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## A Threshold to Utilize Guayule Resin as a New Binder in Flexible Pavement Industry

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### ABSTRACT

This study denotes a threshold to utilize Guayule Resin as a new bio-based binder or asphalt cement (AC) proportion, both with/out crumb rubber modifier (CRM), regarding solutions for environment, cost, and sustainability aspects. Via this research, 15 designated binders will be presented, all of them were tested as unaged and RTFO, whereas eight binders proceeded with PAV. Superpave was utilized to study their physical properties. Viscosity was examined upon some guayule-based binders with/out CRM, getting 114cP (at 135°C) for BGR, increased to about 3 times with 10%CRM (by weight of liquid binder) and about 7-8 times with 20%CRM. Comparisons among binders will be depicted regarding six categories (Neat AC, Neat BGR, AC+BGR, BGR+CRM, AC+CRM, and AC+BGR+CRM). This study initiates a perspective to judge designated binders and get benefit upon performance required. That's why the study reported outcomes regarding the effectiveness of the binder's stiffness, elasticity and performance. Results showed 20%CRM raised BGR performance by about one grade, compared to 5%CRM for AC to boost the same one grade. On another side, RTFOT showed a high mass loss for guayule-based binders, reaching 8.5% for unconditioned BGR, but about 5% by a heat-treatment process. The low-temperature grade was presented for all guayule-based binders indicating a -10 grade.

**Keywords**-Asphalt-rubber, Bio-binder, Bridgestone, CRM, Pavement

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## I. INTRODUCTION

### A. Overview

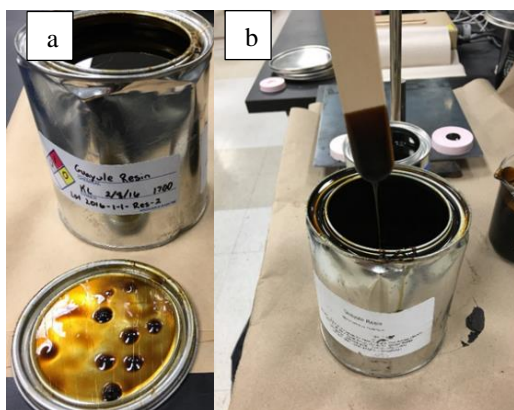
Guayule rubber is in its way to being commercial in competition with Hevea Natural Rubber (NR), the current commercial source of natural rubber [1]. Guayule Rubber has the potential to be a U.S. domestic source of natural rubber, mainly, in the tire industry [1]. Nowadays, the researchers, in this discipline, are focusing on how to reduce the resin percentage in the extracted guayule rubber since it is not desirable in the guayule rubber extraction process [2]. That's because low-molecular-weight components work as a plasticizer (i.e., resin components degrade the desired physical properties of rubber that could increase the amounts of resin extracted [2]. However, this last will have low-molecular-weight components since the rubberized portion encompassed will be significantly diminished. The only obstruction against utilizing Guayule Rubber, via previous hundred years, is not economically affordable in its production cycle. The solution to open the door towards this valuable source of natural rubber is yielding high-value by-products including resin to be commercially attractive [3]. As reported in a publication by Schloman, et al.

"Seasonal Effects on Guayule Resin Composition," Guayule by-products - such as resin, bagasse, wax, seed, and leaves - have the potential to involve about 25% to 50% saving in the guayule rubber production process [3].

As a result, guayule resin utilization in the flexible pavement industry could be an opportunity to gain two aspects: (1) get a benefit from a by-product (cheap material) in such a massive industry like flexible pavement, in addition to (2) equilibrate the production cost of guayule extractables [3].

### B. Guayule Resin Composition

Guayule (whole shrub) resin is composed of monoterpenes, triterpenes, sesquiterpene esters (guayulins A & B) and triglycerides [4]. Chemical structure of guayule resin mainly contains hydrocarbons, which is a viscous organic substance derived from guayule plant as a by-product extractable besides the guayule natural rubber [4]. For instance, the chemical structure of guayulin A, guayulin B and argentatin A are C<sub>24</sub>H<sub>30</sub>O<sub>2</sub>, C<sub>23</sub>H<sub>30</sub>O<sub>3</sub>, and C<sub>30</sub>H<sub>48</sub>O<sub>4</sub> respectively [4]. That indicates containing functional groups in the chemical composition of guayule resin (Fig. 1) simil-



**Figure 1.** Guayule resin material: (a) at room temperature; (b) at 135°C

ar to asphalt binder. Also, asphalt binder, crumb rubber modifier (CRM) and guayule resin are mainly composed of hydrocarbons. That's why the potential of harmony via their interaction is there.

### C. Bridgestone Guayule Resin

Bridgestone Guayule Resin (BGR) denotes the resin separated from the natural rubber extraction process by Bridgestone Corp for its new tire prototype [5]. Bridgestone Corp claimed "In 2015, the Bridgestone Group produced the first tire made from guayule-derived natural rubber. This was an impressive step toward "expansion and diversification of renewable resources [5]." The remaining resin represents a leftover material which is not desired in the rubber extracted for their new tire production. Nevertheless, it could be a potential binder, instead of/associated with asphalt binder, also, adding some enhancers such as CRM could improve the performance behavior as will be elaborated by testing. This is just a threshold as an attempt to utilize guayule resin as a new flexible bio-based binder.

### D. Problem Statement

Because of the gradual increase of asphalt cement (AC) cost [6], environmental problems (e.g., emissions of volatiles, particularly the fume associated to the construction process, heating to relatively higher temperatures that exploits massive energy, as well as unfriendly environmentally for pavement workers as its fumes mixed with the surrounding atmosphere [7, 8], in addition to the sense of sustainability, seeking for new tracks to overcome all these aspects became a must [7]. That's why, getting the advantage of the guayule resin, as a by-product material extracted with the significant extractable "guayule rubber," could be an acceptable approach to think about. Utilizing a bio-based material, instead of a petroleum-based material, has a potential to be implemented as an approach to get rid of the evolutionary petroleum extractables costs in addition to the diminishment of petroleum resources

universally [7, 9]. Research, in 2009, expected that there would not be fossil fuel remaining after 2042 other than coal [10], i.e., no crude oil or gas will be there shortly. Thus no AC will be there since it is a by-product extracted from crude oil. Hence, thinking in alternative binders or binder additives could be a step to balance this diminishment. Guayule resin could reduce the overall flexible pavement costs, especially as a renewable and domestic natural material that grows in the vast arid southwestern zones of the U.S, [1, 5, 11]. FPM, Flexible Pavement Mixture (FPM), is mentioned instead of Asphalt Pavement Mixture (APM), indicating the potential for the flexible pavement to imply materials that can be used instead of/associated with petroleum-based materials as an attempt to involve a spacious terminology of all researched materials that can be employed in the flexible pavement industry [6].

### E. Objective

Via this study, we are going to open the door to utilize guayule resin, as an(associated) binder in the flexible pavement industry as one of the attempts to establish a terminology transition of asphalt pavement as we are confronting an inevitable depletion of crude oil [6]. Using the idea of flexible pavement mixture (FPM) indicates a potential of utilizing flexible binders other than asphalt. This study will look forward to investigating guayule resin as a new binder instead of AC at specific grades.

On the other hand, regarding its high-temperature grade applicability, there is a potential to manipulate the guayule resin, as a virgin binder, at high-temperature application by adding CRM to elevate its grade at high-level temperature. It could be justified as guayule resin is extracted from guayule rubber. As a result, the excellent indication of the overall enhancement here is the harmony (chemistry) between guayule resin and rubber in the first place (both of them mainly hydrocarbons). It is something like extracting guayule resin from guayule rubber (guayule natural rubber as a valuable source in the tire production [1]); restoring some of the recycled rubber (CRM) to the resin to enhance its required characteristics.

Furthermore, for more applicability, a discussion of how guayule resin has a potential to be added to asphalt binder with/out CRM will be presented. This will be studied upon 15 designated binders to render an initial stage for guayule-based binders (mentioned as "no AC included"), compared to asphalt-based binders (mentioned as "any binder including AC").

## II. EXPERIMENTAL PLAN

This work was built upon 15 designated binders, divided into six subsets (Neat BGR, BGR+CRM, AC+BGR+CRM, Neat AC, AC+BGR

**Table 1.** Designated Binders and Implemented Test Conditioning

Base	Subset	Designated Binder <sup>+</sup>	Binder Label	Proportions			Interaction Time, hr	Interaction Speed, rpm (Hz)	Interaction Temp., C	Test Conditioning
				AC	BGR	CRM <sup>++</sup>				
Guayule-Based Binders	Neat BGR	Neat BGR(Unconditioned) <sup>+++</sup>	B(U)	—	—	—	—	—	—	OB, RTFO, PAV
		Neat BGR-3000-190(4hr)	B(4)	—	—	—	4	3000 (50)	190	OB, RTFO, PAV
		Neat BGR-3000-190(6hr)	B(6)	—	—	—	6	3000 (50)	190	OB, RTFO, PAV
	BGR+CRM	BGR-CRM(10:2)-3000-160(4hr)	B+C(160-4)	—	10	2	4	3000 (50)	160	OB, RTFO, PAV
		BGR-CRM(10:1)-3000* <sup>++++</sup>	B+C*	—	10	1	6	3000 (50)	160, 190	OB, RTFO, PAV
		BGR-CRM(10:2)-3000-190(4hr)	B+C(190-4)	—	10	2	4	3000 (50)	190	OB, RTFO, PAV
Asphalt-Based Binders	AC+BGR+CRM	BGR-CRM(10:1)-3000-190(6hr)	B+C(190-6)	—	10	1	6	3000 (50)	190	OB, RTFO, PAV
		AC-BGR-CRM(5:5:2)-3000-190(4hr)	A+B+C(4)	5	5	2	4	3000 (50)	190	OB, RTFO
		AC-BGR-CRM(5:5:1)-3000-190(6hr)	A+B+C(6)	5	5	1	6	3000 (50)	190	OB, RTFO
		AC-BGR-CRM(7.5:2.5:1)-3000-190(6hr)	7.5A	7.5	2.5	1	6	3000 (50)	190	OB, RTFO
	Neat AC	Neat AC <sup>++++</sup>	AC	—	—	—	—	—	—	OB, RTFO, PAV
		AC-BGR(5:5)-3000-190(2hr)	A+B	5	5	—	2	3000 (50)	190	OB, RTFO
		AC-CRM(10:2)-3000-190(4hr)	A+C(4)	10	—	2	4	3000 (50)	190	OB, RTFO
	AC+CRM	AC-CRM(10:1)-3000-190(6hr)	A+C(6)	10	—	1	6	3000 (50)	190	OB, RTFO

<sup>+</sup> For assigned binders preparation, except the unconditioned neat BGR and AC, high shear mixer “HSM-100LCI-T” [as recommended by previous researchers in the asphalt-rubber (AR) mixing [12-17] and also for bio-based binders [30]] was utilized with a temperature control set-up utilizing a heating mantle “Glas-Col - 100C M112” with a benchtop controller “Digi-Sense – TC9100.”

<sup>++</sup> Source: MoDOT from “Liberty Tire Recycling.” Crumb rubber modifier (CRM) was supplied in various distribution. However, CRM 30-40 (i.e., passing sieve#30 and remaining on sieve#40) was designated, since CRM grade 30-40 is one of the recommended sizes by the US standard system [18].

Any stated 10% or 20%CRM means the percentage of CRM concerning the weight of AC and/or BGR

<sup>+++</sup> Source: Bridgestone Americas Center for Research and Technology (BART), 1659 South Main Street Akron, OH 44301 United States, Lot: 2016-1-1-Res-2.

<sup>++++</sup> 4hr interaction for BGR-CRM(10:2)-3000-160 / BGR-CRM(10:2)-3000-190, then 2hr interaction after diluting the mix to include 10%CRM with raising the interaction temperature from 160°C to 190°C.

<sup>+++++</sup> Source: Conoco Phillips terminal in Granite City, Illinois; Superpave PG52-28 (main physical and chemical properties, determined at 20°C and 760 mm-Hg (1atm): black viscous appearance, liquid physical form, >1 vapor density, <1 mm-Hg vapor pressure, > 482°C (900°F) boiling point, 1.005 specific gravity, 8.33 lb/gal bulk density, <1 evaporation rate and 232°C (450°F) flash point); selected for this research work since it is comparable to the neat BGR utilized graded “PG52” as its elevated-temperature grade as will be discussed.

- 2mm gap for testing all “CRM-added” binders considering the rubber particle effect as recommended by previous researchers [19-21].

and AC+CRM) as reported in Table 1. Each subset consisted of one or more binders. Furthermore, each binder was labeled to facilitate its reading hereafter (e.g., Neat BGR(Unconditioned) was labeled B(U)).

All Superpave performance testing was carried out in the asphalt lab, Missouri SS&T&T, regarding all designated binders according to AASHTO/ASTM specifications as following. All identified binders were yielded to Original Binder (OB) and Rolling Thin Film Oven (RTFO) testing. However, eight, mainly guayule-based, binders involved) were exposed to PAV testing as reported in Table 1. As a result, more investigation could be carried out in the future to saturate more testing aspects. Dynamic viscosity was studied in various levels for guayule-based binders (Neat BGR and BGR-CRM binders) as will be explained in Section (4). Likewise, Dynamic Shear Rheometer (DSR) tests

were carried out for the designated binders including the grade determination and master curves for selected binders, unaged for all 15 identified binders and proceeding with aging for the seven guayule-based binders – Section (5). Also, the mass change upon RTFOT for the seven guayule-based binders was studied – Section (6), then Pressure Aging Vessel (PAV) – Section (7). Furthermore, BBR testing was done for various aged BGR-based binders, Section (8).

### III. DESIGNATED-BINDER PREPARATION

The following approaches to materials preparation were selected as a threshold. However, in upcoming studies, we will investigate more approaches to discuss the varieties of materials' behaviors.

### A. Guayule-Based Binders

The “BGR-CRM(10:2)-3000-190(4hr)” and “BGR-CRM(10:1)-3000-190(6hr)” designated binders were prepared as follows:

1. A quart “metal” can of 400g BGR was weighed on the scale, then heated on the mantle at 160°C for 45 min to be a fluid and partially get rid of some moisture inside; the temperature was raised to 190°C. The can’s lid was used to minimize aging, comprising one hole for the thermocouple to control the mix temperature (vertically located one inch above the can’s bottom), in addition to two other holes for ventilation during the heating process.
2. CRM (30-40) was oven dried for 6hr at 110°C, for all CRM used in materials’ preparation, to assure no moisture inside.
3. 80g oven-dried CRM (30-40) was added to the BGR amount to create a BGR with 20% CRM blend.
4. The mixing system was utilized to come up with a mix of BGR & 20%CRM at interaction speed of 3000rpm (50Hz), interaction temperature of 190°C and interaction time of 240 minutes (named “BGR-CRM(10:2)-3000-190(4hr)”).
5. After that, the test proceeded for extra 2hr, but after diluting the mix to comprise 10% of CRM instead of 20%, applying the same interaction speed and temperature, obtaining BGR-CRM(10:1)-3000-190(6hr).

Regarding the moisture aspect, it exists in regular sources of guayule resin as received from the manufacturer. It was predicted, as it is evident for bio-binders by literature [22], as well as observed during the heating process which was associated with foaming around the water boiling point. Nevertheless, it could be annealed (heat-treated) when the material is exposed to temperatures higher than the water boiling point (100°C) as applied via this research work, not only that but also with mixing (as will be discussed via Section 6). Nevertheless, chemical analysis is needed to be in-depth investigated in the future to optimize the approach of water removal.

Same Procedure was applied to acquire other two designated binders (BGR-CRM(10:2)-3000-160(4hr) & BGR-CRM(10:1)-3000\*). The only difference was the kick-off temperature which was 160°C for the first 4hr, then 190°C for extra 2hr.

Likewise, the same procedure was designated to acquire Neat BGR-3000-190(4hr & 6hr). The difference was heating and mixing for only unmodified BGR (i.e., without additives), named “heat-treatment process.”

### B. Asphalt-Based Binders

Procedure to designate AC-BGR-CRM(5:5:2)-3000-190(4hr), AC-BGR-CRM(7.5:2.5:

1)-3000-190(6hr) and AC-BGR-CRM(2.5:7.5:1)-3000-190(6hr) is as follows:

1. 300g AC (PG52-28) was poured in an empty quart can; heated on the mantle until reaching a temperature of 190°C (can’s lid used to minimize aging).
2. 120g oven-dried CRM was added to AC, mixing for 2hr at 3000rpm and 190°C.
3. After 2hr interaction of AC & CRM, 300g of BGR was added to the blend; all mix proceeded for extra 2hr to complete 4hr interaction as the overall interaction time, getting AC-BGR-CRM(5:5:2)-3000-190(4hr).
4. The test proceeded for extra 2hr for two times (one after controlling the outcome mix to comprise AC+BGR+CRM (proportions: 7.5:2.5:1 respectively), the other one to incorporate proportions of 2.5:7.5:1 respectively, applying the same interaction speed and temperature.

Procedure to designate AC-CRM(10:2)-3000-190(4hr), AC-CRM(10:1)-3000-190(6hr) and AC-BGR-CRM(5:5:1)-3000-190(6hr) is as follows:

1. 600g of AC was poured in an empty quart can; heated on the mantle until reaching a temperature of 190°C (can’s lid used to minimize aging).
2. 120g oven-dried CRM was added to AC, mixing for 2hr at 3000rpm and 190°C.
3. After 2hr interaction between AC & CRM, 240g of AC+20%CRM was taken out to be added to 200g neat BGR in a new quart can (to be mixed after that as AC50-BGR50-CRM10).
4. The remaining 480g of (AC+20%CRM) was proceeded for mixing for extra 2hr (to achieve 4hr mix), acquiring AC-CRM(10:2)-3000-190(4hr).
5. After the 4hr interaction, the mix (AC+20%CRM) was diluted by 200g AC to accomplish AC, and 10%CRM and the interaction proceeded for extra 2hr (reaching 6hr mix) obtaining AC-CRM(10:1)-3000-190(6hr).
6. On the other hand, the different blend “AC50-BGR50-CRM10” was mixed for extra 4hr (i.e., to reach out 6hr mix), getting AC-BGR-CRM(5:5:1)-3000-190(6hr).

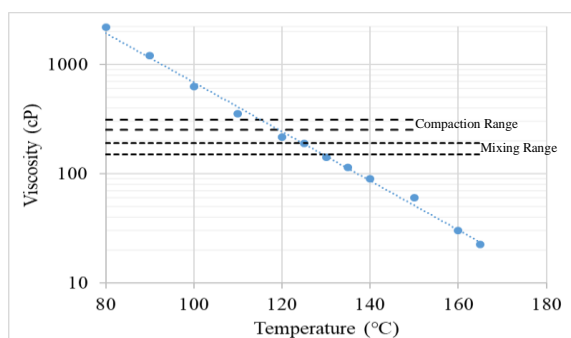
Also, a mix of neat AC and neat BGR was obtained by adding 300g of AC and 300g of BGR in a quart can with applying the same consistency as mentioned above. This mix proceeded only for 2hr, getting AC-BGR(5:5)-3000-190(2hr).

## IV. BGR-BASED BINDERS VISCOSITY TESTING

Viscosity testing was carried out for the virgin BGR, utilizing Brookfield/Rotational Viscometer “DV-III” referring to AASHTO T316 [23]. Fig. 2 shows the relationship between viscosity and temperature in a wide range from 80°C to 165°C.

Likewise, 135°C was considered corresponding to the Superpave standard test of asphalt binder and 165°C as a high temperature, close to RTFOT temperature. This could help to initially recognize the construction temperatures (i.e., mixing and compaction temperatures, [24]) of BGR in case of succeeding as a new flexible binder. Least Square Method was used to identify the best fit of this relationship.

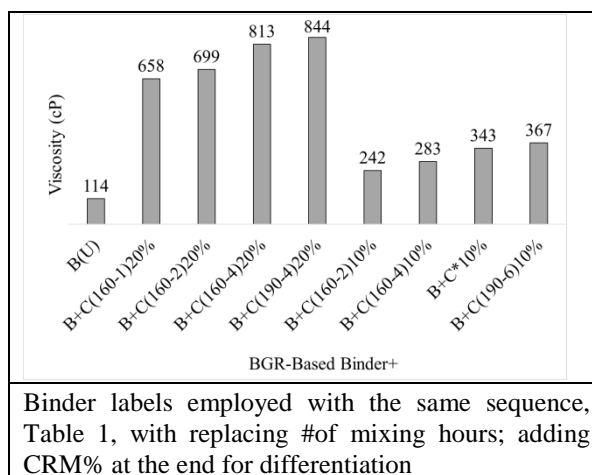
Upon ASTM specs, the mixing temperature range giving a viscosity of  $170 \pm 20$  cP is from 124.5°C to 129°C. Furthermore, the compaction temperature range giving a viscosity of  $250 \pm 30$  cP is from 116°C to 119.5°C [24, 25]. This is an indication that Guayule Resin has a potential to save processing energy with lowering the mixing and compaction temperatures required, compared to regular asphalt binders, even as an additive will reduce the overall processing temperatures (i.e., energy).



**Figure 2.** Neat BGR(Unconditioned) viscosity-temperature relationship & mixing and compaction ranges

Likewise, Brookfield “DV-III” Viscometer was utilized for testing the dynamic viscosity of guayule-based binders, plus extra arbitrary samples extracted during the mixing process to define the evolution of the binder viscosity with time. Standard dynamic viscosity test (at 135°C) was implemented for the selected binders. As observed in Fig. 3, the more the interaction temperature is, the more the viscosity value is yielded. This last could be justified as the gradual increase of CRM particles dissolving raises the overall viscosity of the blend with the normal precipitation of remaining CRM- particle on the RV vessel bottom via the equilibrium time. Moreover, compared to the neat BGR viscosity, CRM additive is significantly effective on increasing the blend viscosity. Regarding partial CRM particles dissolution, the more the interaction parameters are severe (i.e., longer time; higher temperature; higher shear speed), the more the CRM particles are dissolving [17].

Also, ConocoPhillips, Superpave PG52-28 viscosity was measured 309cP, used as a reference to guayule-based binders. It is almost comparable to 10%CRM-added guayule-based binders.



Binder labels employed with the same sequence, Table 1, with replacing #of mixing hours; adding CRM% at the end for differentiation

**Figure 3.** Dynamic viscosity of Guayule-based binders at 135°C

## V. DSR RESULTS & DISCUSSION

All designated binders (unaged & RTFO binders, RTFO process will be discussed later) were tested utilizing the Anton-Paar (Model: MCR302) DSR, employing the grade determination mode upon AASHTO T315, [26]. All tests were kicked off from 46°C until reaching the failure temperature concerning the Superpave minimum rutting parameter ( $G^*/\sin\delta$ ), 1.0kPa (OB) and 2.2kPa (RTFO). The applications for unaged binders aim to show the new materials' quality and the applications for RTFO-aged binder aim to verify the grade and show the aging susceptibility. Furthermore, bio-based binders were tested in PAV conditioning to simulate the long-term aging to be compared with the control (AC: PG52-28). Table 2 depicts all results achieved utilizing the grade determination mode to get the phase angle, the complex shear modulus and the rutting parameter ( $G^*/\sin\delta$ ). Likewise,  $G^*/\sin\delta$  master curves were studied, upon the frequency sweep mode for selected unaged binders to compare their behavior with frequency change.

### A. Complex Modulus Susceptibility

Fig. 4 demonstrates the susceptibility, at 52°C, (OB vs. RTFO) of the complex modulus ( $G^*$ ). Guayule-based binders (Neat BGR, BGR+CRM, AC+BGR+CRM) resulted in high susceptibility. For instance, RTFO Neat BGR (Unconditioned) reached 3.2kPa, compared to 0.4kPa regarding OB (increased about eight times, moisture loss may be a significant factor). However, the regular asphalt reaches 2.2 times. For instance, Neat asphalt (PG52) achieved 2.1kPa (OB) versus 4.6kPa (RTFO) which increased 2.2 times. This may reflect the high change in properties when guayule-based binders are exposed to aging (this point needs more chemical investigation in the future). Even though RTFO B+C(160-4) with 9.5kPa has a relatively high value among all guayule-

based binders; it was sharply degraded to end up with PG58 as recorded in Table 2 and Fig. 4.

### B. Phase Angle Susceptibility

Fig. 5 discusses the phase angle susceptibility (OB vs. RTFO) of all designated binders at 52°C which represents the AC and BGR grades except the untreated one. It's evident that there is almost no change in phase angle for the neat BGR (unconditioned & conditioned), about 87°. In case of BGR-Rubber (BR) binders, CRM has an obvious influence of the elastic behavior particularly the RTFO-aged blends. At 160°C interaction temperature, the more the CRM was included, the less the Phase angle was measured. For 10%CRM and 20%CRM (by weight of BGR), the phase angle was decreased by about 5% and 10% respectively. At 190°C interaction temperature, the phase angle was increased by about 5% for 20%CRM and no-to-little change regarding 10%CRM. For all asphalt-based binders (neat AC, AC+BGR, AC+CRM, and AC+BGR+CRM) the elastic behavior rose from OB to RTFO-conditioned binders. Furthermore, the most enhanced elastic behavior regarding OB and RTFO binders was associated with AC+20%CRM (A+C(4)), 59° for OB and 54° for RTFO.

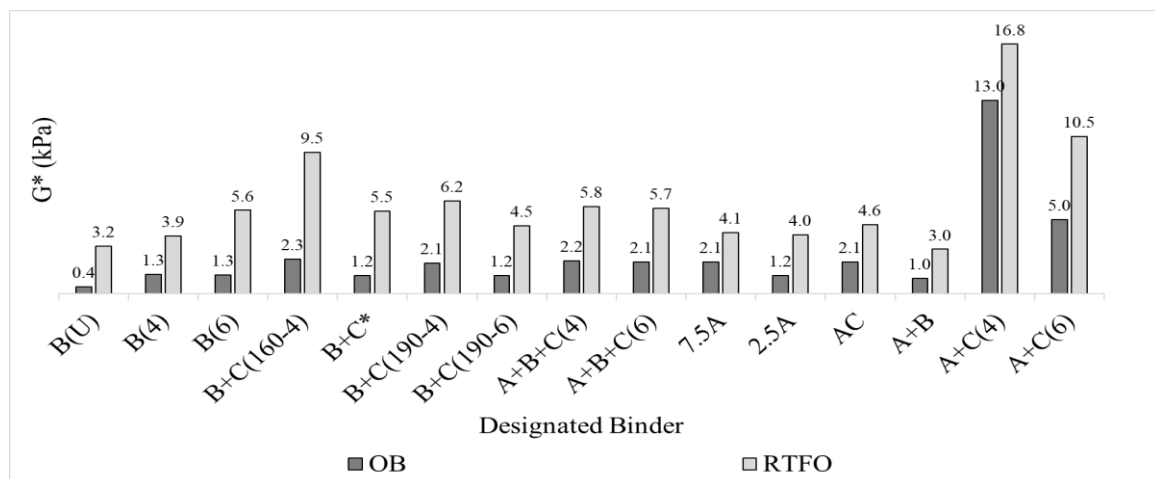
### C. Elevated Temperature Grade

According to the elevated-temperature grade, no significant observation was detected regarding OB compared to RTFO as denoted in Table 2. In other words, the majority of binders involved the same grade experienced by either OB or RTFO except for four binders [B(U), B+C\*, A+B+C(6) and 7.5A] with a variation of only one grade between OB and RTFO. This may reflect the consistency of the binder grade when comparing OB to RTFO-conditioned binders.

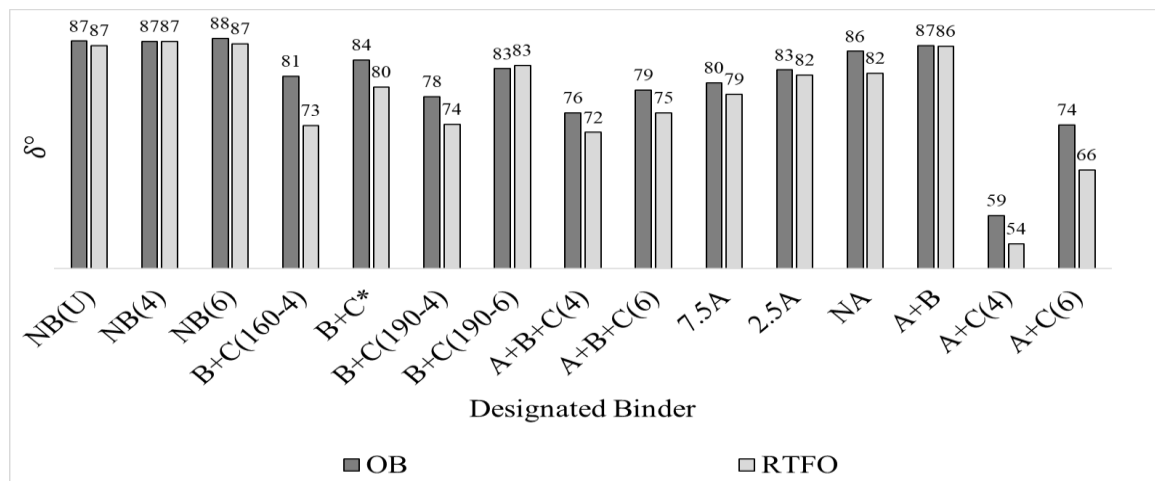
In Table (2) and Fig. 6, Subset (1) as described in Table 1, all evidence show that the neat BGR utilized, regarding the available source, indicate a grade of BG52. This is evident by the neat BGR DSR testing [OB: NB(4) & NB(6); RTFO binders: NB(U), NB(4) & NB(6)]. However, the neat BGR, OB, was the only one which resulted in PG46. That could be analyzed by the influence of moisture involved via the tank binder "B(U)." The heat treatment process to Neat BGR disposed of the moisture inside [22]. Also, RTFO process for Neat BGR (Unconditioned) yielded PG52 instead of PG46 for the BGR tank material. As a result, there was no significant difference among the varieties of Neat BGRs in all cases when kicking the moisture out either by heat-treatment process or aging by RTFO. Through Subset (2), even though, adding 10% to BGR [B+C\* & B+C(190-6)] yielded the same grade as neat BGR; 20%CRM [B+C(160-4) & B+C(190-4)] achieved PG58. Over Subset (3), the blend of

**Table 2.** Elevated-Temperature Parameters for Designated Binders (OB vs. RTFO) and Final Elevated-Temperature Grade

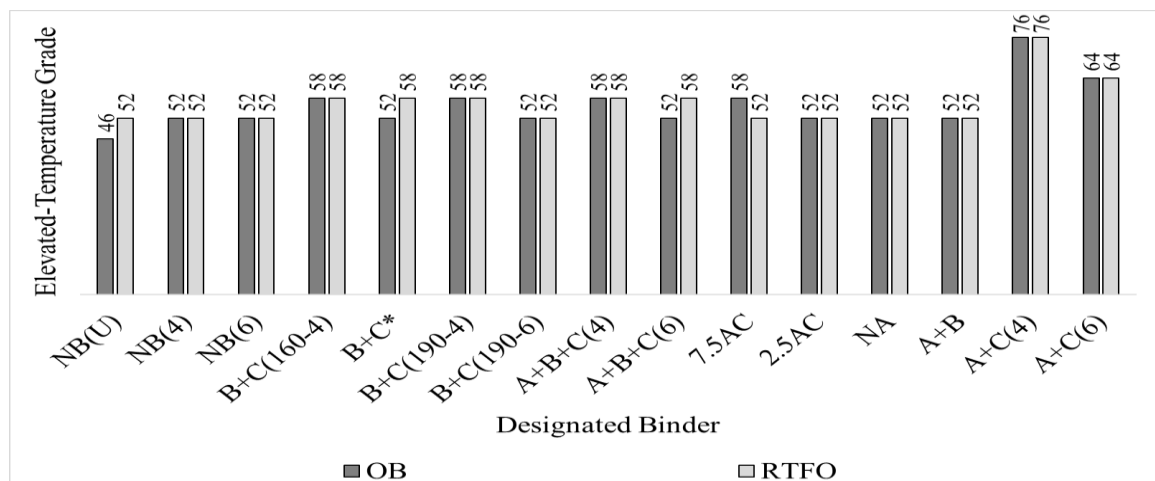
Designated Binder	Binder Code	Temp °C	OB			RTFO			Elevated-Temp. Grade
			G* kPa	δ°	G*/sinδ kPa	G* kPa	δ°	G*/sinδ kPa	
Neat BGR(Unconditioned)	B(U)	46	1.3	85	1.3	10.1	85	10.1	46
		52	0.4	87	0.4	3.2	87	3.2	
		58				1.2	88	1.2	
Neat BGR-3000-190(4hr)	B(4)	46	3.8	86	3.8	12.2	86	12.2	52
		52	1.3	87	1.3	3.9	87	3.9	
		58	0.5	88	0.5	1.4	88	1.4	
Neat BGR-3000-190(6hr)	B(6)	46	3.7	86	3.7	18.2	85	18.2	52
		52	1.3	88	1.3	5.6	87	5.6	
		58	0.5	89	0.5	2.0	88	2.0	
BGR-CRM(10:2)-3000-160(4hr)	B+C(160-4)	46	5.6	77	5.7	23.3	70	24.7	58
		52	2.3	81	2.3	9.5	73	9.9	
		58	1.1	84	1.1	4.0	77	4.2	
		64	0.6	85	0.6	1.9	80	1.9	
BGR-CRM(10:1)-3000*	B+C*	46	3.1	81	3.1	14.9	77	15.3	52
		52	1.2	84	1.2	5.5	80	5.6	
		58	0.6	86	0.6	2.2	82	2.3	
		64				1.0	84	1.0	
BGR-CRM(10:2)-3000-190(4hr)	B+C(190-4)	46	4.7	74	4.9	14.8	71	15.7	58
		52	2.1	78	2.1	6.2	74	6.5	
		58	1.0	81	1.0	2.8	76	2.9	
		64	0.6	83	0.6	1.4	79	1.5	
BGR-CRM(10:1)-3000-190(6hr)	B+C(190-6)	46	2.9	80	3.0	12.8	81	13.0	52
		52	1.2	83	1.2	4.5	83	4.6	
		58	0.6	84	0.6	1.8	85	1.8	
AC-BGR-CRM(5:5:2)-3000-190(4hr)	A+B+C(4)	46	4.6	72	4.8	12.9	70	13.7	58
		52	2.2	76	2.3	5.8	72	6.1	
		58	1.1	78	1.2	2.8	75	2.9	
		64	0.6	80	0.6	1.4	77	1.5	
AC-BGR-CRM(5:5:1)-3000-190(6hr)	A+B+C(6)	46	4.9	76	5.0	13.8	74	14.4	52
		52	2.1	79	2.1	5.7	75	5.9	
		58	1.0	82	1.0	2.5	78	2.6	
		64				1.2	80	1.2	
AC-BGR-CRM(7.5:2.5:1)-3000-190(6hr)	7.5A	46	4.7	78	4.8	9.6	77	9.9	52
		52	2.1	80	2.1	4.1	79	4.2	
		58	1.0	83	1.0	1.9	81	1.9	
		64	0.5	84	0.5				
AC-BGR-CRM(2.5:7.5:1)-3000-190(6hr)	2.5A	46	2.8	81	2.8	10.6	80	10.7	52
		52	1.2	83	1.2	4.0	82	4.0	
		58	0.6	84	0.6	1.6	83	1.6	
Neat AC	AC	46	5.2	84	5.3	11.5	79	11.7	52
		52	2.1	86	2.1	4.6	82	4.7	
		58	0.9	87	0.9	2.0	84	2.0	
AC-BGR(5:5)-3000-190(2hr)	A+B	46	2.6	86	2.6	8.7	85	8.7	52
		52	1.0	87	1.0	3.0	86	3.0	
		58	0.4	87	0.4	1.1	87	1.1	
AC-CRM(10:2)-3000-190(4hr)	A+C(4)	46	23.0	56	27.8	28.2	53	35.2	76
		52	13.0	59	15.2	16.8	54	20.7	
		58	7.3	63	8.2	10.0	56	12.1	
		64	4.1	68	4.5	6.0	58	7.1	
		70	2.3	72	2.4	2.2	65	2.4	
		76	1.3	75	1.4	1.4	69	1.5	
		82	0.8	78	0.8				
AC-CRM(10:1)-3000-190(6hr)	A+C(6)	46	10.4	71	11.0	21.3	64	23.6	64
		52	5.0	74	5.2	10.5	66	11.5	
		58	2.4	77	2.5	5.3	69	5.7	
		64	1.2	79	1.3	2.7	72	2.9	
		70	0.6	82	0.7	1.4	74	1.5	



**Figure4.** Complex modulus susceptibility (OB vs. RTFO) for all designated binders at 52°C



**Figure5.** Phase angle susceptibility (OB vs. RTFO) for all designated binders at 52°C



**Figure 6.** Elevated-Temperature PG of all designated binders: OB vs. RTFO Conditioning

AC+BGR+CRM did not indicate a high difference regarding the final elevated-temperature grade, compared to the mix of BGR+CRM compared to the mix of BGR+CRM. Adding 20%CRM to the binder (AC+BGR) yielded PG58. Likewise, we can declare

A+B+C(6), and 7.5A were very close to achieving PG58. Furthermore, A+B+C(6) acquired PG52 (OB) and PG58 (RTFO), the opposite of 7.5A. Ultimately, upon specs and testing limitations, all of A+B+C(6), 7.5A and 2.5A officially achieved PG52. Through



Subset (4), Neat AC utilized was PG52 indicating the same grade of neat BGR. Likewise, when both (AC & BGR) mixed, they yielded the same rank (PG52). DSR testing depicts the significant impact of adding CRM to the neat AC, Subset (6), versus adding it to the neat BGR, Subset (2). That is clarified by acquiring a grade of PG64 in case of adding 10%CRM to AC, A+C(6), and PG76 in case of adding 20%CRM to AC, A+C(4), however, for the BGR scenarios, PG52, and PG58 respectively. Furthermore, the three-in-blend (AC+BGR+CRM), Subset (3), resulted in disintegrated grades, compared to the two in combination (AC+BGR), Subset (5), however, very close to the two-in-blend (BGR+CRM), Subset (2). As a result, we could proclaim one of two: adding AC to BGR with CRM did not indicate a significant improvement in the performance grade or adding BGR to AC+CRM degrades the performance grade of the overall matrix. However, this last could be beneficial regarding sustainability, economy, and environment.

#### D. Intermediate Temperature Grade

Bio-based binders were conditioned in PAV to simulate the long-term aging to be compared to the control (AC: PG52-28). Regarding Superpave requirements, the fatigue parameter ( $G^*\sin\delta$ ) is required to be lower than 5000kPa corresponding to the intermediate-temperature grade. Upon that, the intermediate-temperature grade was determined for selected binders as reported in Table 3. AC indicated a better performance (not only as a lower stiffness but also as a higher elasticity) compared to guayule-based binders. In other words, all guayule-based binders achieved 25°C as intermediate-temperature grade except for B(6), 28°C, while AC produced 16°C, a significant difference. However, all guayule-based binders accomplished at least the minimum requirem-

ents concerning Superpave Specs except for B(6), whereas Superpave requires the intermediate grade to achieve the average of high and low temperatures plus 4°C (as will be discussed later).

#### E. Master Curves

Master curves were carried out to compare different materials-involved binders. six binders were selected for this which are: (1) AC; (2) B(4), heat treated for 4hr; (3) A+C(4); (4) B+C(4); (5) A+B; (6) A+B+C(4), as described earlier (Table 1). They were created upon frequency sweep from 0.1rad/s to 100rad/sec at different temperatures, and the reference temperature ( $T_{ref}$ ) was selected 50°C. Fig. 7 shows the master curves of those binders. All selected binders except B(4) behaved in similar shapes with a higher grade for A+C(4). However, the neat BGR "B(U)" behaved differently which was more susceptible to frequency change than others, complied with the bio-binders tested by Peralta et al., 2014, [22]. Furthermore, it was observed higher in its master curve than all binders except A+C(4) at lower frequency (before about 0.42Hz). Then, it changed his path to move parallel to other binders (but lower than AC, B+C(4) and A+B+C(4); higher than A+B indicating a higher performance by itself, regarding stiffness and elasticity issues, compared to AC+BGR (50%:50%). Also, the neat asphalt was observed slightly lower than B+C(4) and A+B+C(4) at most frequencies with lower susceptibility for the last two indicating a higher performance for the last two compared to the neat AC (PG52-28) regarding temperature and frequency changes.

### VI. RTFO AGING AND MASS CHANGE

All designated binders were exposed to RTFOT as a simulation for short-term aging utilizing James Cox & Sons RTFO. AASHTO T240 [27] was used to age the assigned binders. Mass changes were measured upon enough replications to verify results and take averages.

Fig.8 depicts the highest mass loss for the unconditioned neat BGR (8.5%). However, when Neat BGR mixed at 3000rpm and 190°C for 4hr and 6hr (i.e., heat-treated) yielded 5.4% and 4.9% respectively, indicating (most likely) most of mass loss caused by water volatiles (complied with bio-binders created by Peralta et al., 2014 [22] & Meier et al., 2013 [28]). Interactions of BGR+CRM via RTFO resulted in a higher mass loss (about 7%), compared to conditioned BGR via 4hr & 6hr mixes which needs more chemical investigation in the future. As RTFO simulates the construction process (mixing & compaction), it is supportive for getting rid of partial-to-all moisture as the act of heat treatment. On the other hand, regarding asphalt binder (AC), the more the asphalt was added, the less the mass loss resulted

**Table 3.** PAV Rheology of Selected Binders Regarding Intermediate-Temperature Grade

Designated Binder	Binder Code	Temp. C	PAV			Intermediate-Temp. Grade
			$G^*$ , kPa	$\delta^\circ$	$G^*\sin\delta$ , kPa	
Neat BGR(Unconditioned)	B(U)	25	4940	74	4746	25
		22	10395	67	9561	
Neat BGR-3000-190(4hr)	B(4)	25	4873	74	4684	25
		22	10871	66	9956	
Neat BGR-3000-190(6hr)	B(6)	25	6762	71	6409	28
		28	3191	77	3110	
BGR-CRM(10:2)-3000-160(4hr)	B+C(160-4)	25	3343	68	3092	25
		22	6923	61	6067	
BGR-CRM(10:1)-3000*	B+C*	25	3210	72	3060	25
		22	7215	66	6579	
BGR-CRM(10:2)-3000-190(4hr)	B+C(190-4)	25	5477	70	5132	25
		22	2700	74	2596	
BGR-CRM(10:1)-3000-190(6hr)	B+C(190-6)	25	4724	71	4456	25
		22	10328	63	9208	
Neat AC	AC	25	2225	46	1610	16
		22	3491	44	2423	
		19	5238	42	3494	
		16	7785	40	4980	
		13	11440	38	7000	



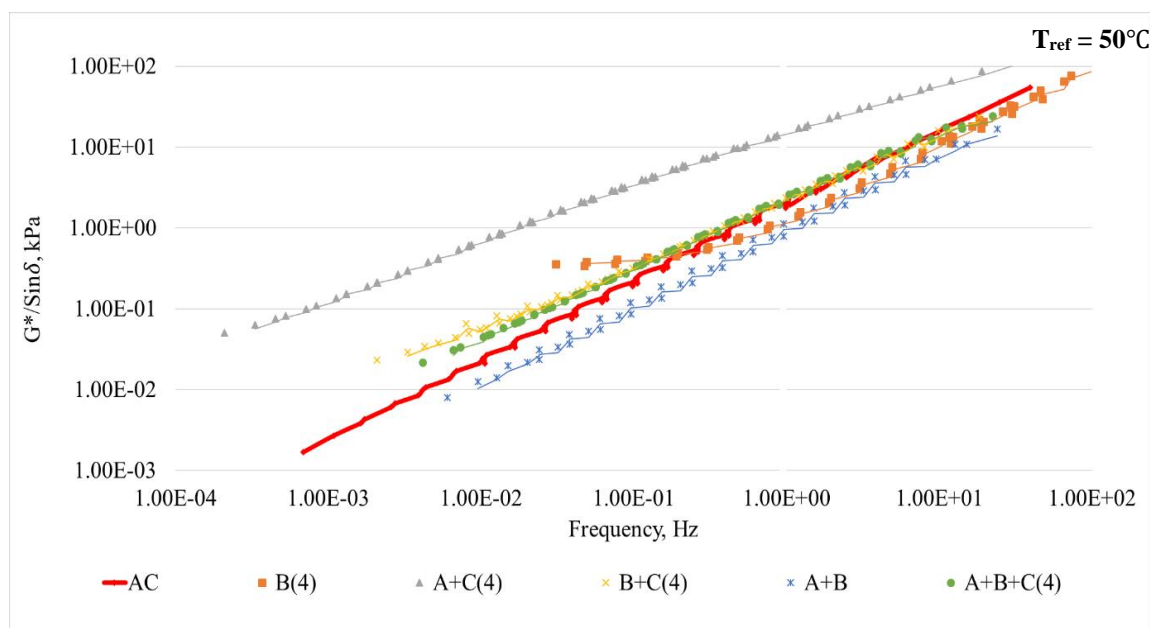


Figure 7. Master curves of different-material-involved binders

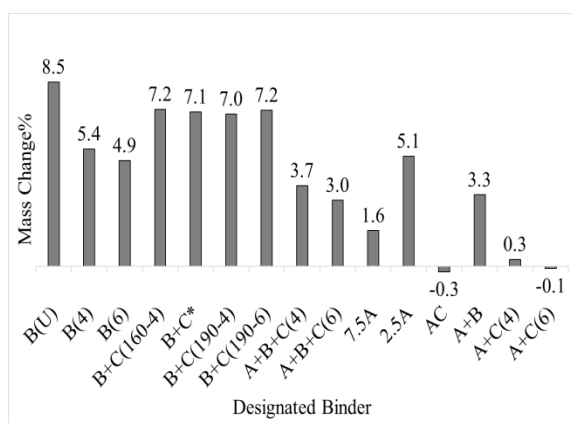


Figure 8. Mass change% for all designated binders by RTFO method

in asphalt-guayule binders. Furthermore, Neat AC or AC+CRM denoted the lowest mass changes via all related assigned binders.

The problem of the relative mass loss of asphalt binder is an environmental issue. That's why ASTM D6373 [29] recommends limitations of  $\pm 1\%$  mass change as complied with either conventional AC or AR binders (Fig. 8). However, a study, in 2005, depicts mass change and emissions associated are not well correlated. Hence, mass change tests do not indicate pass/fail visible emission potential of the binder. Therefore, researchers recommended measuring both mass change and opacity to show the produced visible emissions [8]. Nevertheless, applications of BGR is a new suggestion, and the associated emissions will not be comparable to the asphalt binder emissions which will acquire more investigation in the future. Furthermore, literature regarding bio-binders studies came up with the high

mass loss even with adding only 20% bio-binder to the conventional asphalt [22].

## VII. PAV AGING

The RTFO aged guayule-based binders were exposed to Prentex, Model 9300 PAV System utilizing AASHTO R28 [30]. A 100°C temperature was employed with a pressure of 2.10MPa during conditioning for selected designated samples.

## VIII. LOW-TEMPERATURE GRADE

The guayule-based binders were tested utilizing the Bending Beam Rheometer (BBR) to define their burden at low temperature (AASHTO T313, [31]). Replications were carried out for grade verification. Readings were captured at three different temperatures (0°C, -6°C and -12°C, as reported in Table 4).

Overall observations reveal adding CRM to BGR make the performance worse than unmodified BGR (i.e., neat BGR). It was evident via both stiffnesses (300MPa, max) and m-value, the ability to stress relaxation, (0.300, minimum). Likewise, as a verification, 20%CRM-added had a worse behavior, compared to 10%CRM-added. More dissolution of CRM indicated a worse low-temperature performance, noticed at almost all testing temperatures.

Regarding unconditioned vs. conditioned neat BGR, it was observed that heating neat BGR at 3000-rpm HSM speed for 4hr was the best, at all testing temperatures except -12°C as will be discussed in the upcoming paragraph.

However, raising the mixing time to be 6hr had an adverse effect, even worse than the unconditioned neat BGR. It is expected that, via the

**Table 4.** BBR Results (Stiffness, m-Value and Low-Temperature Grade) for Guayule-Based Binders

Designated Binder	Binder Code	Testing Temp. °C	S Mpa	m-Value	Low-Temp. Grade
Neat BGR(Unconditioned)	B(U)	0	140	0.527	-10
		-6	321	0.335	
		-12	740	0.249	
Neat BGR-3000-190(4hr)	B(4)	0	132	0.541	-10
		-6	319	0.373	
		-12	827	0.262	
Neat BGR-3000-190(6hr)	B(6)	0	164	0.500	-10
		-6	332	0.322	
		-12	797	0.226	
BGR-CRM(10:2)-3000-160(4hr)	B+C(160-4)	0	137	0.468	-10
		-6	476	0.345	
		-12	611	0.251	
BGR-CRM(10:1)-3000*	B+C*	0	154	0.529	-10
		-6	365	0.371	
		-12	628	0.271	
BGR-CRM(10:2)-3000-190(4hr)	B+C(190-4)	0	244	0.398	-10
		-6	472	0.304	
		-12	854	0.221	
BGR-CRM(10:1)-3000-190(6hr)	B+C(190-6)	0	171	0.514	-10
		-6	429	0.332	
		-12	806	0.252	

first 4hr, moisture was released with some volatiles. Nevertheless, during the extra 2hr, the material lost much more low-molecular-weight volatiles (that is supposed to help for better performance at low temperature) indicating worse behavior regarding stiffness and ability to stress relaxation (m-value).

Noticed that all binders significantly passed regarding stiffness and m-value at 0°C. At -6°C, all failed regarding stiffness but kept the ability to stress relaxation. Likewise, all significantly failed at -12°C. Both, higher ability to stress relaxation and stiffness, were not owned by neat BGR at this temperature, unlike testing at 0°C and -6°C. Nevertheless, the variation in readings is not high to record a specific behavior upon the implemented testing.

## IX. CONCLUSION

In this research, 15 created binders [divided into six subsets: Neat AC, Neat BGR, AC+BGR, BGR+CRM, AC+CRM, and AC+BGR+CRM]. All of them were studied (OB vs. RTFO), whereas eight selected binders proceeded with PAV. The following points conclude the significant outcomes:

1. CRM increased the neat BGR viscosity from 114cp to about three times (10%CRM) and about 7-8 times (20%CRM) while the utilized AC (PG52-28) achieved 309cp; corresponding to the BGR+10%CRM binders.
2. Noticed 20%CRM raised the BGR binder performance by about one grade, compared to about four grades for AC.
3. The unconditioned neat BGR accomplished PG46 as unaged, however, when heat-treated or aged, achieved PG52. This last is the same grade of the utilized AC (control), selected to facilitate the comparison to some extent. Nevertheless, both did not have the same low-temperature

grade, -10 and -28 respectively. This was reflected on the intermediate-temperature grade when observing 25°C for all BGR-based binders (except Neat BGR-3000-190(6hr), achieved 28°C) and 16°C for AC utilized.

4. Likewise, master curves showed the behavior of different materials-involved binders (Neat AC, Neat BGR, AC+CRM, BGR+CRM, AC+BGR and AC+BGR+CRM). All of them resulted in the same regular shape (except Neat BGR) with a higher grade for A+C(4), Fig. 7.
5. Interaction of AC+CRM yielded higher performance compared to BGR+CRM. However, focusing on the potential of utilizing BGR (un/modified) could open the door to use a new bio-based binder in flexible pavement industry. Furthermore, employing BGR as an additive to AC (un/modified) has the potential to be comparable to the version AC. Results demonstrated a high-temperature performance of most of the designated binders is equivalent to the neat AC (i.e., Neat BGR, BGR+10%CRM (by wt. of BGR), AC+BGR+10%CRM ((by wt. of AC+BGR); even one more grade when adding 20%CRM to BGR or AC+BGR.
6. Mass loss was observed significantly high, compared to AC. However, mass change is a debatable issue; determined  $\pm 1\%$  by specs for AC regarding the environmental aspect which is not the same hazards regarding bio-binders. Furthermore, many studies created upon bio-binders resulted in high mass losses as well [22, 28].
7. Upon BBR testing, results indicated a pass at 0°C for all guayule-based binders. However, all did not pass at fewer temperatures. Adding 10% or 20%CRM did not denote a positive behavior of the designated binders at low temperature.
8. Ultimately, the guayule resin has the potential as a bio-material to break through the asphalt (flexible pavement) industry to contribute with economic, environmental and sustainable aspects.
9. Furthermore, chemical and further physical testing could help improve the related interactions to accomplish better performance in the future.

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Any perspectives, outcomes, discussions, and conclusions expressed in this study are those of writer(s).

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