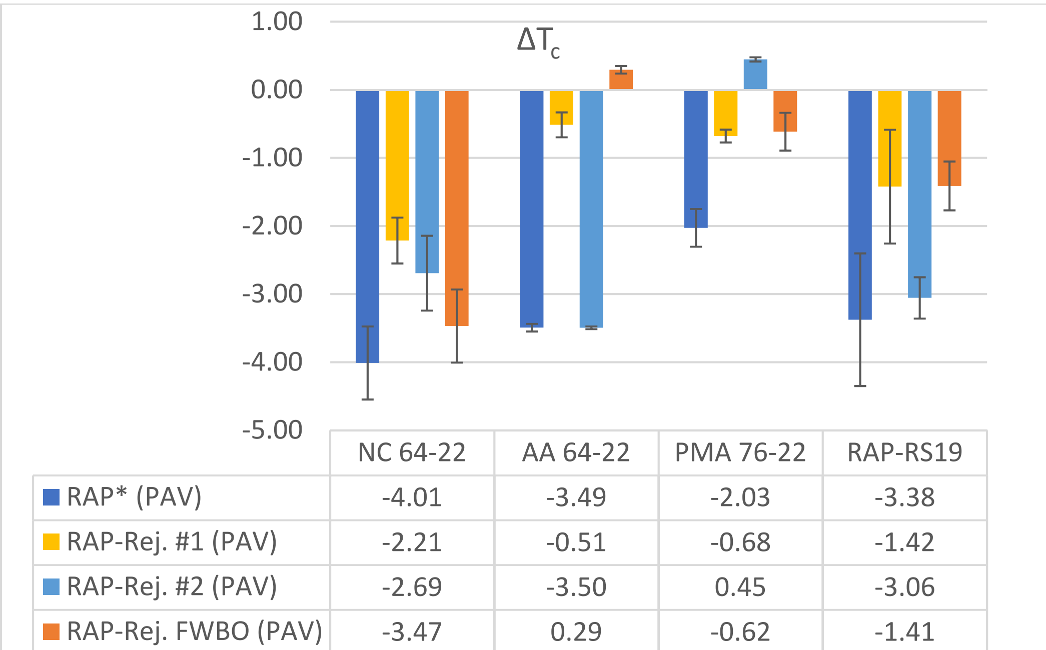


Figure 5. ΔT_c of the investigated PAV-aged rejuvenated and unrejuvenated RAP binders. * RAP term includes both artificial RAP and field-extracted RAP. (Symbol "#" is rejuvenator number).

The new proposed FWBO significantly enhances the resistance to thermal cracking when introduced to all the investigated RAP binders. The ΔT_c values ARAP-NC, ARAP-AA, ARAP-PMA, and RAP-RS19 are found to increase by 13%, 110%, 70%, and 26%, respectively, compared to their corresponding PAV-aged unrejuvenated RAP. These results show the effectiveness of FWBO in retarding the aging of the different binders compared to the two on-market rejuvenators, especially when introduced to the ARAP-AA and RAP-RS19. It is important to note that all the results of the blends and binder are within the acceptable performance recommended by Anderson et al. [37].

Figure 6 shows the ICO index correlation with the ΔT_c. It is clear that the PBO has the lowest rejuvenating effect compared to the VBO rejuvenators. On the contrary, as illustrated, the VBO rejuvenators can retard the aging indices to a level exceeding RAP binders' original condition before being subjected to PAV aging.

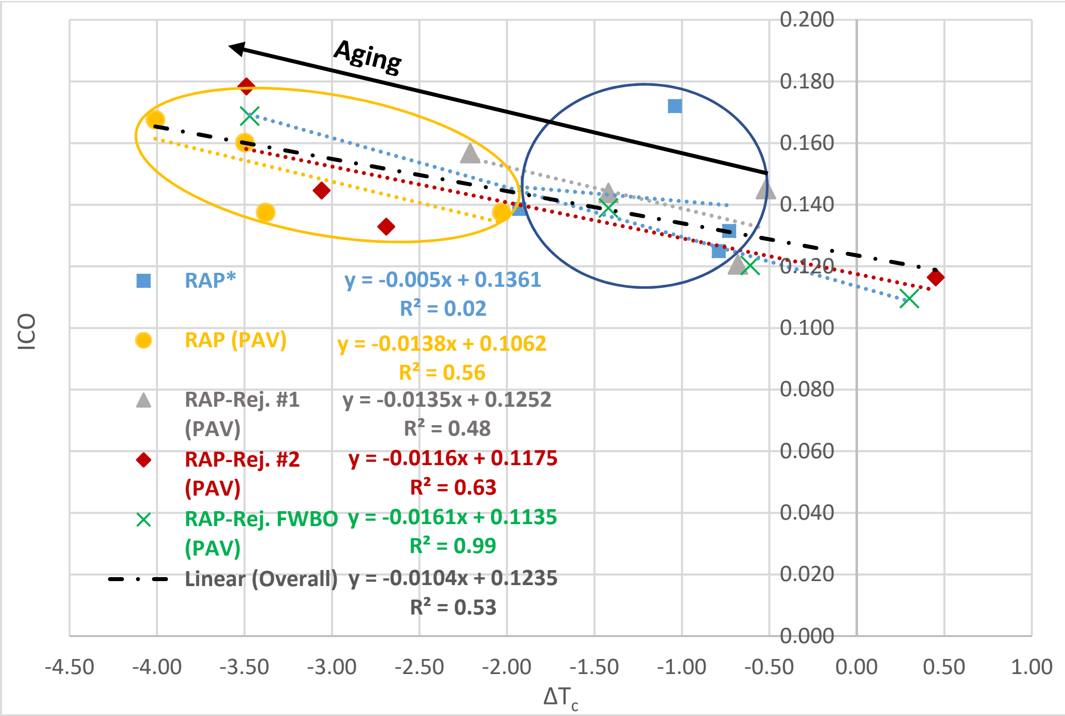


Figure 6. Correlation between the carbonyl index (ICO) and ΔT_c of the investigated rejuvenated and unrejuvenated RAP binders. * RAP term includes both artificial RAP and field-extracted RAP.

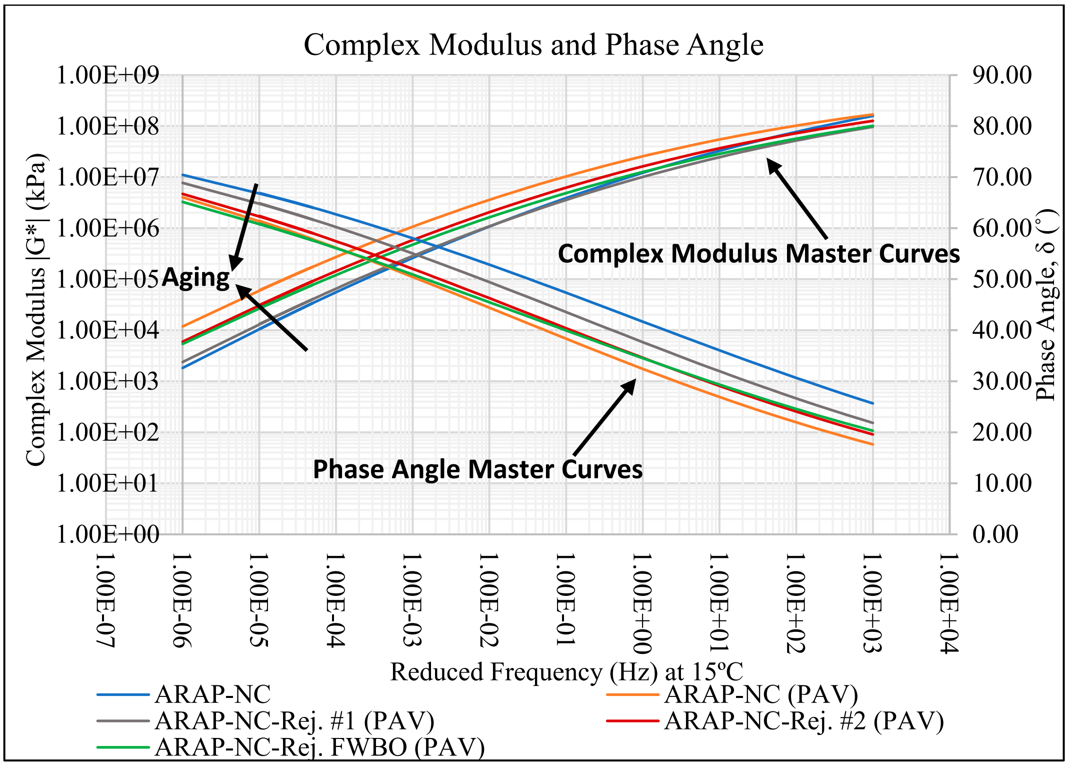
3.3. Frequency Sweep Test Results

The tested binders' stiffness and phase angle were obtained from the asphalt binder frequency weep test. The Christensen–Andersen–Marasteanu (CAM) model, as shown in Equation (3), was used to construct the asphalt and RAP master curves. The shift factor was estimated based on the Williams–Landel–Ferry WLF equation. Figure 7 depicts the complex modulus and the phase angle master curves for the tested asphalt binder and RAP.

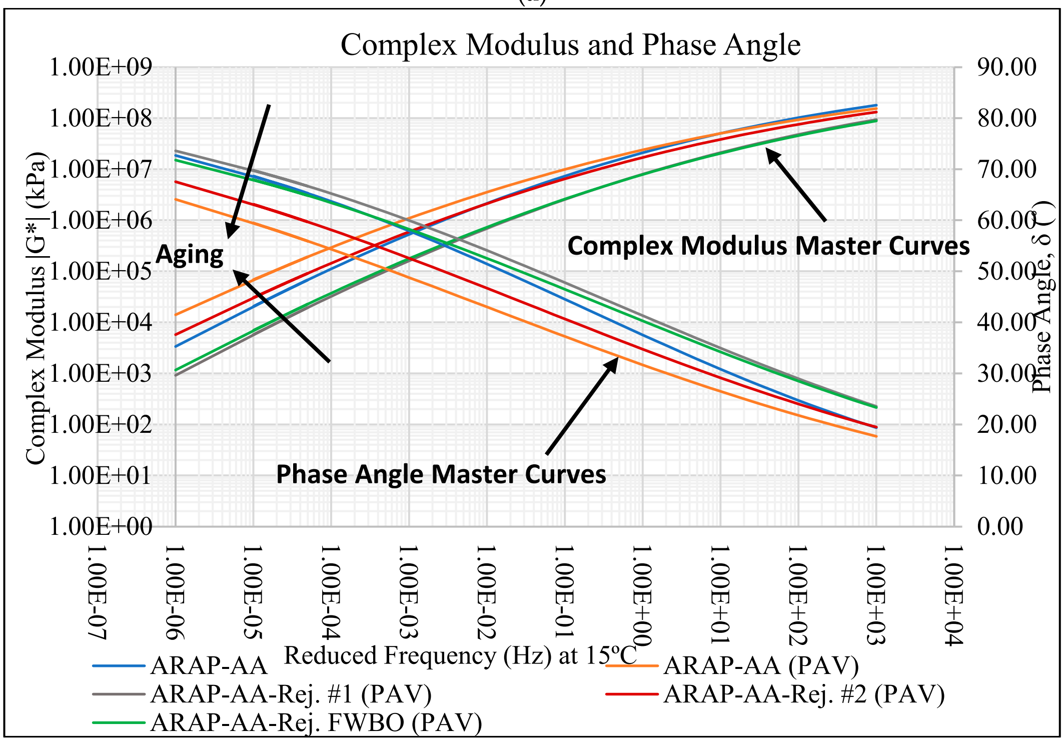
$$|G^*| = \frac{{G_g}^*}{\left[1 + \left(\frac{\omega_c}{\omega_r}\right)^k\right]^{\frac{m_e}{k}}} \quad (3)$$

where

$|G^*|$ = complex shear modulus (Pa);
${G_g}^*$ = glassy modulus (Pa);
ω_c = crossover frequency (rad/s);
ω_r = reduced frequency (rad/s);
m_e, k = fitting coefficients.

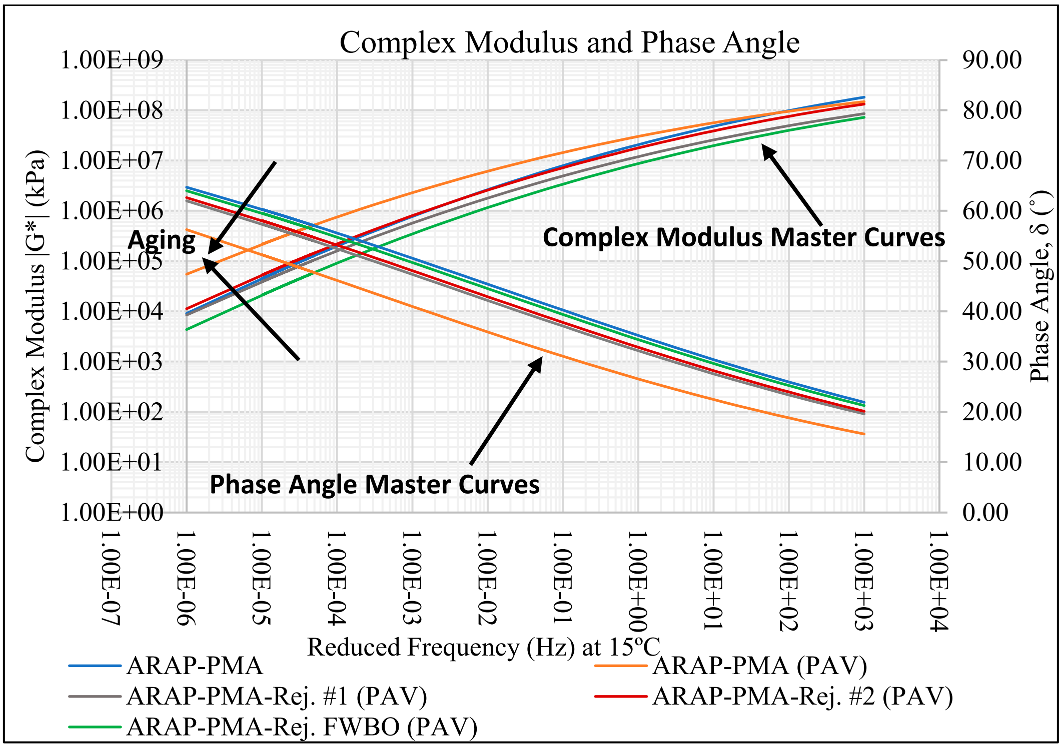


(**a**)

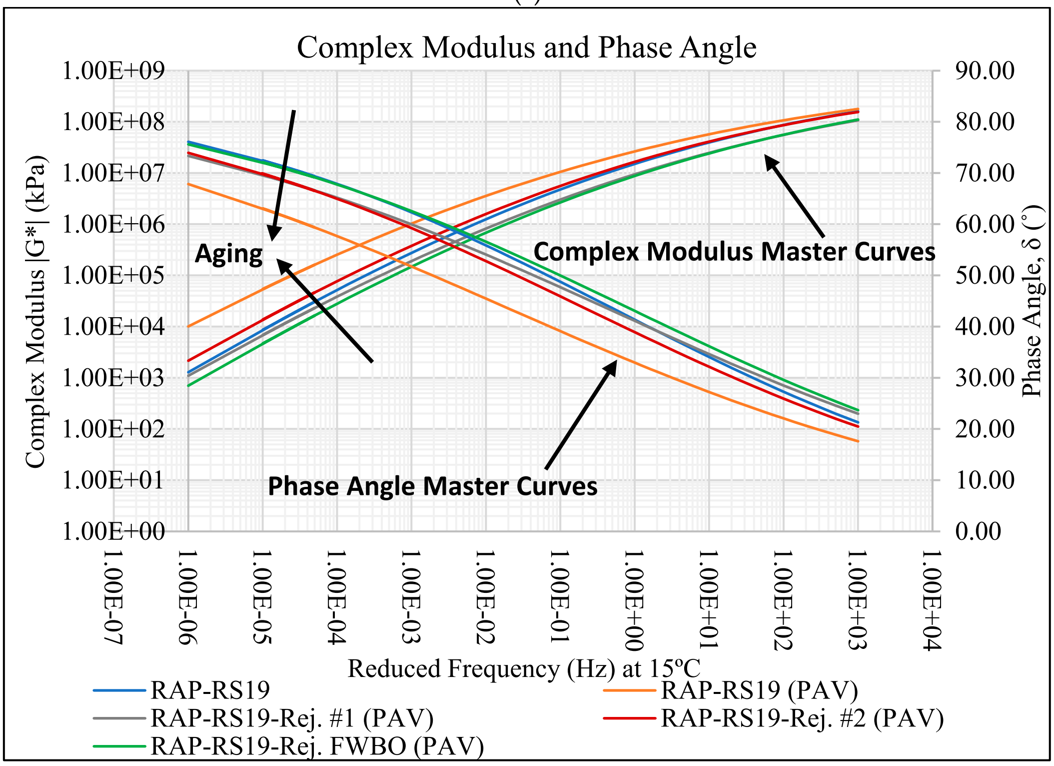


(**b**)

Figure 7. *Cont.*



(**c**)



(**d**)

Figure 7. Asphalt complex modulus and phase angle master curves for (**a**) ARAP-NC, (**b**) ARAP-AA, (**c**) ARAP-PMA, and (**d**) RAP-RS19. (Symbol "#" is rejuvenator number).

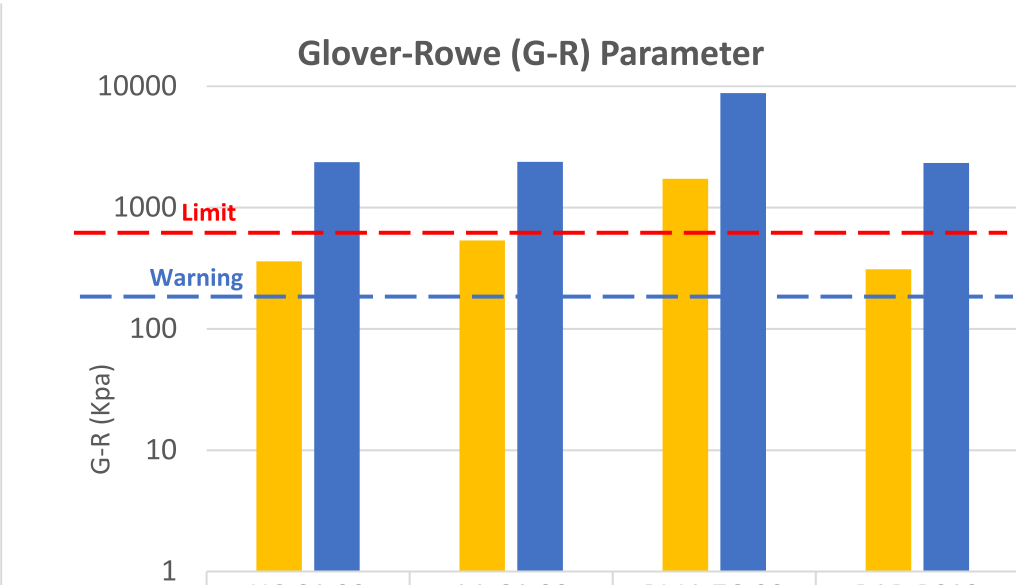
The complex modulus increases, and the phase angle decreases, when the investigated asphalt binders are subjected to the second round of PAV aging and when RAP is subjected to PAV aging. As expected, introducing the different rejuvenators to the aged asphalt binder reduces the stiffness. Following the FTIR results trends, the VBO rejuvenators have a more dominant effect than the PBO. Introducing rejuvenator #1 to the unmodified binder NC reduces its stiffness and increases its phase angle, accordingly enhancing its elastic component. As shown in Figure 7a, rejuvenator #1 enhances the elastic component to a limit higher than the binder after its first round of PAV aging. The PBO rejuvenator #2 performs the least with both investigated unmodified binders. It has an intermediate performance with the PMA binder and the real extracted RAP.

The proposed VBO performs efficiently with unmodified binder AA, PMA binders, and the extracted RAP compared to the two market rejuvenators. Furthermore, it is found that introducing the FWBO enhances the elastic component of the PAV-aged binder more effectively with the RAP, as shown in Figure 7b–d.

3.4. Grover–Rowe Parameter (G-R)

The G-R parameter is an aging indicator calculated from the DSR frequency sweep test results, used to evaluate the cracking resistance of an asphalt binder. Durability thresholds were translated into G-R = 180 kPa (corresponding to a 5 cm ductility and 0.0009 MPa/s for the initial DSR function) to indicate the onset of cracking (warning). G–R = 600 kPa (corresponding to 3 cm ductility, 0.003 MPa/s DSR function) was used to indicate extensive block cracking (limit) [37,39].

Figure 8 shows the results of the G-R parameter for the different investigated RAP at different aging levels. To begin with, all the RAP binders exceed the warning cracking limit (180 KPa). However, only the ARAP-PMA exceeds the cracking limit line (600 KPa). The reason behind that could be the polymerization of the asphalt binder, as the PMA binder is stiffer than the unmodified binder when aged. As expected, the different investigated RAP binders are more prone to fatigue cracking when exposed to a cycle of PAV aging, as suggested by the G-R parameter.



	NC 64-22	AA 64-22	PMA 76-22	RAP-RS19
RAP*	361	535	1728	310
RAP (PAV)	2369	2378	8776	2333

Figure 8. G-R parameter of the investigated RAP binders before and after subjection to PAV Aging. * RAP term includes both artificial RAP and field-extracted RAP.

Figure 9 depicts the results of the investigated PAV-aged rejuvenated and unrejuvenated RAP binders. The VBO rejuvenator #1 has the highest performance when introduced to ARAP-NC and ARAP-AA. It reduces the susceptibility to the intermediate temperature cracking by 80% and 90% for ARAP-NC and ARAP-AA binders, respectively, compared to their corresponding unrejuvenated PAV-aged RAP binders. On the contrary, when introduced to the different RAP binders, the PBO rejuvenator #2 has the lowest performance. Compared to the unrejuvenated PAV-aged RAP binders, it enhances the intermediate temperature performance by 50%, 48%, 76%, and 84% for the ARAP-NC, ARAP-AA, ARAP-PMA, and RAP-RS19, respectively.

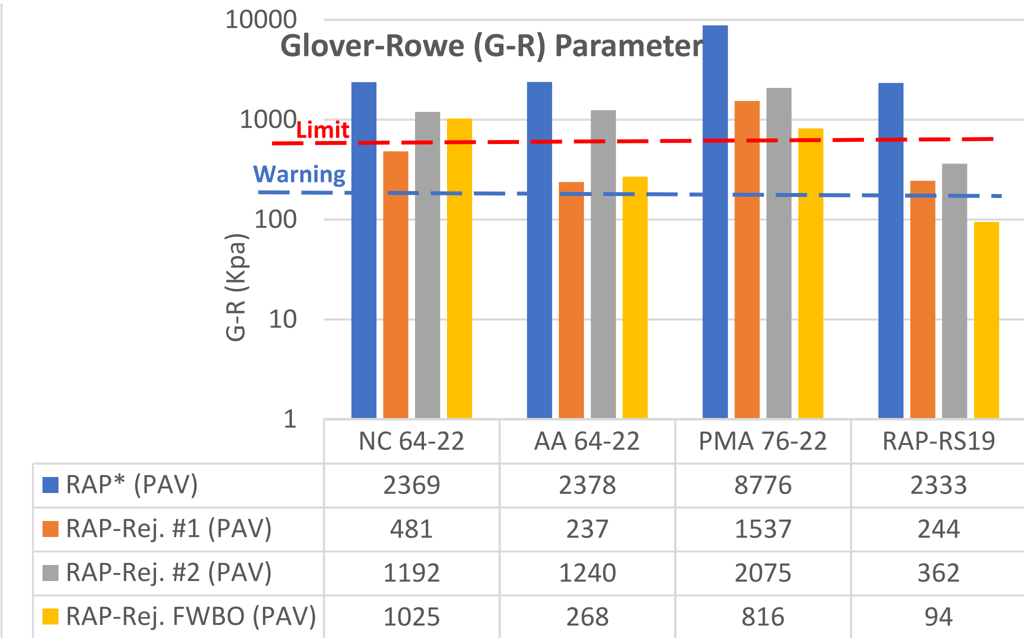


Figure 9. G-R parameter of the investigated PAV-aged rejuvenated and unrejuvenated RAP binders. * RAP term includes both artificial RAP and field-extracted RAP. (Symbol "#" is rejuvenator number).

The proposed VBO rejuvenator reduces the susceptibility to the intermediate temperature, cracking the most with the ARAP-PMA binder and RAP-RS19. Relative to unrejuvenated PAV-aged RAP, it reduces the G-R values by 91% and 96% for the ARAP-PMA and RAP-RS19, respectively.

Both VBO rejuvenators enhance the intermediate temperature performance to a limit lower than the extensive cracking limit (600 KPa) with the ARAP-AA binder. Moreover, the new FWBO, the VBO rejuvenator, reduces the RAP-RS19's susceptibility to the intermediate temperature cracking to a limit lower than the warning cracking limit (160 KPa). In general, the VBO rejuvenators show a higher intermediate temperature performance than the PBO rejuvenator.

3.5. Crossover Modulus and Frequency (G_c, ω_c)*

Table 3 shows the crossover modulus and frequency results for the different investigated unrejuvenated/rejuvenated RAP binders. Following the previously illustrated results, the crossover modulus and frequency of the different investigated asphalt RAP binders decrease when subjected to PAV aging.

In general, introducing the rejuvenators restores the crossover modulus and frequency to values higher than the unrejuvenated PAV-aged RAP binders. However, all the investigated rejuvenators did not restore the crossover modulus for the ARAP-NC. In contrast, the investigated rejuvenators could change the crossover frequency to a higher value than the unrejuvenated PAV-aged RAP binders. This suggests that the rejuvenator's synergetic behavior depends on the binder that it is mixed with.

Table 3. Crossover modulus and frequency values of the investigated unrejuvenated/rejuvenated RAP binders.

Asphalt Binder /RAP	Index	RAP *	RAP (PAV)	RAP-Rej. #1 (PAV)	RAP-Rej. #2 (PAV)	RAP-Rej. FWBO (PAV)
NC PG 64–22	G_c* (KPa)	9950	4777	4172	4073	2879
	ω_c (Hz)	0.651	0.018	0.138	0.040	0.031
AA PG 64–22	G_c* (KPa)	9485	3742	5283	4165	4708
	ω_c (Hz)	0.169	0.011	0.419	0.039	0.328
PMA PG 76–22	G_c* (KPa)	2899	977	977	1637	1049
	ω_c (Hz)	0.012	0.000	0.003	0.004	0.008
RAP-RS19	G_c* (KPa)	11,211	4972	6881	8131	8339
	ω_c (Hz)	0.530	0.019	0.513	0.211	0.900

* RAP term includes both artificial RAP and field-extracted RAP. Symbol "#" is rejuvenator number.

Following the previously discussed results, the VBO rejuvenators increase the aging resistance when introduced to the ARAP-AA. The VBO rejuvenator #1 has a higher viscoelasticity restoration when introduced to the ARAP-AA binder. On the other hand, the PBO rejuvenator #2 restores the viscoelastic components the most when introduced to the ARAP-PMA binder. As suggested by increasing the crossover modulus and frequency, the new VBO rejuvenator is very promising in rejuvenating the RAP-RS19 binder. When introduced to the ARAP-PMA and ARAP-AA binders, it also shows an acceptable rejuvenation performance.

3.6. Rejuvenators' Efficiency (RE) Analysis

The previously illustrated chemical and rheological aging indices will be utilized in this section for evaluating the investigated rejuvenators' efficiency. Equation (4) is used in evaluating the rejuvenator's chemical efficiency in reducing the carbonyl aging index.

$$\text{Rejuvenator Efficiency}_{\text{ICO}} = \frac{\text{ICO}_{\text{Unrejuevnated /RAP}}}{\text{ICO}_{\text{Rejuevnated Aged /RAP}}} \tag{4}$$

Intermediate and low-temperature rheological performance enhancement by rejuvenators is evaluated through Equations (5)–(8). According to these equations, the rejuvenator is considered efficient when it achieves an RE value higher than a value of 1.0.

$$\text{Rejuvenator Efficiency}_{\Delta T_c} = 1 + \frac{\Delta T_{\text{cUnrejuevnated Aged ARAP/RAP}} - \Delta T_{\text{cRejuevnated Aged ARAP/RAP}}}{\Delta T_{\text{cUnejuevnated Aged ARAP/RAP}}} \tag{5}$$

$$\text{Rejuvenator Efficiency}_{G-R} = \frac{\text{Log G} - \text{R}_{\text{Unrejuevnated Aged RAP}}}{\text{Log G} - \text{R}_{\text{Rejuevnated Aged binderRAP}}} \tag{6}$$

$$\text{Rejuvenator Efficiency}_{G_{C^*}} = \frac{\text{LogG}_c{}^{*}{}_{\text{Rejuevnated Aged RAP}}}{\text{LogG}_c{}^{*}{}_{\text{Uejuevnated Aged RAP}}} \tag{7}$$

$$\text{Rejuvenator Efficiency}_{\omega_c} = \frac{\omega_{\text{cRejuevnated Aged RAP}}}{\omega_{\text{cUnejuevnated Aged RAP}}} \tag{8}$$

Table 4 demonstrates the different investigated rejuvenators' efficiency. For the ARAP-NC binder, both rejuvenators #1 and #2 are chemically efficient, reducing the carbonyl index. They also have higher efficiency with improving the low-temperature thermal cracking resistance. Moreover, they improve the intermediate-temperature performance as they reduce the G-R parameter. However, both rejuvenators are found to perform as softeners, since they increase the crossover frequency and do not increase the crossover modulus. The newly proposed VBO rejuvenator follows the same trend. However, it fails to reduce the carbonyl index when introduced to the ARAP-NC binder.

Table 4. Different investigated rejuvenators' efficiency summary.

Rejuvenator Efficiency	Asphalt Binder Type	Rejuvenators' Rheological Efficiency				
		ICO	ΔT_c	G-R	ω_c	G_c*
RE#1	ARAP-NC	1.069	1.449	1.258	7.672	0.984
RE#2		1.261	1.329	1.097	2.224	0.981
RE#FWBO		0.993	1.135	1.121	1.746	0.940
RE#1	ARAP-AA	1.104	1.851	1.421	37.688	1.042
RE#2		0.897	1.003	1.091	3.475	1.013
RE#FWBO		1.461	2.086	1.390	29.512	1.028
RE#1	ARAP-PMA	1.141	1.665	1.237	17.070	1.000
RE#2		1.181	2.222	1.189	23.817	1.075
RE#FWBO		1.143	1.700	1.354	48.956	1.010
RE#1	RAP-RS19	0.979	1.580	1.411	26.310	1.038
RE#2		0.974	1.095	1.316	10.821	1.058
RE#FWBO		1.014	1.580	1.705	46.214	1.061

Note: red background: RE < 1.0, Bad rejuvenator efficiency; orange background: RE = 1.0, Fair rejuvenator efficiency; green background: RE > 1.0, Good rejuvenator efficiency.

For the ARAP-AA binder, both VBO rejuvenators improve the chemical and rheological performance when introduced to the ARAP binder. The PBO rejuvenator #2 is found to perform fairly well with the ARAP-AA binder. However, it did not reduce the carbonyl index when introduced to the ARAP-AA binder. It has lower rejuvenation efficiency when compared to the VBO rejuvenators.

For the ARAP-PMA binder, the PBO rejuvenator #2 has superior rejuvenation efficiency compared to the VBO rejuvenators. In addition, it reduces the oxidation levels of ICO the most compared to the VBO rejuvenators. Moreover, it enhances the low-temperature thermal cracking resistance the most. However, the proposed VBO has the dominant improvement with the intermediate temperature performance when introduced to the ARAP-PMA binder.

For the field-extracted RAP-RS19 binder, the proposed FWBO rejuvenator has the most significant rejuvenation efficiency. Moreover, it manages to improve both the chemical and rheological performance of the RAP. On the other hand, both obtained market rejuvenators #1 and #2 cannot reduce the carbonyl index. Compared to the VBO rejuvenators, the PBO rejuvenator has a lower rejuvenation efficiency when introduced to the RAP-RS19.

4. Conclusions

In this research, three asphalt binder asphalt rejuvenators were introduced to three aged asphalt binders and RAP. Two market rejuvenators were evaluated side by side with a new FWBO rejuvenator. The two market rejuvenators are VBO and PBO. The new FWBO rejuvenator is a VBO rejuvenator. The binders used in this study are unmodified and PMA asphalt binders. Investigated binders underwent the RTFO cycle and the first PAV cycle before the testing samples were taken. Then, the rejuvenators were introduced to the different asphalt binders.

Further, each sample underwent the second PAV cycle. The main goal of this research was to evaluate the rejuvenators' efficiency on asphalt binder aging. The efficiency of rejuvenators was evaluated through leading aging indicators, such as FTIR, ΔT_c, G-R parameter, ω_c, and G_c*. This section summarizes an extensive testing program that included many samples and testing.

1. Oxidation (FTIR)
    - When introduced to the different investigated ARAP/RAP, the proposed FWBO is the only rejuvenator showing a consistent reduction in the ICO aging index;

- Compared to the PBO rejuvenator, the VBO rejuvenators are more efficient in reducing the oxidation levels when introduced to the unmodified binder and RAP. On the contrary, the PBO rejuvenator performs more efficiently when introduced to the PMA binder;
- The new VBO rejuvenator has superior efficiency in lowering the oxidation levels of the extracted RAP.

2. Low-Temperature Durability (ΔT_c)
    - The VBO rejuvenators significantly enhance the low-temperature thermal cracking resistance of all samples. On the other hand, the PBO rejuvenator is especially synergetic with the PMA binder, and its enhancement of the thermal cracking resistance is more pronounced;
    - There is a strong coloration between the chemical oxidation index, ICO, and the low-temperature durability index, ΔT_c;
3. Intermediate temperature Durability (G-R)
    - Compared to the PBO rejuvenator, the VBO rejuvenators have higher efficiency in enhancing the intermediate temperature cracking resistance when introduced to all investigated aged binders and RAP.
4. Rejuvenator vs. Softener (crossover frequency and modulus (G_c*), (ω_c))
    - All the investigated rejuvenators are found to perform as softeners when introduced to the unmodified binder NC, as they increase the crossover frequency and do not increase the crossover modulus;
    - The PBO rejuvenator has higher rejuvenation efficiency in restoring both the viscous and elastic behavior when introduced to the PMA binder. On the contrary, the VBO rejuvenators are found to have a higher rejuvenation efficiency when introduced to the unmodified binders.
    - When introduced to the extracted RAP, the proposed FWBO increases the crossover modulus and frequency, indicating high rejuvenation efficiency.
5. Based on the results, FWBO acquires the merit of being an eco-friendly rejuvenator in the asphalt pavement industry compared to the two market-available rejuvenators.
6. These significant findings are currently being complemented with mixture testing.
7. A complete life cycle assessment (LCA) is necessary to quantify the reduction in energy and waste due to utilizing the proposed FWBO.

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