

Nonlinear Viscoelastic Response Assessment of Sustainable Asphalt Binders Containing Guayule Resin and Crumb Rubber Using MSCR and Interrupted Shear Flow Tests

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ABSTRACT

This study evaluated the nonlinear viscoelastic (NLVE) response and structural recovery characteristics of sustainable asphalt binders incorporating guayule resin and crumb rubber modifier (CRM). Capturing NLVE behavior is critical to represent field-like conditions under heavy traffic and high temperatures, conditions where traditional linear metrics fail to distinguish binder performance. By assessing guayule resin's potential, the study addresses the growing demand for environmentally responsible materials in modern pavement design.

The experimental matrix included sixteen binders: nine asphalt–rubber–guayule (ARG) blends with varying AR and CRM dosages, five corresponding asphalt–rubber–asphalt (ARA) controls, and two neat references. Performance was evaluated using Multiple Stress Creep Recovery (MSCR) and Interrupted Shear Flow (ISF) tests, capturing elastic recovery, stress sensitivity, and structural reorganization, supported by DSR-based rheology and viscosity profiling. This comprehensive testing framework offers insights into both nonlinear and linear binder behavior.

MSCR results demonstrated that although ARG binders exhibited measurable elastic recovery and nonlinear response, they were generally outperformed by their corresponding asphalt rubber binders in terms of percent recovery and stress sensitivity. However, several ARG formulations still matched or exceeded the performance of the control asphalt binder, indicating their viability as sustainable alternatives. ISF testing further highlighted the unique recovery capacity of guayule–rich binder through rebuilding internal structure within 5 to 10 seconds of rest, behavior not captured by conventional testing or observed in asphalt rubber binders. These findings underscore the active role of guayule resin in nonlinear recovery and structural resilience, supporting its use in sustainable binder technologies.

Keywords: Asphalt Binder Modification, Guayule Resin, Interrupted Shear Flow (ISF), Multiple Stress Creep Recovery (MSCR), Resilience, Sustainability

INTRODUCTION

The pursuit of sustainable infrastructure has accelerated the development of renewable and environmentally sustainable asphalt binder alternatives [1]. Among these, bio-based modifiers such as vegetable oils, lignin, waste-derived oils, and rubber extracts have gained recognition for their ability to reduce reliance on fossil resources and minimize environmental impact [2, 3]. However, many of these materials face issues related to phase separation [4], inadequate high-temperature performance, or oxidation instability, particularly when used in modified or recycled systems [5, 6].

Guayule (*Parthenium argentatum*) is a perennial shrub native to arid regions of the southwestern U.S. and northern Mexico [7]. Guayule resin, a viscous co-product of rubber extraction from *Parthenium argentatum*, is rich in terpene-based compounds and resin acids [8] that impart asphalt-like thermoplastic and viscoelastic behavior at service temperatures [9]. Hemida and Abdelrahman [10] introduced guayule resin as a partial, and in some cases, entire, replacement for conventional asphalt binder [10]. When used alone, guayule resin exhibited a binder performance grade comparable to PG 58-11 [11], highlighting both its promise and the inherent challenges in developing it into a fully competitive alternative [12]. However, its standalone use has been associated with lower stiffness and reduced rutting resistance, underscoring the need for further research to meet a full range of performance requirements [12]. Consequently, recent studies have incorporated crumb rubber modifier (CRM) to create asphalt–rubber–guayule (ARG) systems to enhance the mechanical performance of guayule resin–based binders [13]. CRM contributed to elastic recovery, resistance to permanent deformation, and long-term durability [14]. When blended with guayule resin, interactions might develop that promote internal elastic networking [9, 15] and mitigate phase instability [12, 13, 16]. These benefits were particularly impactful when combined with guayule resin, which otherwise offered limited structural reinforcement alone [17].

While the Superpave system traditionally uses the $|G^*|/\sin(\delta)$ parameter to assess rutting resistance, it is limited in capturing the complex, nonlinear viscoelastic (NLVE) behavior of highly modified binders. This parameter, measured at a single frequency (10 rad/s), often fails to distinguish between binders with significantly different field performances, especially under high stress or traffic loading [18, 19]. To address these limitations, the Multiple Stress Creep Recovery (MSCR) test was developed and standardized under AASHTO T 350 [20]. It evaluates binder recovery and stress sensitivity under repeated loading at two stress levels (0.1 and 3.2 kPa), producing non-recoverable creep compliance (J_{nr}) and percent recovery (%R) as key indicators of NLVE response [19]. Prior studies have confirmed MSCR's effectiveness in characterizing CRM-modified and bio-blended systems under more realistic loading conditions than traditional oscillatory tests [14, 19, 21]. Capturing NLVE response is essential to simulate field-like behavior, particularly in climates with high heat and heavy traffic [22]. Such response governs rutting resistance and recoverability beyond what linear tests can detect [23].

To further probe time-dependent recovery behavior, the Interrupted Shear Flow (ISF) test has recently emerged as a promising evaluation tool for modified asphalt binders [24–27]. This method applies cyclic shear–rest loading in controlled shear rate mode and captures the stress overshoot and recovery response following each rest interval, thereby revealing the binder's capacity for internal structural reorganization [28], often referred to as thixotropic behavior [29, 30]. This characteristic is particularly critical for pavements subjected to repeated loading interruptions, such as intersections, ramps, and stop–and–go urban environments [30]. These insights underscore the importance of supplementing traditional DSR-based testing with performance-oriented approaches, such as MSCR and ISF, to fully capture the dynamic behavior of sustainable binder systems.

OBJECTIVE AND SCOPE

This study aimed to evaluate the NLVE response, stress sensitivity, and structural recovery behavior of ARG binders using advanced rheological testing methods. By linking binder formulation and rheological response to performance indicators derived from MSCR and ISF tests, this research clarified the mechanistic role of guayule resin in enhancing binder recoverability and resilience under traffic-like stress conditions. The broader objective is to support the transportation community's shift toward performance-based specifications and sustainable infrastructure solutions by advancing the understanding and practical viability of guayule-based binder technologies.

A total of sixteen binders were prepared. These included: nine ARG binders with AR contents of 25%, 50%, and 75% (by total binder weight), each modified with 10%, 15%, and 20% CRM (by asphalt portion weight); five corresponding ARA binders in which guayule resin was replaced by asphalt; and two control samples, neat asphalt and guayule resin. The evaluation matrix included MSCR testing at 0.1 and 3.2 kPa to quantify %R, J_{nr} , and stress sensitivity (J_{nr_diff}), followed by ISF testing under a shear rate of 2 s^{-1} with rest intervals ranging from 5 to 60 seconds to assess time-dependent structural rebuilding and thixotropic behavior. Basic rheological characterization was also conducted using the conventional dynamic shear rheometer (DSR) measurements to determine complex modulus ($|G^*|$), phase angle (δ), and the Superpave rutting parameter $|G^*|/\sin(\delta)$. Steady shear viscosity testing was also performed on neat guayule resin to evaluate its temperature-dependent flow characteristics. This multi-layered testing framework delivers a comprehensive assessment of binder behavior across both nonlinear and linear viscoelastic domains, offering performance insights directly relevant to the design of flexible pavements subjected to realistic traffic conditions.

MATERIALS AND METHODS

Rheological characterization of the binder systems was conducted through a structured testing sequence designed to align with the study's multi-level analysis of nonlinear viscoelasticity, structural recovery, and fundamental binder properties. **Figure 1** demonstrates the methodology followed in this study.

Materials

The experimental work utilized three primary components: (1) conventional asphalt binder (A), (2) heat-treated guayule resin (G), and (3) crumb rubber modifier (CRM; mesh #30–40 consistent with U.S. standards) [10, 31–33]. **Table 1** summarizes the key physical properties of the base asphalt (A) and guayule resin (G).

Table 1. Properties of Control Asphalt vs. Heat-Treated Guayule Resin

Property	A	G	Method
Flash Point, °C	320	242	ASTM D92 [34]
Fire Point, °C	330	261	ASTM D92 [34]
Density at 25°C, g/dm ³	1028	1038	ASTM D70 [35]
Penetration at 25°C	50/60	40/50	ASTM D5 [36]
Viscosity at 135°C, Pa.s	0.403	0.203	ASTM D4402 [37]
Softening Point, °C	47	48	ASTM D36 [38]
Standard PG	64-22	58-10	AASHTO M 320 [39]
Continuous PG	67-25	58-11	

Three AR blends were first prepared with CRM contents of 10%, 15%, and 20% by asphalt weight, designated as (10), (15), and (20). Subsequently, a total of nine ARG binders were prepared by combining AR and guayule resin (G) at AR:G ratios of 25:75, 50:50, and 75:25 by total binder weight, yielding the ARG25, ARG50, and ARG75 series for each CRM level, led to varies CRM dosage

between 2.3% to 12.5% by total binder weight. Additionally, to isolate the resin's effect and equivalence the CRM dosage, five asphalt–rubber–asphalt (ARA) control binders, ARA25, ARA50, and ARA75, were also made by blending AR and base asphalt (A) at the same ratios. **Figure 2** illustrates the sample preparation sequence, and **Table 2** lists all binder designations and proportions. For example, ARG50(15):50 denotes a blend containing 50%AR (15%CRM) and 50% G, while ARA50(15):50 denotes a blend containing 50% AR (15% CRM) and 50% A.

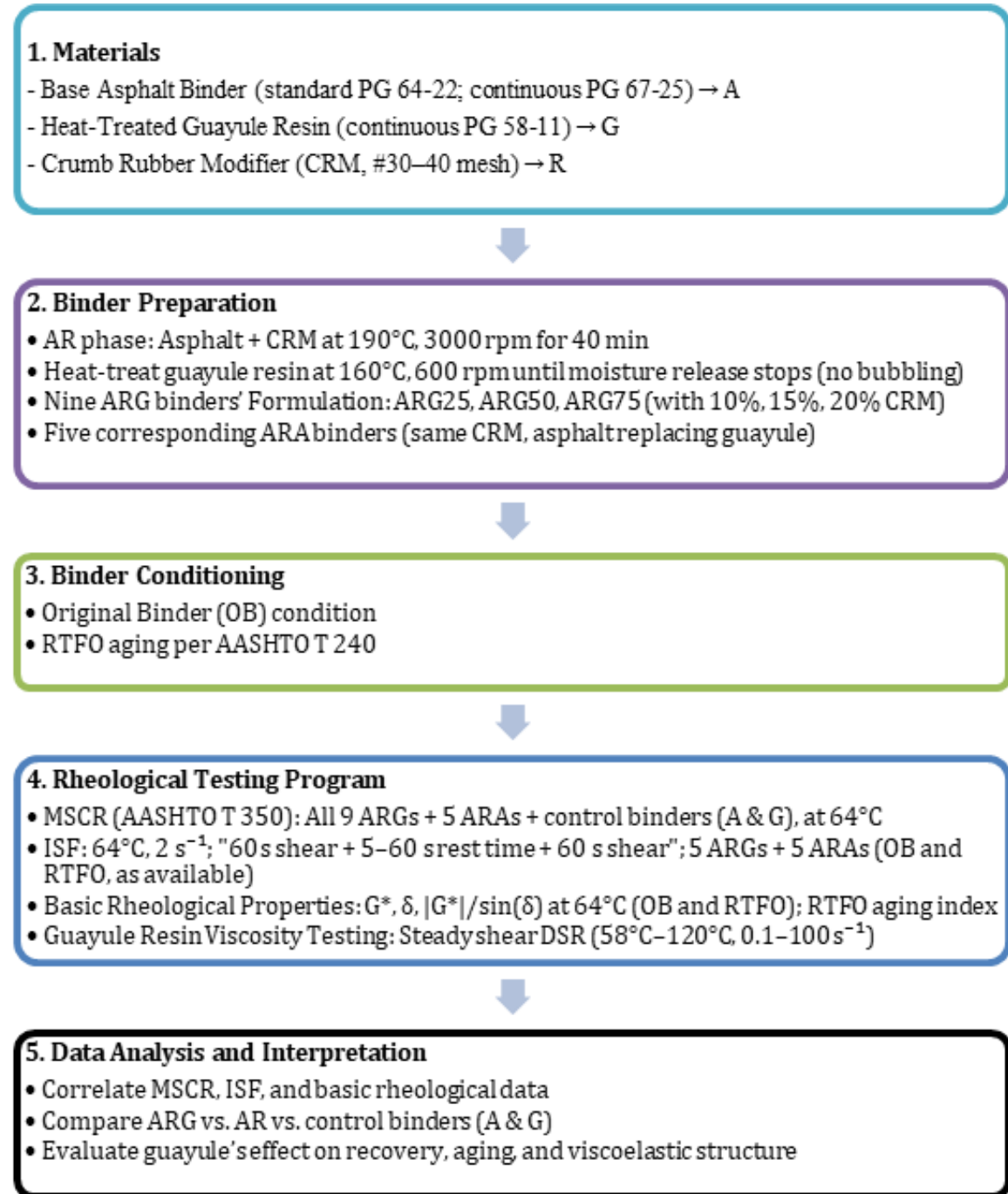
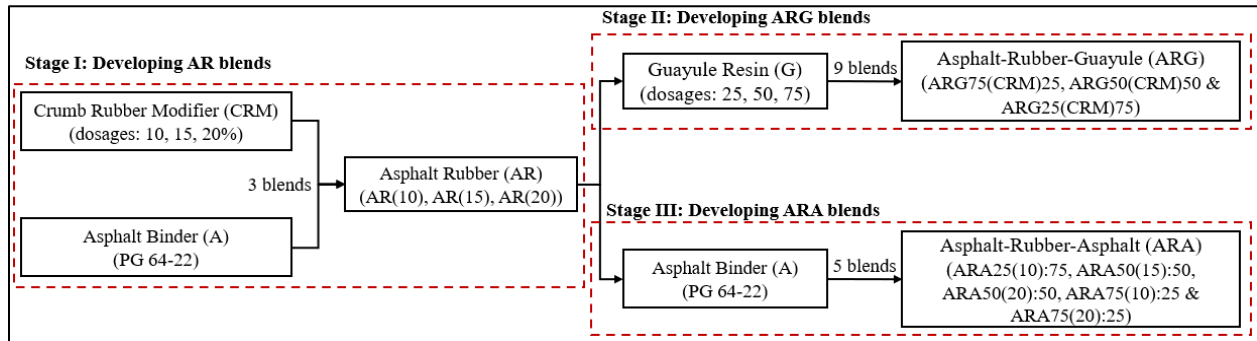


Figure 1. Study Methodology

Table 2. Binder Designation and Proportions

Group	Binder Designation "Blend-AR _{dosage} - CR _{dosage} -G/A _{dosage} "	Binder Proportions			
		A%	G%	CRM%	AR%
A	A	100	0		
G	G	0	100		
ARG	ARG25(10):75	22.7	75	2.3	25
	ARG25(15):75	21.7	75	3.3	25
	ARG25(20):75	20.8	75	4.2	25
	ARG50(10):50	45.5	50	4.5	50
	ARG50(15):50	43.5	50	6.5	50
	ARG50(20):50	41.7	50	8.3	50
	ARG75(10):25	68.2	25	6.8	75
	ARG75(15):25	65.2	25	9.8	75
	ARG75(20):25	62.5	25	12.5	75
ARA	ARA25(10):75	97.7	0	2.3	25
	ARA50(15):50	93.5	0	6.5	50
	ARA50(20):50	91.7	0	8.3	50
	ARA75(10):25	93.2	0	6.8	75
	ARA75(20):25	87.5	0	12.5	75

A: control asphalt PG64-22; G: control heat-treated guayule resin; AR: asphalt-rubber binder; ARG: asphalt-rubber-guayule blend; ARA: asphalt-rubber-asphalt blend.

**Figure 2. Sample Preparation Sequence**

These nine formulations were designed to systematically evaluate the influence of guayule resin on the viscoelastic and rutting performance of AR binders. While five of these blends (ARG25(10):75, ARG50(15):50, ARG50(20):50, ARG75(10):25, and ARG75(20):25) were selected for extended rheological testing (e.g., $|G^*|$, δ , ISF), all nine ARG blends were subjected to MSCR evaluation to capture the full spectrum of behavior across the formulation matrix. Additionally, for reference, the two neat binders, control asphalt binder and heat-treated guayule resin, were also tested to characterize their individual contributions to the composite blends.

Sample Preparation

All binder blends were prepared using a multi-stage high-shear mixing process to ensure homogeneous dispersion of CRM and full integration of guayule resin within the binder matrix. The AR blends were first prepared with varies CRM concentrations (10%, 15%, or 20% by asphalt portion weight) by gradually adding oven-dried CRM into the preheated asphalt binder at a temperature of 190°C. This mixture was blended for 40 minutes at 3000 rpm to promote CRM swelling and uniform interaction with the base asphalt.

For ARG blends, firstly, the acquired guayule resin was subjected to a thermal conditioning step in which it was heated at 160°C under agitation at 600 rpm until the moisture was eliminated (**Figure 3**). The endpoint of the heat treatment was determined by the complete disappearance of surface bubbling, ensuring stability for subsequent blending. Following these preliminary steps, the asphalt–rubber and guayule components were mixed in designated proportions to produce the nine asphalt–rubber–guayule (ARG) binders. The final ARG and ARA blends were mixed at 160°C for an additional 60 minutes at 600 rpm.



Figure 3. Processing Stages of Guayule Resin from Initial Storage to Heat Treatment

Methods

Binder Conditioning

All prepared binders were subjected to short-term aging using the Rolling Thin Film Oven (RTFO) method, as specified in AASHTO T240 [40], to simulate the oxidative conditioning encountered during plant mixing and field application.

Basic Rheological Characteristics

Basic rheological parameters were measured using oscillatory DSR testing at 64°C. These included complex shear modulus ($|G^*|$), phase angle (δ), and the computed rutting parameter ($|G^*|/\sin(\delta)$) was measured. Tests were conducted on both original binder – OB (unaged) and RTFO-aged binders to evaluate short-term oxidative hardening effects. The RTFO aging index, expressed as the ratio of $|G^*|/\sin(\delta)$ after and before aging, was calculated for each formulation. These measurements served to define the binders' stiffness–elasticity balance and aging susceptibility.

Furthermore, the flow behavior of neat guayule resin was investigated through steady shear testing using the DSR. Tests were conducted over a temperature range of 58°C to 120°C and shear rates from 0.1 to 100 s⁻¹. The resulting viscosity curves and shear stress–shear rate relationships were analyzed to evaluate flow behavior (Newtonian or non-Newtonian) and to assess the guayule resin's temperature-dependent rheological stability.

Multiple Stress Creep Recovery (MSCR) Test

The MSCR test served as the primary evaluation method for assessing nonlinear viscoelastic behavior and rutting resistance. The test was conducted on the RTFO-aged binders per AASHTO T350 [20] at 64°C, which exceeds the typical linear viscoelastic threshold for asphalt binders and simulates traffic-like shear loading [19, 21]. Each binder underwent 10 creep-recovery cycles at 0.1 kPa, followed by 10 cycles at 3.2 kPa. Each cycle consisted of a 1-second load application followed by a 9-second recovery period. The testing temperature was selected to allow a consistent comparative framework across all binder

formulations, rather than for PG grading purposes. Key output parameters included the non-recoverable creep compliance (J_{nr}), percent recovery (%R), and stress sensitivity index (J_{nr_diff}), representing the binder's ability to resist permanent deformation and recover elastically under increasing load intensity.

Interrupted Shear Flow (ISF) Test

The interrupted shear flow (ISF) test was performed to assess the structural recovery capacity and internal reorganization of the binders under cyclic shear–rest conditions [25]. Testing was conducted at 64°C in controlled shear rate mode (2 s^{-1}) [17, 24–27]. Each ISF cycle comprised a 60-second continuous shear flow segment, followed by a rest period ranging from 5 to 60 seconds, replicating the same conditions as the initial cycle. Both OBs and RTFO-aged binders were tested to evaluate the impact of oxidative aging on structural integrity. During each rest interval, key features such as stress overshoot, steady-state plateau, and post-rest decay were closely monitored. Generally, the ISF test provided a deeper view of structural recovery, flow stability, and viscoelastic reorganization under high temperature loading conditions. While MSCR quantifies instantaneous recovery and stress sensitivity, ISF captures how modified binders respond over extended loading–rest sequences.

RESULTS AND DISCUSSION

To assess the viscoelastic performance of ARG binder blends, this section presents a structured analysis of their nonlinear recovery behavior, structural rebuilding capacity, and fundamental rheological characteristics. The discussion begins with the Multiple Stress Creep Recovery (MSCR) test results, which provide insight into the binders' elastic response and stress sensitivity under repeated high-temperature loading. This is followed by an evaluation of structural recovery patterns using the Interrupted Shear Flow (ISF) test, which is designed to capture thixotropic and time-dependent behaviors that are not evident in conventional tests. Lastly, key findings from basic rheological characterization—including complex modulus ($|G^*|$), phase angle (δ), and $|G^*|/\sin \delta$ —are interpreted in relation to the advanced performance parameters to establish a comprehensive understanding of binder behavior across both linear and nonlinear domains.

Multiple Stress Creep Recovery (MSCR)

At constant temperature, decreasing the value of J_{nr} parameter and increasing the value of %R parameter, indicating the binder's ability to resist permanent deformation and elastic rebound capacity, respectively.

MSCR Test Parameters: ARGs vs. ARAs

Figure 4 summarizes the MSCR results for the proposed blends. As guayule resin content decreased in the blend, the J_{nr} values decreased substantially and recovery rates improved, compared to neat asphalt binder (A). Incorporating 25–50% guayule resin in place of neat asphalt binder (A) delivered recovery rates and stiffness values that met or exceeded those of the control. Although the J_{nr} values of ARG50(10):50 and ARG50(15):50 exceeded the threshold for heavy traffic classification, their controlled deformation and moderate recovery indicate balanced viscoelastic performance, making them suitable for moderate traffic applications. Notably, while the ARG50 binders exhibited limited elastic recovery ($R_{0.1}$ between 18.6% and 33.1%) and negligible recovery at higher stress ($R_{3.2} \approx 0\%$), they demonstrated load tolerance and structural integrity, which are desirable properties for flexible pavement layers. While the ARG75(20):25 recorded the lowest J_{nr} values among the ARG series and achieved the highest percent recovery, with $R_{0.1} = 54.1\%$ and $R_{3.2} = 11.4\%$, reflecting strong elastic response. On the other side, the ARA binders consistently outperformed the ARG series in both J_{nr} and %R values, reflecting their inherently stronger elastic response and superior rutting resistance. For example, ARA75(20):25 showed the lowest J_{nr} and highest $R_{0.1}$, benefiting from high CRM content and uninterrupted interaction with a stiffer asphalt matrix.

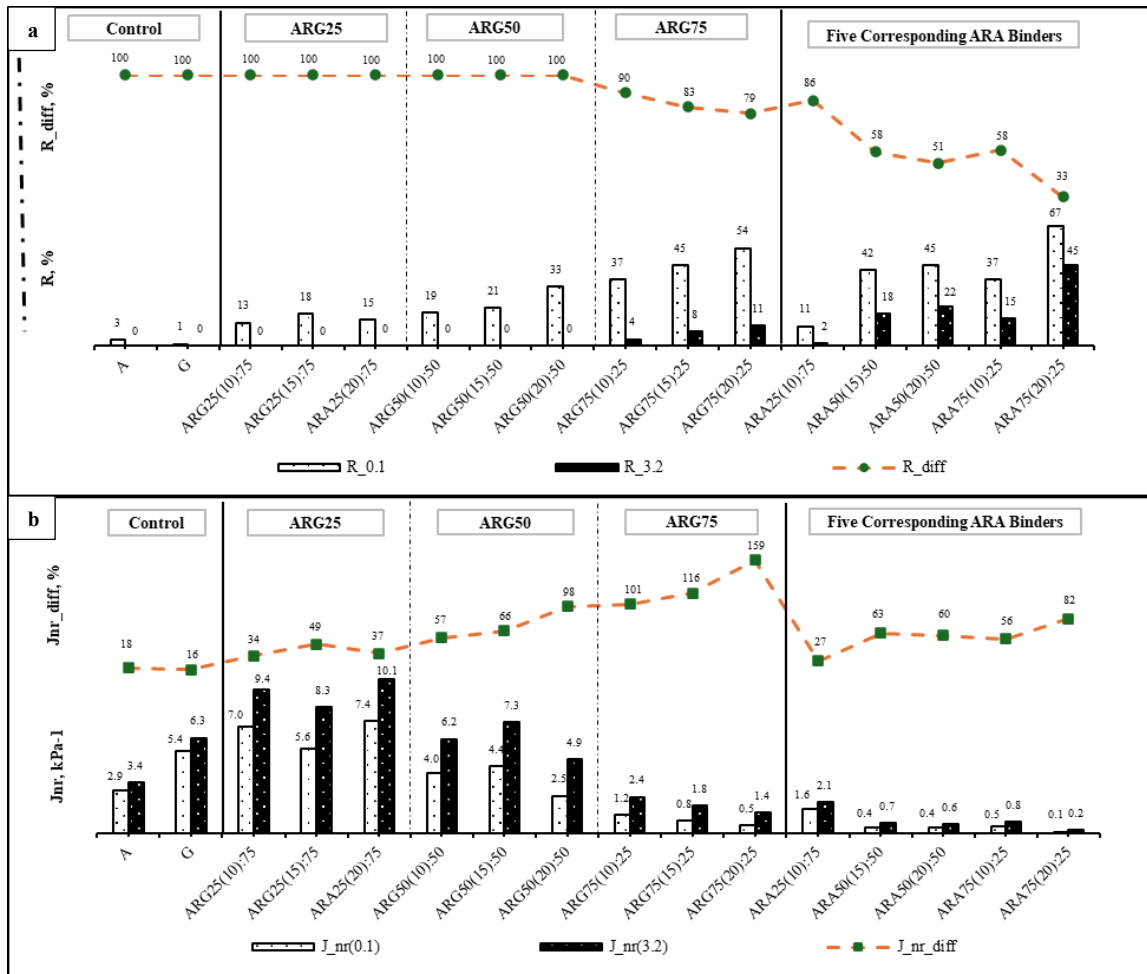


Figure 4. MSCR Test Results of the ARG and ARA Binders: (a) Recovery and (b) Creep Compliance

Stress sensitivity index (J_{nr_diff}), the percentage increase in non-recoverable creep compliance (J_{nr}) from 0.1 to 3.2 kPa, provides insight into the binder's stability under increasing load intensity. The ARG75 series exceeded the 75% J_{nr_diff} limit in AASHTO M332 [41], indicating pronounced stress sensitivity. This behavior is typical of highly modified CRM binders: dispersed rubber particles create an elastic-rich, networked microstructure, introducing nonlinearity that amplifies changes in J_{nr} under higher stresses [42]. Conversely, ARG50(10):50 and ARG50(15):50 maintained acceptable J_{nr_diff} values (57% and 66%, respectively), suggesting more stable behavior under varying loads. As CRM content increased across the ARA series (from ARA25 to ARA75), the binders exhibited reduced creep compliance, confirming enhanced stiffness. However, this improvement came with a trade-off: the J_{nr_diff} values also rose, reaching 82% for ARA75(20):25.

Finally, these trends demonstrated that increasing AR content effectively enhanced binder stiffness and recoverability, even when using guayule resin as a partial replacement for traditional asphalt. The ARG showed comparable and even superior recovery behavior, but lower stiffness, to neat binder, indicating the valueability of utilizing guayule in cold to moderate climate conditions. While ARG75 exhibited high-temperature NLVE behavior comparable to ARA25 blend. This formulation strategy not only improved performance metrics but also promotes environmental sustainability by utilizing guayule resin byproducts and recycled rubber materials.

Insights from Creep–Recovery Response Curves: ARGs vs. ARAs

The time-strain curves in **Figure 5a–f** offer visual confirmation of the MSCR results. At 0.1 kPa, the ARG75 binders showed partial recovery after each load, while ARG50 binders exhibited moderate rebound, and ARG25 binders demonstrated little to no recovery. At 3.2 kPa (**Figure 5c**), all ARG binders experienced dominant viscous flow with negligible rebound, consistent with the reduced %R observed.

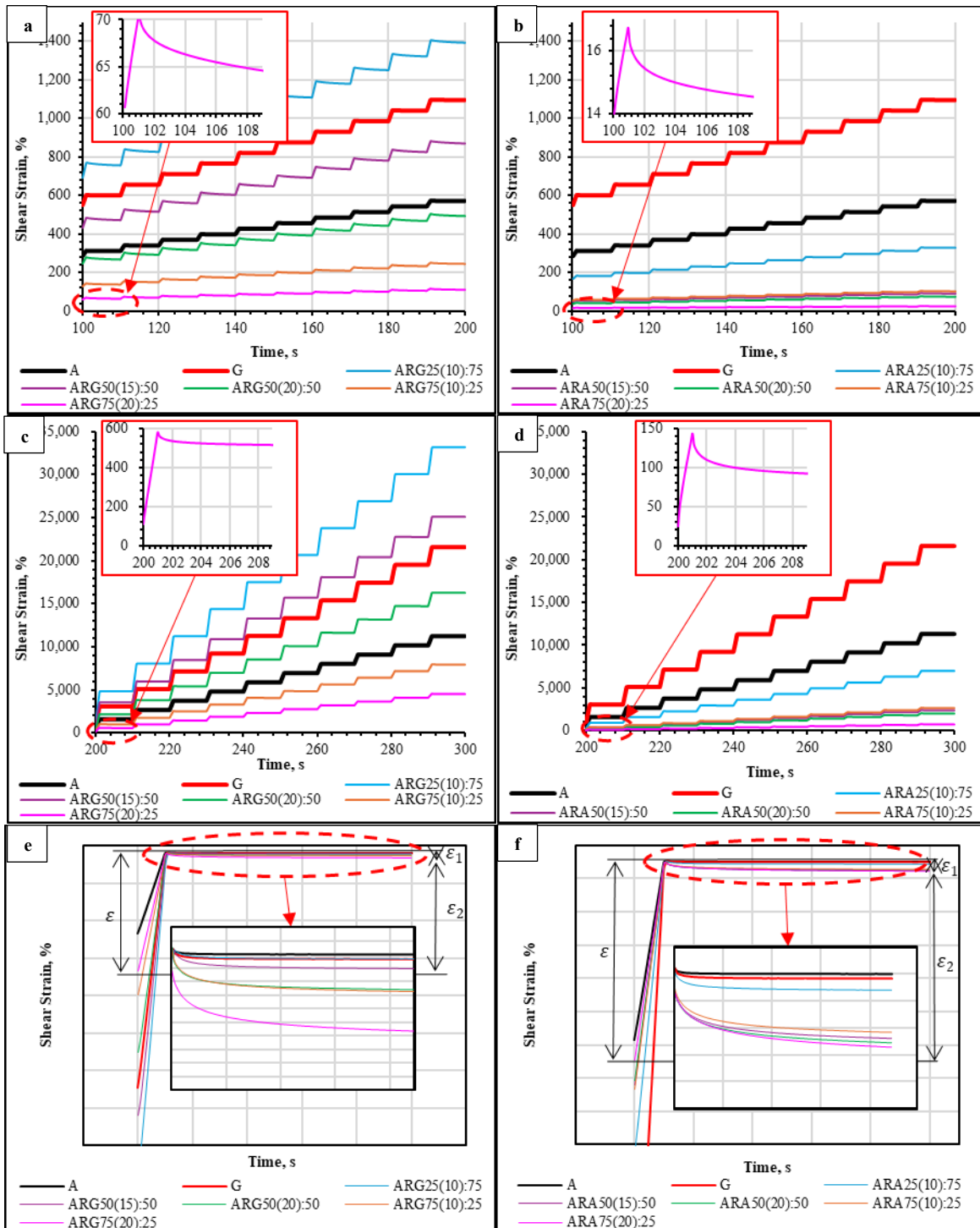


Figure 5. MSCR Creep-Recovery Cycle Response of ARG (Left) and ARA (Right) Binders: (a&b) at 0.1 kPa Stress Level; (c&d) at 3.2 kPa Stress Level; (e&f) Normalized Strain Response Single Cycle to Control Asphalt Peak

Normalized cycle responses (**Figures 5e&f**) further highlight the differences between the ARG and ARA systems. The ARA binders displayed sharper recovery peaks and lower total strain, whereas ARG binders showed higher total deformation and less elastic rebound. Nevertheless, ARG75(20):25 exhibited slightly greater recoverable strain than ARA75(20):25, indicating that guayule resin might enhance elasticity at high CRM levels, likely due to its polymeric contribution to the overall binder matrix [9].

Strain Breakdown and Performance Interpretation: ARGs vs. ARAs

Figure 6 provides a detailed breakdown of total, recoverable, and non-recoverable strain for both binder families at 3.2 kPa. As expected, ARA binders showed lower total strain and higher recoverable strain, confirming a more robust elastic network. ARG binders, while more deformable, benefited from increasing CRM content. For instance, the progression from ARG50(15):50 to ARG50(20):50 (with a 2% increase in CRM) resulted in a notable improvement in recoverable strain, a trend less evident in the corresponding ARA binders. This indicates that CRM is especially effective in enhancing the mechanical response of guayule-containing systems, even more so than in ARA blends in certain cases.

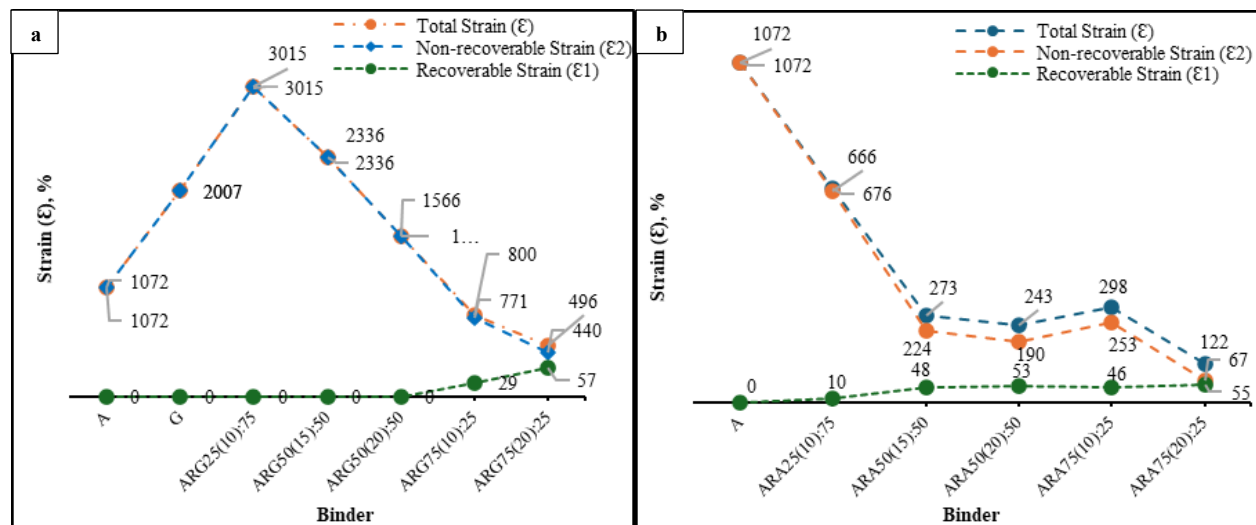


Figure 6. Strain (Total, Recoverable, and Non-Recoverable) Response of (a) ARG and (b) ARA Binder Blends at 3.2 kPa Stress Level

Overall High-Temperature MSCR Performance Observations

As summarized in **Figure 7**, the addition of CRM to both binder systems improved high-temperature performance. ARA binders, formulated with conventional asphalt, naturally exhibited stronger MSCR metrics. However, CRM addition to guayule–asphalt systems produced consistent improvements, confirming guayule resin’s compatibility with traditional modifiers [43]. Given its lower base PG classification (PG 58-11), guayule resin began from a softer performance baseline, yet when combined with increasing CRM, demonstrated a progressively stronger response.

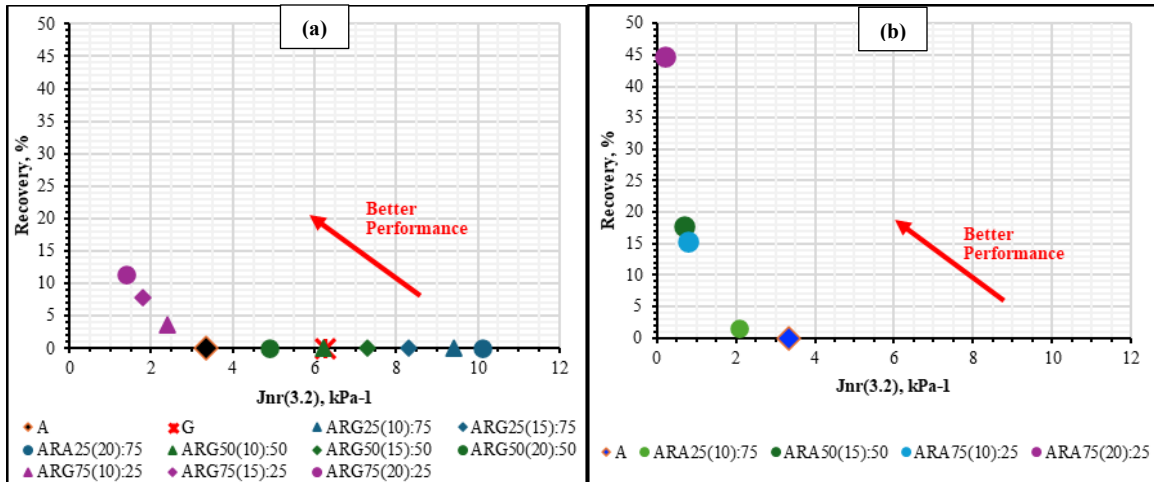


Figure 7. Overall High-Temperature Performance of the designated binders based on the MSCR test: (a) ARG binders and (b) ARA Binders

These results support the conclusion that guayule resin, though not equivalent to conventional asphalt in initial stiffness, can serve as a viable binder base in engineered systems. When paired with CRM, it forms composite binders capable of tailored stiffness and recovery characteristics, making it a promising candidate for sustainable binder formulations, especially in climates or applications where enhanced flexibility and environmental considerations are priorities.

Interrupted Shear Flow Analysis

The ISF test provides valuable insight into the short-timescale recovery dynamics, complementing the MSCR test by capturing the flow stability and internal reorganization of binders under cyclic loading conditions relevant to high-temperature service environments. As shown in **Figure 8**, for each binder, the lower profile corresponds to the OB condition (solid lines), and the upper profile reflects the RTFO-aged binder (dotted lines); in cases with only one curve, only the RTFO-aged binder was tested.

A distinctive feature observed across all modified binders, both ARG and ARA, is the presence of a sharp initial shear stress overshoot, followed by a brief degradation in stress before transitioning into either a steady-state plateau or a gradually decaying trend. This pattern suggests that modified binders exhibit viscoelastic domains that activate upon sudden shear, resulting in an elastic recoil, followed by the breakdown of weak structural elements that cannot sustain prolonged deformation. Notably, this overshoot–degradation pattern was absent in the base PG 64-22 binder, both in its OB and RTFO-aged states, which displayed smooth, continuous shear flow with no elastic features in the nonlinear stress range. This contrast highlights the NLVE behavior introduced by crumb rubber and guayule resin, as evidenced by stress overshoot, elastic recoil, and structural recovery—features absent in conventional PG 64-22 asphalt.

Among the ARG binders, ARG25(10):75, ARG50(15):50, and ARG50(20):50 showed a unique and consistent behavior: a repetitive onset stress overshoot of equal magnitude after every rest period across the full 5–60 s range. This behavior strongly suggests that these formulations possess a rebuildable internal structure, capable of fully recovering its elastic response between cycles. This dynamic reactivation was not observed in ARA binders or highly rubberized ARG binders (e.g., ARG75(20):25), where either the network is too rigid to reconfigure or the oxidative stiffening from RTFO aging suppresses recovery. These findings implied that guayule-rich or moderately rubberized ARG systems may offer superior adaptability to intermittent shear conditions, such as those experienced in stop-and-go traffic zones, where structural rebuilding between load events is beneficial.

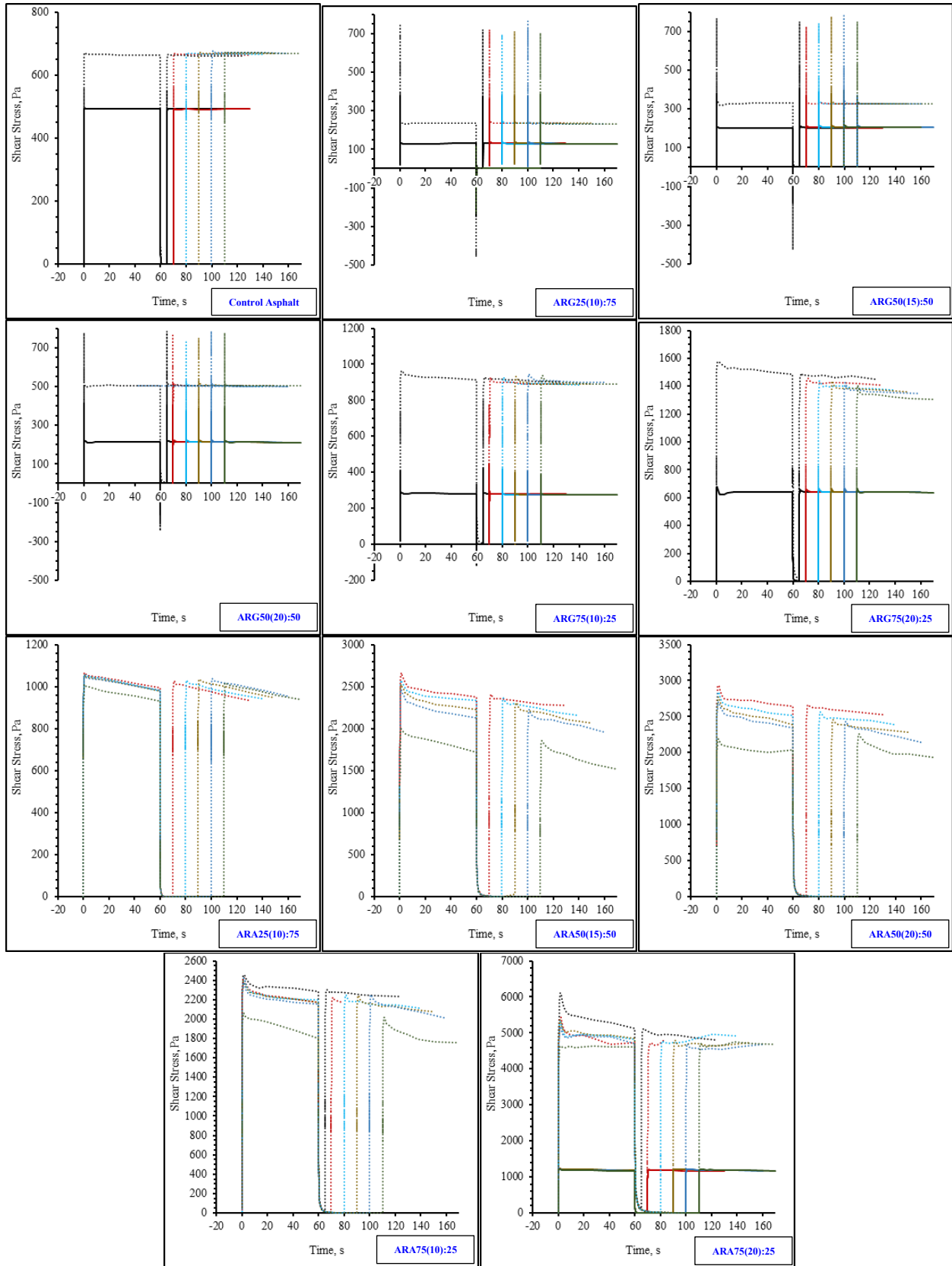


Figure 8. Interrupted Shear Flow Test Results of A, ARG, and ARA Binders

Overall, the results confirmed that ARG binders with balanced guayule–CRM content (e.g., ARG50(15):50) exhibited superior structural resilience and recovery dynamics under cyclic shear, supporting their viability as sustainable alternatives to conventional ARA systems and superior to neat binder, especially for pavements exposed to repetitive or variable high-temperature loading patterns.

Insights into Basic Rheological Properties

Guayule Resin Viscosity Behavior

Although guayule resin exhibited Newtonian behavior under steady shear testing at low stress levels (**Figure 9**), its nonlinear viscoelastic (NLVE) response became evident during MSCR and ISF testing. This contrast reflects the resin’s internal structure, which remains inactive under low-strain flow but responds to high-stress, cyclic deformation. Guayule resin was evaluated separately to establish its baseline rheological behavior prior to blending. Future studies should extend viscosity characterization to guayule–CRM hybrid binders to better understand interactive effects on flow behavior and processability.

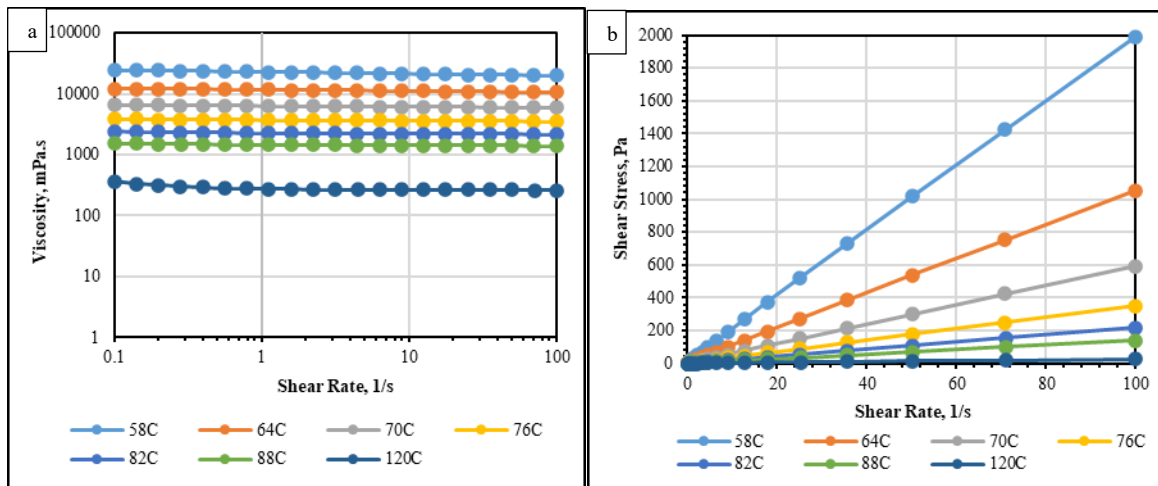


Figure 9. Guayule Resin Viscosity Characteristics: (a) Viscosity and (b) Shear Stress, per Shear Rate

Complex Modulus ($|G^*|$) and Phase Angle (δ)

Figure 10 presents a detailed evaluation of the basic viscoelastic behavior of the ARG binders and their corresponding ARA blends at a test temperature of 64°C, under both OB and RTFO-aged conditions. As shown in **Figures 10a-b**, ARA binders consistently exhibited higher complex modulus ($|G^*|$) and lower phase angle (δ) compared to their ARG counterparts at both aging states. The elevated $|G^*|$ reflects greater viscoelastic stiffness, while the lower δ indicates more elastic-dominant behavior. These trends are especially evident at higher CRM contents. For example, at the 75(20):25 modification level after RTFO aging, the ARA binder reached a $|G^*|$ of 15.6 kPa, while the corresponding ARG binder measured only 5.8 kPa, a nearly threefold difference. Such a disparity cannot be attributed solely to the difference in base binders, as guayule resin inherently exhibits significantly lower stiffness and higher phase angles compared to conventional asphalt.

This phenomenon can be partially explained by the diluting or plasticizing effect of guayule resin when blended into asphalt rubber systems. Literature revealed that, in the absence of asphalt, CRM-modified guayule resin has been shown to provide only modest improvements in high-temperature performance [43], with an approximately 1.4-fold increase in rutting resistance [12]. In contrast, the same CRM level added to neat asphalt produced a sixfold enhancement [12]. This suggests that CRM particles interact more effectively with the aromatic and asphaltene-rich environment of conventional asphalt than with the

comparatively soft and polymeric matrix of guayule resin. Therefore, in ARG binders, guayule resin tended to disrupt or weaken the elastomeric network typically formed between CRM and asphalt, resulting in lower stiffness and elasticity metrics. Additionally, the higher phase angle values (δ) observed across all ARG systems compared to their ARA counterparts further support this interpretation. Elevated δ values indicate a shift toward a more viscous, less recoverable behavior, confirming that guayule resin introduced greater molecular mobility and energy dissipation into the binder system. This observation aligns with earlier MSCR findings, where ARG binders consistently showed higher non-recoverable creep compliance (J_{nr}) and lower percent recovery (R%), suggesting reduced elastic response under stress and increased susceptibility to permanent deformation.

From a viscoelastic perspective, these results confirm that guayule resin, while contributing to processability and sustainability, also modifies the binder’s stiffness–elasticity balance by softening the CRM-induced network. This trade-off is particularly important for pavement designs targeting high-temperature rutting resistance. Therefore, the incorporation of guayule resin should be optimized not only for environmental and constructability benefits but also in the context of mechanical performance, particularly in applications subject to severe loading and elevated temperatures.

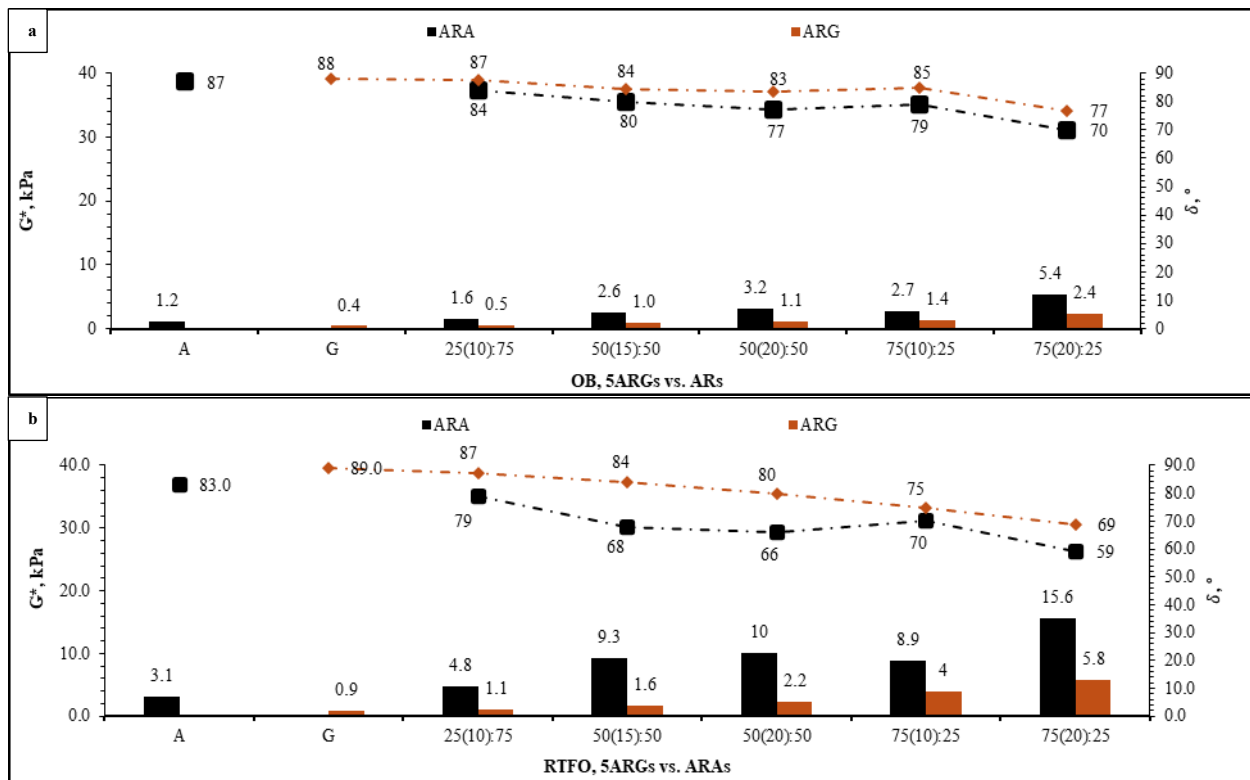


Figure 10. Basic Rheological properties of ARG and ARA binders at 64°C: (a) $|G^*|$ & δ (OB) and (b) $|G^*|$ & δ (RTFO)

Rutting Parameter $|G^|/\sin(\delta)$: Superpave High-Temperature Performance*

Building upon the stiffness–elasticity characteristics discussed previously (**Figure 10**), **Figure 11** presents the Superpave rutting parameter ($|G^*|/\sin(\delta)$) for both ARG and ARA binder systems at 64°C in their OB and RTFO-aged conditions. This parameter, derived from linear viscoelastic theory, combines binder stiffness ($|G^*|$) with phase angle (δ) to estimate rutting resistance under oscillatory shear. Higher values indicate greater resistance to permanent deformation, especially in traffic conditions dominated by elastic response.

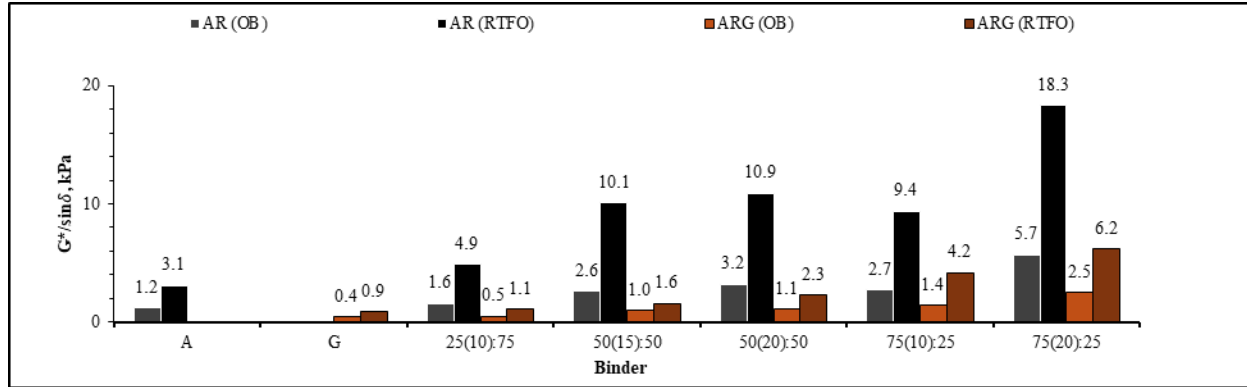


Figure 11. Superpave Rutting Resistance at 64°C: ARGs vs. ARs

Across both aging states, ARA binders consistently demonstrated higher $|G^*|/\sin(\delta)$ values than their ARG counterparts, suggesting enhanced rutting resistance from a stiffness-dominated perspective. For instance, after RTFO aging, AR75(20):25 reached 18.3 kPa, compared to only 6.2 kPa for the corresponding ARG binder. While this difference reflects the inherently stiffer and more elastic nature of ARA systems, driven by effective CRM–asphalt interactions within the aromatic-rich matrix [44], it should not be interpreted in isolation. A relative comparison reveals that the $|G^*|/\sin(\delta)$ value increased by a factor of 3.2 in AR75(20):25 and 2.5 in ARG75(20):25 from OB to RTFO. This indicates that guayule-based systems, although starting from a softer baseline, still respond substantially to oxidative aging, reinforcing their potential adaptability in engineered binder systems.

The consistently lower $|G^*|/\sin(\delta)$ values in ARG binders reflect the softening influence of guayule resin, which introduces more viscous character and attenuates the formation of a highly elastic CRM network. As seen in prior $|G^*|$ and δ results (**Figure 10**), guayule resin shifted the viscoelastic profile toward higher δ values and lower stiffness, confirming a reduction in elastic rigidity. However, this trade-off may be beneficial under specific conditions: ARG binders exhibited more stable δ values and lower aging indices ($|G^*|/\sin(\delta)$ RTFO/OB ratios), suggesting enhanced tolerance to oxidative hardening and better stress accommodation under variable environmental and loading conditions.

These trends become more meaningful when interpreted in conjunction with MSCR test results. ARA binders showed lower non-recoverable creep compliance (J_{nr}) and higher elastic recovery (%R), indicating stronger resistance to deformation under repeated traffic-like shear loading. In contrast, ARG binders had higher J_{nr} and lower %R values, consistent with their lower $|G^*|/\sin(\delta)$ and more viscous response. This reinforces the understanding that guayule resin modifies binder structure not only by reducing stiffness, but also by diluting the CRM-induced elasticity, leading to a softer viscoelastic framework that may limit rutting resistance under heavy or sustained loading.

Nevertheless, it is essential to acknowledge that $|G^*|/\sin(\delta)$ alone does not fully characterize binder performance, particularly for non-traditional or bio-modified systems like ARG. While ARA systems excel in stiffness-driven rutting control, ARG binders offered improved flexibility and aging resilience, attributes that may be advantageous in climates with high thermal cycling or in applications where cracking resistance is prioritized. Therefore, performance evaluation of guayule-modified binders should be context-specific and supported by complementary mixture- and field-level assessments [45].

RTFO Aging Index: Resistance to Short-Term Oxidative Aging

The aging index, calculated as the ratio of $|G^*|/\sin(\delta)$ after RTFO aging to its value in the OB condition, provides insight into the oxidative hardening susceptibility of different binder systems. **Figure 12** illustrates the RTFO aging indices for both ARG and ARA binders at 64 °C, revealing a consistent trend: ARA binders

exhibited higher aging indices than their ARG counterparts, indicating more pronounced stiffening upon short-term aging. For example, AR50(20):50 demonstrates an aging index of 3.4, whereas the corresponding ARG binder shows a more moderate value of 2.1. This distinction suggests that incorporating guayule resin may confer oxidative stability, likely due to its lower concentration of oxidizable constituents and potentially inherent antioxidant properties. As a result, guayule-modified systems undergo less aggressive stiffening during thermal aging, supporting their role in performance formulations that prioritize long-term flexibility and durability. However, this improved aging resistance comes with a trade-off: ARG binders typically begin with a lower original $|G^*|$, reflecting their softer viscoelastic nature. While ARA systems showed greater stiffening after aging, they started from a higher baseline stiffness due to stronger CRM–asphalt interactions. The lower aging index of ARG systems should be seen not as a drawback, but as a strategy that promotes more consistent performance over time and lowers cracking susceptibility by limiting oxidative hardening.

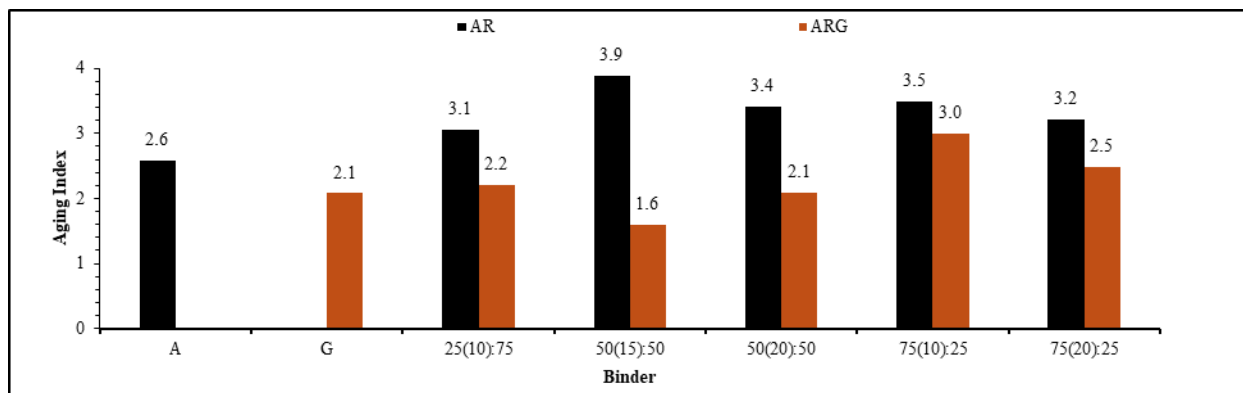


Figure 12. RTFO Aging Index at 64°C: ARGs vs. ARs

The behavior of the individual base binders supports this interpretation. The A binder (neat asphalt) has an aging index of 2.6, while the guayule resin (G) exhibits a lower value of 2.1, reinforcing that guayule ages more slowly than conventional asphalt. Although guayule resin showed a weaker initial mechanical profile, its aging response suggests a built-in resistance to thermal-oxidative transformation, which could be valuable in climate regions or pavement designs sensitive to aging-induced embrittlement. This aging trend aligns with findings from the MSCR test, particularly in terms of stress sensitivity. Specific ARG formulations, especially those with lower CRM content, exhibited more stable performance across applied stress levels, reflected by lower J_{nr_diff} values. This suggests that guayule-based systems may be less prone to stress-dependent degradation, further supporting their application in environments where thermal and load-induced fatigue are key concerns. The use of MSCR and ISF tests provided mechanistic insight into binder resilience, recovery, and stress adaptability, essential features for sustainable pavement performance. These results verify that ARG systems offer technically competitive solutions without compromising mechanical integrity..

Summary and Practical Implications for Transportation Infrastructure

This study investigated the high-temperature nonlinear viscoelastic (NLVE) response of sustainable asphalt binders formulated with guayule resin and crumb rubber modifier (CRM), focusing on their mechanical integrity, recoverability, and aging characteristics through a suite of advanced rheological tests, including MSCR and interrupted shear flow (ISF). The results revealed that guayule-based binders (ARG series), particularly those with moderate CRM content (e.g., ARG50(15):50 and ARG50(20):50), can achieve performance levels that meet or exceed those of conventional control asphalt (PG 64-22) across multiple key indicators, whereas virgin guayule resin itself could not reach that performance.

A critical and distinctive contribution of guayule-modified systems emerged from the ISF testing. Unlike conventional asphalt or fully rubberized formulations, moderate-CRM ARG binders consistently demonstrated full structural recovery after each rest period, with repeatable stress overshoots and stable plateau behavior across 5–60 s rest cycles. This indicates a rebuildable internal structure, likely attributable to guayule’s polymeric network interactions, that offers a degree of dynamic elasticity and self-reorganization under cyclic loading not observed in the control asphalt or ARA systems. Such behavior makes guayule-based binders uniquely suitable for pavement applications subjected to repeated shear-rest conditions, such as urban intersections and braking zones.

Further, while ARG binders inherently start from a softer baseline due to the PG58-11 nature of guayule resin, several formulations achieved MSCR parameters—particularly percent recovery and non-recoverable compliance (J_{nr}) that approached or matched values typically seen in more conventionally stiff systems. Notably, ARG75(20):25, despite its bio-based composition, recorded a high $\%R_{0.1}$ (54.1%) and improved J_{nr} , showcasing that guayule, when properly blended, can support CRM activation and contribute significantly to elastic recovery. This performance was achieved without relying on highly stiff, petroleum-based base binders, reinforcing guayule’s dual role as both a performance-enhancing and sustainability-driven component. CRM served a pivotal role in enabling elastic recovery and enhancing stiffness in guayule-rich systems, supporting their use under diverse pavement conditions.

Moreover, guayule-based systems displayed reduced RTFO aging indices compared to control asphalt, indicating lower susceptibility to oxidative hardening. This trait, combined with its favorable recoverability and flow stability, underscores the promise of guayule resin in flexible pavement design, particularly in environments subject to thermal cycling or load-induced fatigue.

In summary, this research established that guayule resin is more than an eco-friendly additive—it is a functional, rheologically active component that imparts unique structural recovery dynamics and aging resilience to asphalt binders. When engineered with appropriate CRM content, guayule-based systems offer an effective path toward high-performance, sustainable asphalt technologies that can meet modern pavement demands without compromising mechanical integrity. In addition to its environmental merits, guayule resin enhanced recoverability, reduced oxidative hardening, and enabled structural rebuilding, attributed that made it a compelling alternative for pavement systems exposed to thermal and mechanical fatigue.

CONCLUSIONS

This study advanced the understanding of guayule resin–modified asphalt binders by incorporating advanced rheological testing protocols, namely, Multiple Stress Creep Recovery (MSCR) and Interrupted Shear Flow (ISF), to evaluate performance beyond conventional stiffness and elasticity indicators. While $|G^*|/\sin(\delta)$ offered valuable insight into binder rigidity, it proved insufficient on its own to capture the stress-dependent recovery and structural rebuilding behaviors critical for modified binder systems under field-representative loading.

MSCR results demonstrated that select ARG binders—particularly ARG50(15):50 and ARG75(20):25—achieved nonlinear viscoelastic recovery and stress tolerance levels comparable to or exceeding those of the control asphalt binder, despite containing significant bio-derived content. ISF testing further confirmed the ability of these formulations to rebuild internal structure under cyclic shear–rest conditions, as evidenced by consistent stress overshoot behavior and minimal flow decay. This recoverable microstructural resilience, not observed in asphalt rubber binders, underscored guayule’s potential to support network integrity under repeated loading.

The integration of MSCR and ISF testing revealed that guayule resin serves not only as a sustainable additive but also actively contributes to binder resilience, oxidation stability, and nonlinear performance under traffic-like conditions. These findings position guayule-based binder systems—with optimized CRM content—as technically viable and environmentally progressive alternatives for high-temperature flexible pavement applications. More broadly, the study underscores the importance of advanced, mechanism-based evaluations in the performance specification of future sustainable asphalt technologies. Guayule’s ability to improve recovery, limit oxidative aging, and integrate effectively with CRM positions it as a high-potential, bio-based component in modern binder design.

To support implementation, further studies should expand rheological and mixture-level evaluation of guayule–CRM systems and validate their performance under field conditions. This will help translate laboratory findings into practical specifications and support the transportation community’s shift toward durable, sustainable asphalt technologies.

AUTHOR CONTRIBUTIONS

The authors confirm their contribution to the paper as follows: study conception and design: A. Hemida, M. Abdelrahman; data collection, analysis and interpretation of results: A. Hemida; draft manuscript preparation: A. Hemida and A. S. El-Ashwah. All authors reviewed the results and approved the final version of the manuscript.

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