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EVALUATION OF GUAYULE RESIN AS AN INNOVATIVE BIO-BASED ASPHALT ALTERNATIVE IN MIX PERFORMANCE

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ABSTRACT

Literature revealed the potential of using guayule resin for asphalt cement replacement from the binder's perspective. However, monitoring guayule resin through binder-aggregate mixture could disclose its performance through field. In this study, designated binders were employed to investigate the applicability of such an innovative replacer through mixture, which were neat asphalt and guayule-based binders (neat guayule, asphalt-rubber-guayule, guayule-rubber binders). Consecutively, field-simulated lab mixtures were prepared to investigate the major distresses. Moisture damage, rutting, fatigue cracking, and thermal cracking resistances were investigated using the modified Lottman (TSR) test, rut test by asphalt pavement analyzer (APA), semi-circular bending (SCB) test, and disk-shaped compact tension (DCT) test, respectively. Additionally, the Hamburg wheel-tracking (HWT) test was employed to evaluate moisture susceptibility and rutting resistance. Outcomes revealed that the neat guayule was susceptible to moisture damage at a 7% air content (V_a) when the TSR test was employed. In contrast, all investigated mixtures yielded perfect performances against moisture susceptibility under the HWT test. Guayule-based mixtures perfectly resisted rutting, as analyzed by the rut test and HWT test. Generally, changing parameters (e.g., V_a, rubber addition, and partial asphalt replacement by guayule and rubber) enhanced the guayule-based mixture resistance to rutting and moisture damage resulting in acceptable performances. Guayule-based mixture had a high fracture toughness at low temperatures, hence fatigue fracture resistance at intermediate temperatures. Neat guayule mixture with or without rubber addition did not entirely resist thermal fracture. However, partial asphalt replacement by guayule and rubber resisted the thermal fracture to a great extent.

Keywords: Bio-Binder, Guayule Resin, HMA, Mix Performance, Superpave Mix Design, Sustainability

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1. INTRODUCTION

Guayule resin is inevitably extracted during the production of guayule natural rubber (current target guayule product) in the same proportion (1:1), at the very least (*1*). Guayule resin is a by-product (leftover) (*1; 2*), but it has the potential to become an asphalt cement alternative (*3-9*). Hemida and Abdelrahman (*3-9*) investigated its applicability from the perspective of binder performance. It is an asphalt-like material (*8*). It provides rheological properties comparable to asphalt cement, thermo-plastic visco-elastic, and susceptible to temperature change (viscoelastic at room temperature, liquid at high temperature, and solid at low temperature) (*8*). Therefore, the guayule-based binder could represent an innovative approach to replace asphalt cement for a sustainable, flexible pavement industry (*8; 9*).

Several bio-oils were investigated in literature for asphalt binder replacement (*10*). The distinction of guayule resin other than several bio-oils is that it is ready to be directly employed in the massive, flexible pavement industry. The only process needed is a simple heat treatment to ensure no moisture and/or low molecular weight components inside before further processing (*4*). Nevertheless, guayule resin is not identical to asphalt cement (*7*). Guayule resin offered a lower viscosity for the construction process than conventional asphalt at the same performance grade high temperature (PG HT) grade(*9*). This could indicate plant energy consumption and environmental emissions savings (*9*). According to the literature, the as-received guayule resin could provide a PG HT of up to 58℃ (*7*) and a performance grade low temperature (PG LT) of -16℃ (*9*). These performance grades are not widely applicable in several locations (*7*). Therefore, guayule modifications potentially enhance the guayule-based binder's performance. For instance, it was proved that the crumb rubber modifier (CRM or rubber) improved the PG HT of the neat guayule (*7; 9*). However, CRM did not enhance the PG LT (*9*). Nothing, so far, enhanced the guayule-based binder's PG LT except for the partial asphalt replacement by guayule (*7; 9*). Rejuvenators can be used in the future to enhance the PG LT of the guayule-based binders. In other words, asphalt cement performance was enhanced via many years of research, and guayule has the potential to achieve a wide tolerance of high- and low-temperature performance grades by research as well.

It is known from the literature that CRM enhanced the conventional asphalt's rheological properties to a great extent (*11*). However, the harmony of the asphalt-rubber blend was found higher than that of the guayule-rubber blend (*9*). For instance, at high temperatures, it was found that 20% of CRM (by weight of asphalt) enhanced the neat asphalt by about three grades, while the same CRM concentration enhanced the neat guayule by only one grade (*9*). Therefore, applying a blend of asphalt, rubber, and guayule resin, as a partial asphalt cement replacement could be highly recommended to present a competitive performance to regular asphalts at high-, intermediate-, and low-temperature performances (*3; 7; 9*).

Major distresses of asphalt mixture were evaluated by several methods, including permanent deformation (rutting), load-associated cracking, thermal cracking, and moisture susceptibility (*12*). Superpave mix design recognized the modified Lottman test to assess the mixture's moisture susceptibility, stripping (*13*). Even though many researchers employed this standard method, there is a belief that it is not highly correlated to the field performance (*14*). Therefore, the Hamburg wheel tracking (HWT) test is commonly used to provide a high correlation with the actual performance and provide the rutting assessment by measuring rut depth (*14; 15*). On the other hand, researchers used fracture mechanics to evaluate fracture cracking at intermediate (Fatigue cracking) and low (thermal cracking) temperatures (*16*). Simply, fracture energy represents the energy required to form a new fracture surface (*16*). This kind of testing is based on initiating a notch to control the crack propagation direction (*17*). Two of the most commonly used fracture resistance tests to evaluate the mixture cracking are the semi-circular bending (SCB) and disk-shaped compact tension (DCT) tests. The SCB test is used to assess the fatigue fracture resistance (*17*). The DCT test is one of the most recommended tests to evaluate the mixture thermal cracking (*18; 19*), which has an adequate cracking path due to its relatively long cracking path compared to other tests (e.g., SCB test at low temperatures) (*16; 18*).

Based on the literature, the guayule-based binder can not be fully assessed without investigating its behavior in the binder-aggregate mixture. Therefore, this study aimed to evaluate the behavior of previously established guayule-based binders in the mixture by carrying out commonly used asphalt mixture tests. Five designated mixtures were selected to address the asphalt replacement by guayule resin (called guayule as well in this study) and CRM. The tests involved assessments of the major distresses encountering flexible pavement: moisture susceptibility, rutting resistance, fatigue cracking resistance, and thermal cracking resistance. Modified Lottman test was used to evaluate moisture susceptibility. Rut test using asphalt pavement analyzer (APA) was employed to assess rutting resistance. Additionally, the HWT test was used to evaluate both moisture susceptibility and rutting resistance at the same time. Fatigue cracking and thermal cracking resistances were evaluated by the fracture energy mechanism employing the

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SCB and DCT tests. Therefore, the applicability of guayule resin in the flexible pavement mixture could be initiated. Subsequently, guayule-based mixtures' enhancements will be founded in the future.

2. MATERIALS AND METHODS

2.1. Materials

2.1.1 Binders' Preparation

From previous studies by the same research group (*3; 4; 6; 7*), designated binders were selected to evaluate the performance of the guayule-based binder through the mixture. The designated binders were established based on the following material sources: asphalt from Philips 66 Company, IL, CRM from Liberty Tire Recycling, and guayule resin from Bridgestone Corp. The CRM was received in different gradation, but the CRM30-40 (passed mesh #30 and retained on mesh #40) was employed according to the US standard system (*20; 21*). The designation implied five binders, which were neat asphalt (PG64-22), neat guayule (PG58-10), one asphalt-rubber-guayule blend (ARG(75-20)), and two guayule-rubber blends (GR(12.5) and GR(25)). The ARG(75-20) binder included 75% asphalt rubber (20% rubber by weight of asphalt) plus 25% guayule. The GR(12.5) blend included a guayule-rubber binder (12.5% rubber by weight of blend). The GR(25) binder included a guayule-rubber blend (25% rubber by weight of blend). The Superpave performance grade of each designated binder was reported in **Table 1**. Blending was carried out using a high shear mixer, temperature controller, and a heating mantle. Before blending, guayule was heat-treated at a 160°C blending temperature with a 600-rpm rotational speed until no bubbling or foaming visually appeared to ensure that no moisture or low molecular weight components were involved. Regarding the preparation of the asphalt-rubber-guayule blend, first, asphalt was mixed with rubber for 40 min at 190°C and 3000 rpm. Then, guayule was added, and the entire blend was mixed for 1 h at 160°C and 600 rpm. Regarding the preparation of the guayule-rubber blend, the same process of the asphalt-rubber-guayule blend was followed except for the asphalt portion, which was replaced by guayule.

Table 1. Binders' Data

Binder	Code	Proportions			Superpave Performance Grade[1] [°C]		
		A%	G%	CRM%	PG HT[2]	PG IT[3]	PG LT[4]
Neat Asphalt	A	100			64	20	-22
Neat Guayule	G		100		58	31	-10
Asphalt-Rubber-Guayule Blend	ARG(75-20)	62.5	25	12.5	70	22	-16
Guayule-Rubber Blend (1)	GR(12.5)		87.5	12.5	58	34	-10
Guayule-Rubber Blend (2)	GR(25)		75	25	64	34	-4

[1]Superpave performance grades were listed based on the dynamic shear rheometer (DSR) and bending beam rheometer (BBR) measurements. [2]PG HT: Performance grade high temperature; [3]PG IT: Performance grade intermediate temperature; [4]PG LT: Performance grade low temperature.

2.1.2 Aggregate Gradation

According to AASHTO M 323 (*22*) and MoDOT 403 (*23*), a job mix formula was followed to investigate the guayule-based binders in the field-simulated lab mixtures. Five individual aggregates were employed to make an accepted aggregate blend with the MoDOT's Superpave mix design procedure. The five aggregates' types and proportions were as follows: three Potosi Dolomite Formation (29% of 9/16″ clean, 29% of 3/8″ clean, and 15% of screenings), 25% of manufactured sand (crushed gravel), and 2% of mineral fillers (-#200). The aggregate blend had a 12.5-mm (1/2") nominal maximum aggregate size (NMAS), called SP125 in the Superpave mix design procedure. **Figure 1** illustrates the combined aggregate gradation, compared to the Superpave and MoDOT specification limits: Superpave upper and lower specification limits (USL and LSL, respectively), and MoDOT 403 SP125 USL and LSL.

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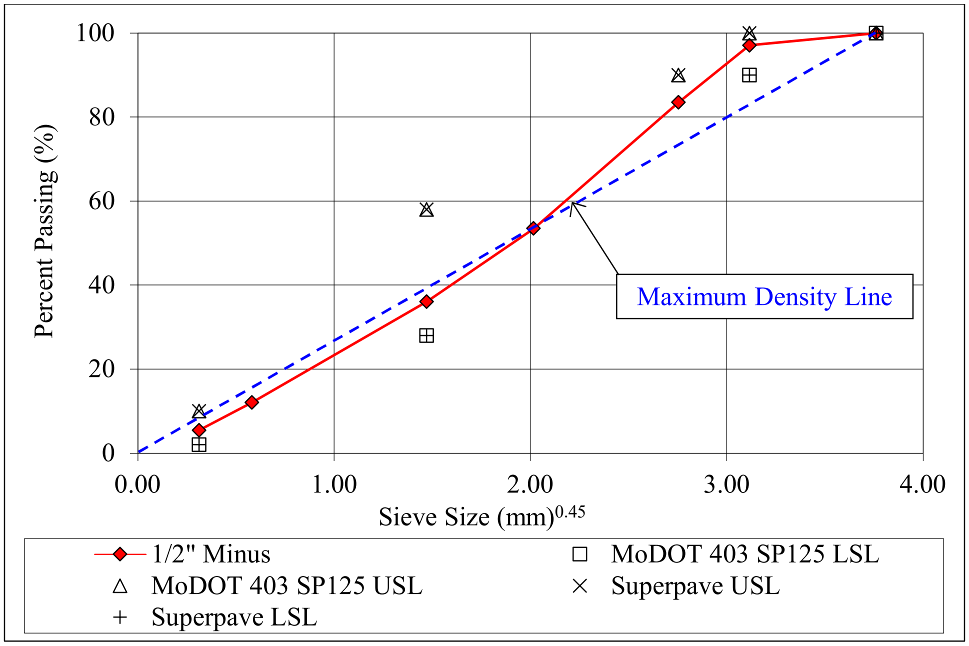
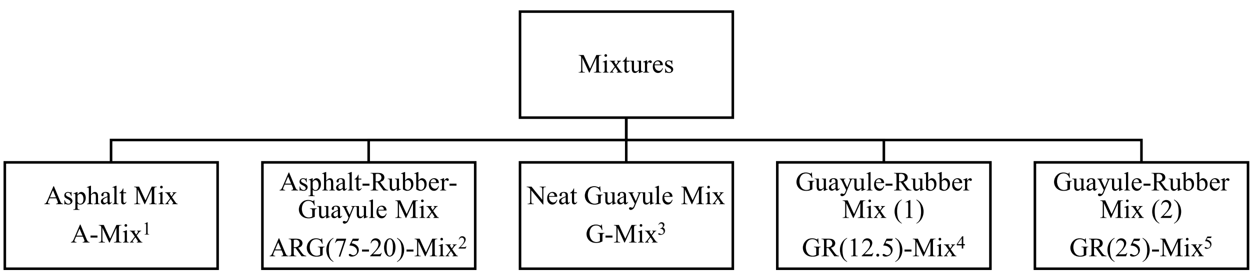


Figure 1. Aggregate gradation.

2.1.3 Investigated Mixes

Figure 2 shows the flowchart of the selected five mixtures for investigations. The five mixtures were determined to address the effect of the binder replacements on the mixture performance. Mixtures' IDs were defined in the footnote underneath the flowchart. The neat asphalt mixture (A-Mix) was selected to be compared with the asphalt-rubber-guayule mixture (ARG(75-20)-Mix). On the other hand, guayule was investigated in mixtures as an entire asphalt cement alternative. A neat guayule mixture (G-Mix) was assessed based on its performance limitations. **Figure 3** shows the neat guayule mix as a loose mix and compacted mix. Two guayule-rubber mixtures were selected to analyze the performance changes by rubber addition in two different concentrations (12.5% and 25%, by weight of blend), named GR(12.5)-Mix and GR(25)-Mix, respectively.



[1] A-Mix: included neat asphalt cement (PG64-22).
[2] ARG(75-20)-Mix: included 75% asphalt rubber (20% rubber by weight of asphalt) plus 25% guayule.
[3] G-Mix: included neat guayule (i.e., 100% asphalt cement replacer).
[4] GR(12.5)-Mix: included guayule-rubber blend (12.5% rubber by weight of blend).
[5] GR(25)-Mix: included guayule-rubber blend (25% rubber by weight of blend).

Figure 2. Investigated mixtures.

Figure 3. Neat guayule mixture (G-Mix): loose and compacted.

To determine the air content (V_a) of each compacted mixture, the theoretical maximum specific gravity (G_{mm}) and bulk specific gravity (G_{mb}) were determined, according to AASHTO T 209 (*24*) and AASHTO T 166 (*25*), respectively. **Table 2** illustrates the G_{mm} value of each designated mixture.

Table 2. G_{mm} Values of Designated Mixtures

Designated Mix	G_{mm} [Unitless]
A-Mix	2.526
G-Mix	2.540
ARG(75-20)-Mix	2.549
GR(12.5)-Mix	2.550
GR(25)-Mix	2.546

3. METHODS

3.1. Mixing and Compaction Temperatures

A rotational viscometer was used to determine the mixing and compaction temperature ranges for the five mixtures. **Table 3** demonstrates the accepted temperature ranges, according to viscosity values of 0.170±0.020 Pa.s and 0.280±0.030 Pa.s, respectively (*26*). Additionally, the applied mixing and compaction temperatures are stated between the two brackets.

Table 3. Mixing and Compaction Temperatures

Designated Mix	Temperature Range [°C]	
	Mixing	Compaction
A-Mix	152-158 (155)*	135-143 (143)
G-Mix	141-146 (143)	121-127 (127)
ARG(75-20)-Mix	176-181 (176)	164-169 (165)
GR(12.5%)-Mix	146-153 (150)	132-138 (135)
GR(25%)-Mix	172-178 (176)	159-165 (165)

*The number between the two brackets indicates the selected temperature for mixing or compaction.

3.2. Mixing and Compaction Processes

The individual aggregates were oven-dried until a constant mass was achieved, indicating no further moisture inside, then combined. The mixing temperature was used for mixing pans, mixing paddles, combined aggregate, and asphalt binder. AASHTO R 30 (*27*) was followed for aging process. A mechanical mixer was used to prepare the loose mixtures at the optimum asphalt content, P_b = 4.7%. A Superpave gyratory compactor was used to prepare the Superpave mix cores, according to AASHTO T 312 (*28*), in which G_{mb} was determined based on each V_a requirement.

3.3. Mixture Tests

Mixture tests were selected to address the major distresses (rutting, fatigue cracking, and thermal cracking) in addition to moisture susceptibility evaluations. **Figure 4** illustrates the applied mixture tests in this study, associated with the followed standards/specifications. Superpave recognized the modified Lottman test to assess moisture susceptibility. Therefore, it could be an initial indicator to predict the applicability of the guayule-based mixtures against moisture damage (stripping). Even though many researchers employed this standard method of moisture sensitivity assessment, there is a belief that it is not highly correlated to the field performance (*14*). The HWT test is not a standard method recognized by Superpave. Nevertheless, the HWT test could be a representative tool to evaluate the moisture susceptibility besides the associated rutting potential. The rut test by APA is a common technique directly relevant to the rutting resistance assessment used in this study. To predict cracking potential in the designated mixes at intermediate and low temperatures, the concept of fracture energy was utilized. The SCB test was used to evaluate the fatigue cracking resistance at the intermediate temperature. The DCT test was used to assess the thermal cracking resistance at low temperatures. The DCT test is more reliable than the SCB tests regarding the thermal cracking assessment because of the long crack path that provides adequate time to analyze the crack propagation at low temperatures (*16*). However, the validity of the DCT test applications was only offered at low temperatures up to +10℃ (*29*).

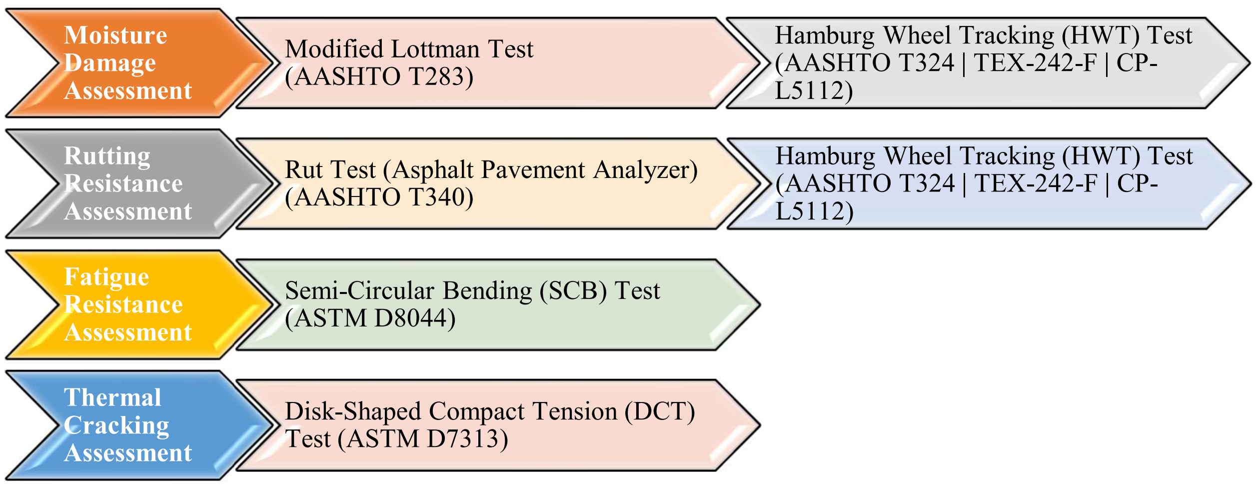


Figure 4. Flowchart of mixture tests.

3.3.1. Moisture Susceptibility

The Modified Lottman test is included in the Superpave Mix Design procedures (*13*). In this study, AASHTO T 283 (*30*) was followed to investigate the moisture susceptibility of all five designated mixtures. Six-core specimens were made with a V_a of 6.5–7.5% and divided into two sets (dry and wet). The first set involved three dry cores (control), and the other set involved three wet cores (conditioned), which were exposed to vacuum saturation of 70–80% with water. The wet set was exposed to one freezing cycle for 16 h at -18℃ and one thaw cycle in a 60℃ water bath for 24 h. Afterward, both sets were conditioned in a 25℃ water bath for 2 h before testing. The indirect tensile strength was measured (using a load rate of 2 inches/minute), and averages were calculated to acquire the tensile strength ratio (TSR), according to **Equation 1** (*13*). Many agencies recommended the TSR to be no less than 70% (*13*).

$$TSR = \frac{Indirect\ Tensile\ Strength\ of\ Conditioned\ set}{Indirect\ Tensile\ Strength\ of\ control\ set} \quad (1)$$

3.3.2. Rutting Susceptibility

The mixture's rutting susceptibility was investigated using the APA. The rut test was carried out according to AASHTO T 340 (*31*). A 64℃ testing temperature was selected to compare stiffer mixtures (A-Mix, ARG(75-20)-Mix, and GR(25)-Mix). A 58℃ testing temperature was selected to compare softer mixtures (GR(12.5)-Mix and G-Mix). Stiffer and softer mixtures were identified according to the binders' high-temperature performances (rutting parameter). The core samples — involving a V_a of 6.5–7.5% — were installed in the molds and set in the APA chamber for 6 h before testing to ensure the isothermal condition. Eight thousand passes were applied based on 60 cycles per minute at the test temperature. **Figure 5** shows technical steps from the rut test procedures conducted by the APA.

Figure 5. Technical steps from the rut test procedures.

3.3.3. HWT

The HWT test was employed to investigate moisture susceptibility and rutting resistance of the designated mixtures using the modified APA. Moisture damage could occur for many reasons, such as cohesion failure induced by moisture (*14*). AASHTO T 324 reported that the testing temperature is specified by the agency (*32*). Colorado Department of Transportation (CDOT) Test Criteria (CP-L5112) specified the test temperature based on the binder's PG HT (i.e., 40℃ for PG52, 45℃ for PG58, 50℃ for PG64, and 55℃ for PG70 or higher) (*33*). The lab-compacted specimen is required to contain a 6±2% V_a. CDOT defined the failure when the rut depth went beyond 4 mm at 10,000 passes (*34*). Texas Department of Transportation (TxDOT), TEX-242-F, specified a constant temperature of 50±1℃ regardless of the binder grade (*35*). The lab-compacted specimen is required to contain a 7±1% V_a. The test outcome is considered a failure if the rut depth becomes greater than 12.5 mm (*33*). TxDOT identified the minimum number of passes according to the binder grade (i.e., 10,000 passes for PG64 or lower, 15,000 passes for PG70, and 20,000 passes for PG76 or higher) (*33*). In this study, the HWT test was mainly carried out with monitoring the outcomes according to the two specifications. **Figure 6** shows technical steps from the HWT test procedures.

Figure 6. Technical steps from the HWT test procedures.

3.3.4. Fatigue Fracture Resistance

The concept of the SCB test was introduced by Mull et al. (2002) (*36*) to evaluate asphalt mixtures involving CRM. Afterward, this concept was utilized to investigate fatigue fracture resistance of asphalt mixtures in Louisiana (*37; 38*). In this study, the five designated mixtures were analyzed using the SCB test at 25℃. This test is highly recommended by Louisiana Transportation Research Center (*39*) and found suitable by several researchers to estimate the mixture's fatigue fracture resistance (*37*). The 25℃ test temperature was used in literature to address the fatigue

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cracking resistance as a representative intermediate test temperature (*37-40*). At a rate of 0.5 mm/min, the three-point bending test was conducted, according to ASTM D8044 (*15*), in which the specimen represented a half-disk with a notch cut depth parallel to the loading and vertical axis. The specimen was loaded monotonically up to fracture failure occurrence (*15; 37*). The applied contact load was 0.045 kN. The target V_a was 6.5–7.5% (*15*). Louisiana Department of Transportation and Development (LADOTD) recommended three sets of specimens with notch depths of 25, 32, and 38 mm (*15; 37-40*). Technical steps from the SCB test procedures are shown in **Figure 7**. The critical strain energy release rate (J-integral or J_c) end result parameter, illustrated in **Equation 2**, was utilized to evaluate the fatigue fracture resistance. The J-integral is a function of the rate of change of strain energy per notch depth (dU/da) (*37*). Several studies revealed that softer binders might reduce fracture resistance at intermediate temperatures (*37; 39; 41*).

Figure 7. Technical steps from the SCB test procedures.

$$J_c = -\left(\frac{1}{b}\right) \frac{dU}{da} \tag{2}$$

Where:

J_c	critical strain energy release rate, kJ/m^2
b	specimen thickness, mm
a	notch depth, mm
U	strain energy to failure (area under the load-displacement curve to peak load), N.mm
dU/da	change of strain energy with notch depth (strain energy-notch depth slope)

LADOTD recommended a minimum of 0.45 kJ/m^2 to indicate a threshold acceptance of a mixture's resistance to fatigue fracture cracking (*39*). Studies reported that the higher the J_c value, the higher the fracture resistance to fatigue cracking (*42*).

3.3.5. Low-Temperature Cracking

The DCT test was selected to investigate the fracture energy (G_f) at low temperatures, illustrated in **Equation 3** (*29*), to evaluate the thermal fracture properties of the designated mixtures. Technical steps from the DCT test procedures are shown in **Figure 8**. The target V_a was 6.5–7.5%. Literature reported that the quality of the DCT results goes down when temperatures go higher than +10°C (*29*). The better DCT fracture energy outcomes were associated with softer binders at low temperatures (*43; 44*). Based on the literature, the test temperature was selected to be 10℃ greater than the PG LT (*29; 45*). Besides carrying out measurements at 10℃ greater than the PG LT, measurements at different low temperatures were taken to investigate further the effect of low-temperature change on some designated mixtures. ASTM D7313 (*29*) was followed to conduct this test. A constant crack mouth opening displacement (CMOD) rate of 0.017 mm/sec (approximately 1 mm/min) controlled the DCT test (*29; 45*). The seating (contact) and post-peak loads were applied 0.1 kN. The specimen geometry was set with respect to ASTM D73-13 (*29*). Specimens were

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temperature-conditioned in the DCT instrument's environmental chamber for 2 h to ensure the isothermal condition (*29*).

$$G_f = \frac{Area}{B(W-a)} \tag{3}$$

Where:

G_f	fracture energy, J/m^2
Area	Area under load-CMOD curve up to 100 N, N.m
B	specimen thickness, m
W-a	ligament length, m

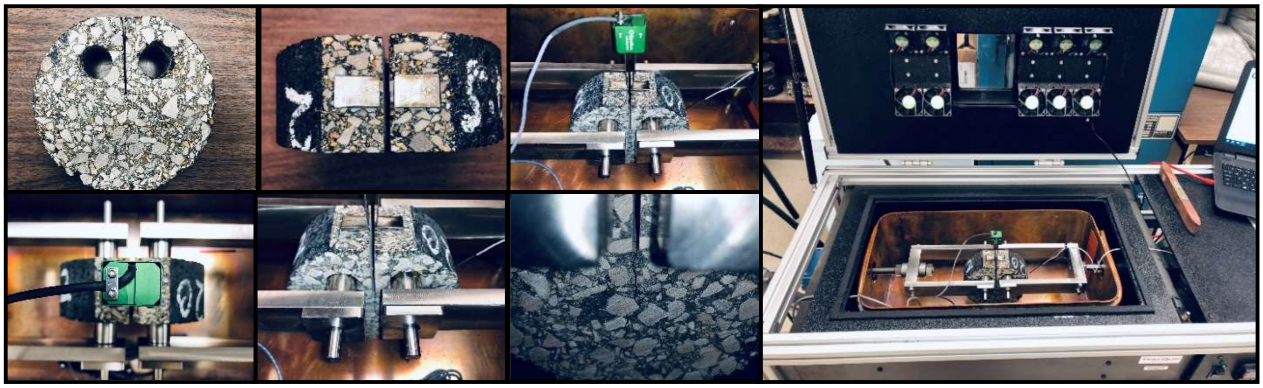

Figure 8. Technical steps from the DCT test procedures.

Studies reported a threshold G_f value of 400 J/m^2 to indicate an acceptable threshold value of fracture energy to resist low-temperature cracking (*45*).

4. RESULTS AND DISCUSSION

4.1. Moisture Susceptibility

Figure 9 illustrates the TSR results. G-Mix resulted in a dramatically low TSR (40%). In contrast, A-Mix resulted in an 82% TSR at the same mixture parameters, indicating potentially significant moisture damage to the G-Mix at a 7% V_a level. Using guayule as a 100% asphalt replacer in the mixture will require mix parameters' change according to the standard (TSR) test criteria, such as P_b, V_a, and/or anti-stripping agent addition parameters. For instance, changing V_a to 3.5% changed the TSR of G-Mix to 71%, indicating a significant moisture-resisting enhancement to the neat guayule mix. Additionally, the CRM concentration gradually increased the moisture damage resistance. For instance, adding 20% CRM to guayule in GR(25)-mix changed the TSR from 40% to 73% at the same V_a (7%). The ARG(75-20)-Mix provided enhanced TSR values at 7% V_a and 3.5% Va, 86% and 96%, respectively.

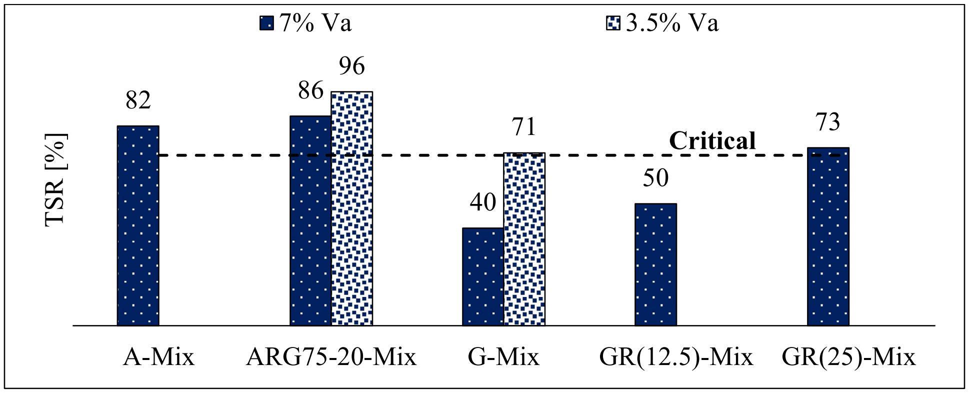

Figure 9. Moisture susceptibility (TSR) results from the modified Lottman test.

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4.2. Rutting Susceptibility

The PG HTs of A-Mix, ARG(75-20)-Mix, and GR(25)-Mix were 64°C, 70°C, and 64°C, respectively. Nevertheless, to compare the novel binders' behaviors in the mixture to the A-Mix, rut depths were addressed at a 64°C test temperature. On the other hand, G-Mix and GR(12.5)-Mix were tested at a 58°C test temperature since they had the same PG HT (58°C).

As shown in **Figure 10a**, the results showed that the rut depth trend was more minor with GR(25)-Mix, followed by ARG(75-20)-Mix, then A-Mix. Compared to the measured binder performance at high temperatures, the rut test revealed that the GR(25)-Mix had significantly low rut depth (0.4 mm), indicating that the GR(25)-mix could provide a high enhancement to the rutting resistance more than what was expected according to the Superpave criteria of the binder performance. As expected, ARG(75-20)-Mix presented a better performance than that of A-Mix since ARG(75-20) had a 70°C PG HT, and A-Mix had a 64°C PG HT. Ultimately, the three mixtures provided undoubtedly excellent resistance to rutting.

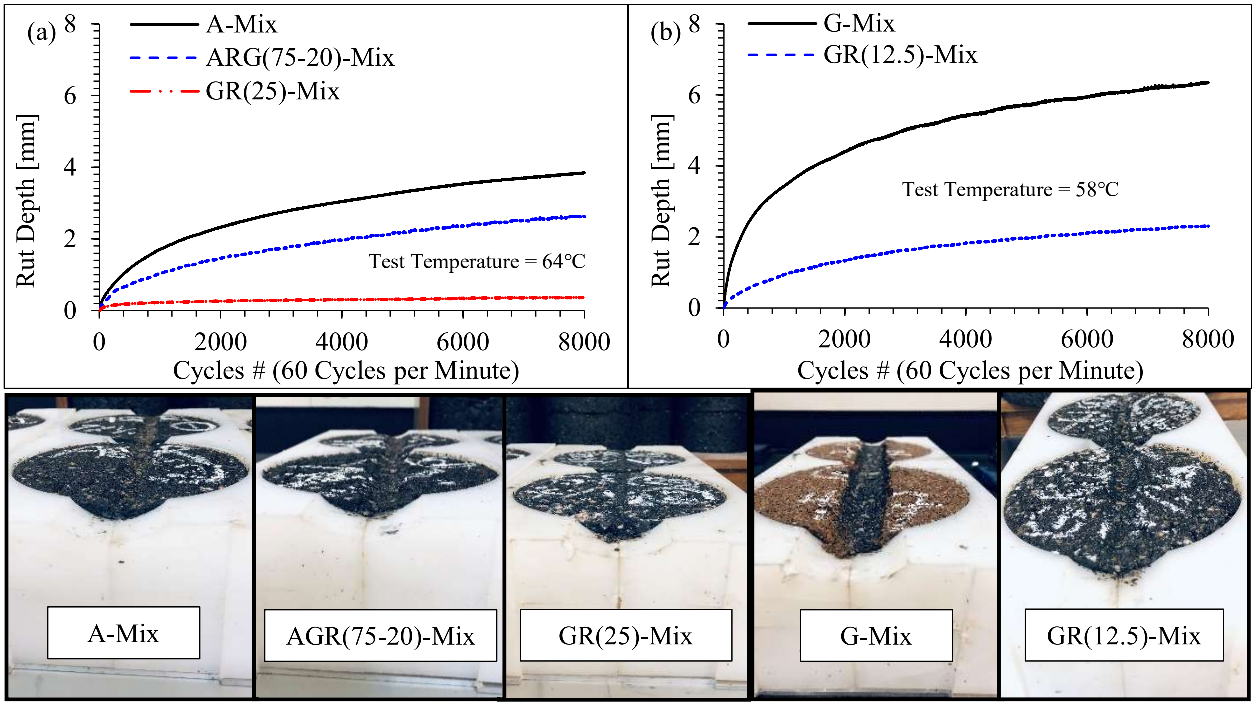

Figure 10. Rut test results: (a) A-Mix, ARG(75-20)-Mix, and GR(25)-Mix at a 64°C test temperature, and (b) G-Mix and GR(12.5)-Mix at a 58°C test temperature; associated with the specimens' appearances after testing.

As shown in **Figure 10b**, G-Mix presented an accepted rut depth at a 58°C test temperature, which was compatible with the binder's rheological performance. The maximum rut depth associated here reached up to 6.35 mm. At the same test temperature (58°C), GR(12.5)-Mix presented an enhanced rutting resistance (rut depth = 2.3 mm) compared to G-Mix, indicating the enhancement provided by the CRM to the neat guayule at high temperatures. In all studied cases, the rut depth went much lower than the limits recommended by many DOTs (*46*).

4.3. HWT

Figure 11 illustrates the designated mixtures' performances using the HWT test. Most mixtures were tested here at two different air contents: 4% and 6%. Generally, all designated mixtures behaved perfectly despite their exposure to severe environmental and load parameters. In addition, the stripping inflection point was not reached for all designated mixtures, indicating no moisture damage (stripping) potential at this level of testing. Because of the binder performance outcomes mentioned in Section 2.1.1, G-Mix and GR(12.5)-Mix were tested at a 45°C test temperature, whereas A-Mix, ARG(75-20)-Mix, and GR(25)-Mix were tested at 50°C test temperature. G-HMA exhibited an outstanding performance after 10,000 passes (in agreement with CPL 5112) and after the extended 20,000 passes, as shown in **Figure 11a**. The rut depth decreased when modifying guayule by CRM in GR(12.5)-Mix. As expected, the

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evolution of V_a slightly increased the rut depth, as observed from the difference between GR(12.5)-Mix [4%V_a] and GR(12.5)-Mix [6%V_a].

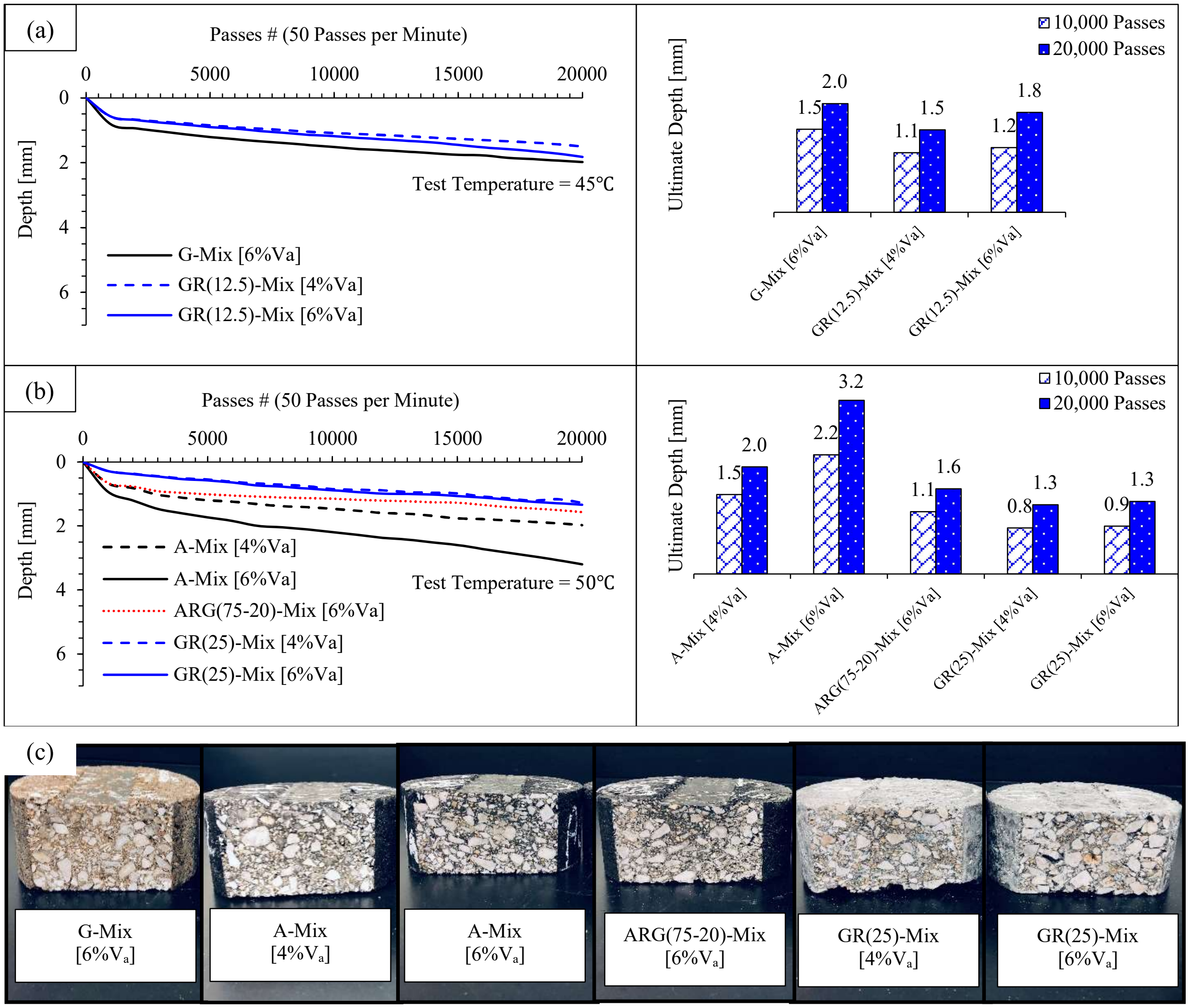

Figure 11. HWT test results: (a) G-Mix and GR(12.5)-Mix at a 45℃ test temperature, (b) A-Mix, ARG(75-20)-Mix and GR(25)-Mix at 50°C test temperature, and (c) some specimens' appearances after 20,000 passes.

At a 64°C test temperature, A-mix, ARG(75-20)-Mix, and GR(25)-Mix were HWT tested. These mixtures were not exposed to stripping at this level of testing since their stripping inflection points were not reached. Results revealed that all of these mixtures passed the HWT test with respect to all checked standards/specifications after either 10,000 or 20,000 passes, as shown in **Figure 11b**. **Figure 11c** shows the appearance of some core specimens after HWT testing. The V_a parameter slightly changed the rut depth when comparing A-Mix [4%V_a] to A-Mix [6%V_a]. However, this slight change was not noticed for GR(25)-Mix. GR(25)-Mix at the two levels of air contents (4% and 6%) almost resulted in the same rut depth at either 10,000 or 20,000 passes. ARG(75-20)-mix presented an enhanced moisture resistance compared to A-Mix. Therefore, the three designated mixtures' performances against moisture damage could be descendingly ranked as follows: GR(25)-Mix, ARG(75-20)-Mix, then A-Mix.

4.4. Mixture Performance at Intermediate Temperature

Figure 12 illustrates the strain energy vs. notch depth charts of the designated mixtures in linear regression to acquire the resultant slope (dU/da), and J_c values. The steeper the slope, the tougher the material (*40*). **Figure 12a** demonstrates comparable A-Mix and ARG(75-20)-Mix slope values. Further, it demonstrates comparable G-Mix, GR(12.5)-Mix, and GR(25)-Mix slope values. In **Figure 12b**, the J_c of A-Mix resulted in 0.46 kJ/m^2. This value could be considered the control J_c value to assess the novel guayule-based mixtures. The asphalt-rubber-guayule mixture (ARG(75-20)-Mix) yielded a J_c value of 0.48 kJ/m^2. This value indicated the predicted applicability or harmony among asphalt, rubber, and guayule in the mix performance against fatigue fracture resistance. Additionally, this application explains the excessive compensation of conventional asphalt performance by rubber and guayule at this level of testing and material parameters. The neat guayule mix (G-Mix) yielded a J_c value of 0.66 kJ/m^2, contrasted with the binder's intermediate-temperature performance assessment, but in agreement with the SCB testing background (*37; 39; 41*). The neat asphalt binder presented a better performance at intermediate temperatures (i.e., the neat asphalt possessed a lower PG IT) than the neat guayule. The 0.66-kJ/m^2 J_c value demonstrates G-Mix's high fatigue fracture resistance compared to A-Mix, which was better than expected. The guayule-rubber mixtures resulted in comparable mix performances to the G-Mix against fatigue fracture, 0.65 kJ/m^2 for GR(12.5)-Mix and 0.69 kJ/m^2 for GR(25)-Mix. This could be an initial indication of the effect of rubber concentration increase/decrease on the fatigue fracture resistance of guayule-rubber mixtures.

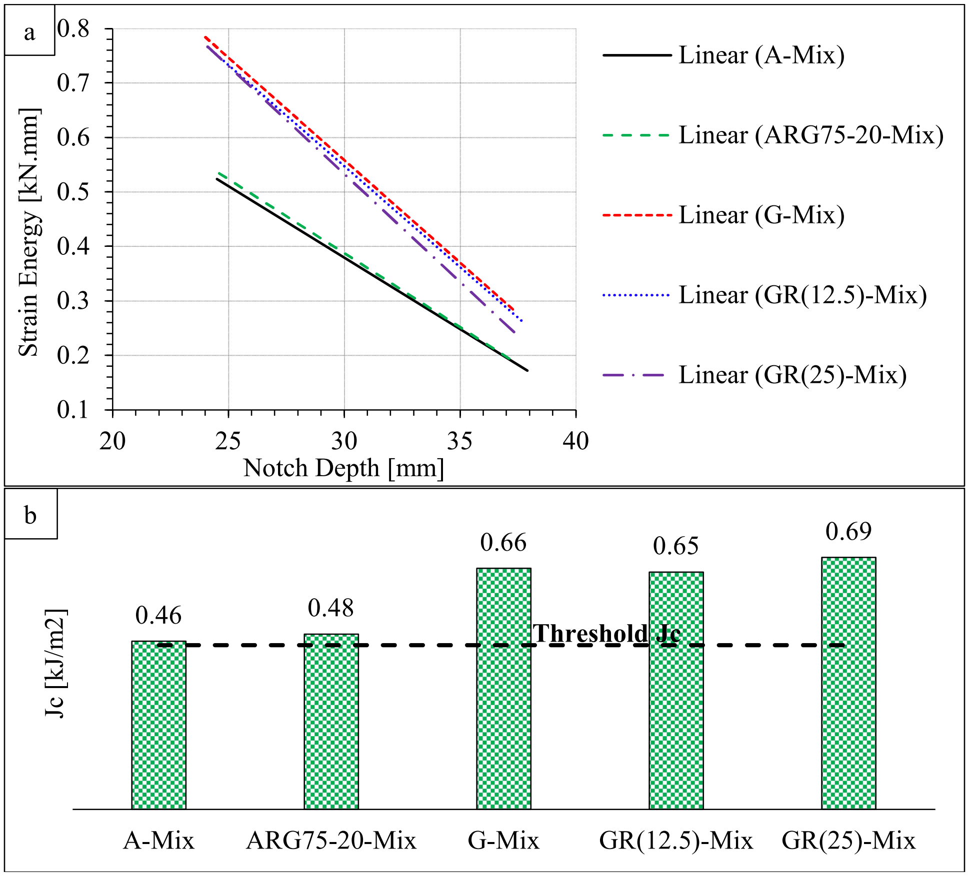

Figure 12. SCB test results: (a) rate of change of strain energy per notch depth, strain energy-notch depth slope (dU/da), and (b) Critical strain energy release rate (J_c).

The positive impact of the guayule-based binders in mixtures regarding the fracture resistance reflected the great fracture toughness of partial or entire asphalt cement replacement by guayule. Guayule presented a better performance in the mixture than the neat asphalt. The reason for that might be the ignorance of fracture toughness assessment for binders. The guayule-based mixture offered a steeper absolute value of the slope (dU/da), i.e., a higher rate of change of strain energy per noth depth, which indicated a tougher material at this level of testing (*40*).

4.5. Mixture Performance at Low Temperature

Figure 13a shows an example of a G-Mix specimen before and after the DCT test. **Figure 13b** illustrates the fracture energy (G_f) of the designated mixtures at 10°C greater than the PG LT. Some mixtures were exposed to other low temperatures to monitor the differences in their behaviors. Results showed that the A-Mix yielded a G_f value of 429 J/m^2 at a -12℃ test temperature, which passed the threshold value initiated through literature (*45*). The neat guayule mixture or its modification by rubber would not improve the low-temperature cracking resistance. According to the Superpave criteria, binder investigations revealed the destructive behaviors of guayule binders at low temperatures, but not to that extent shown by the mixture outcomes. The threshold G_f value (400 J/m^2) was not reached for any of G-Mix, GR(12.5)-Mix, or GR(25)-Mix at 6℃ or 0℃ test temperatures. This could indicate the difficulty of using guayule (as a 100% asphalt replacer) with or without CRM, at this level of material and interaction parameters, to resist the potential thermal cracking, indicating a worse low-temperature performance than what was predicted from the binder investigations.

On the other hand, ARG(75-20)-Mix, which had a PG LT of -16℃, remarkably provided an excellent fracture resistance at the corresponding test temperature (-6℃), 591 J/m^2. That was why the same mixture was exposed to a -12℃ to monitor its performance at that low test temperature. The results of ARG(75-20)-Mix positively ended with a G_f value of 409 J/m^2, indicating a potentially accepted mixture at a PG LT of -22℃.

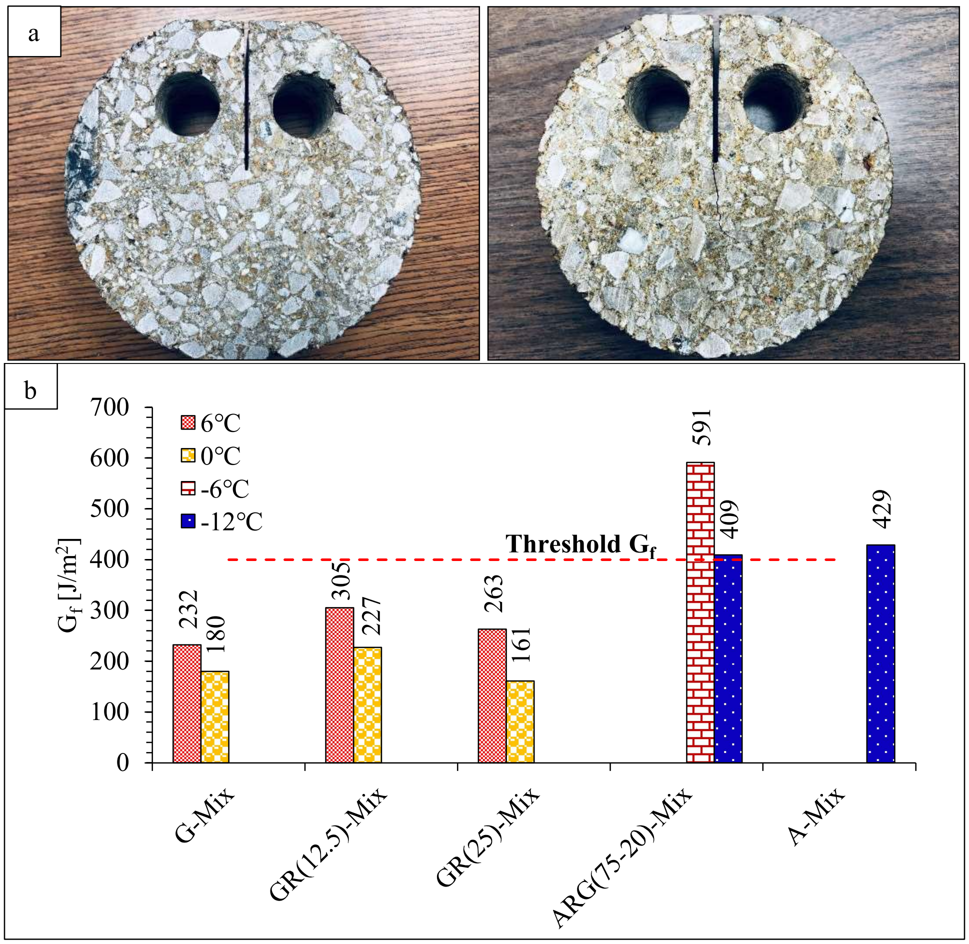

Figure 13. DCT test: (a) example of a G-Mix before and after fracture, and (b) fracture energy (G_f) results.

4.6. Summary

In **Table 4,** a summary of the major data acquired through this study was reported to summarize the input parameters (test temperature and air content) and end result parameters accomplished (TSR, rut depth (by APA), HWT rut depth, J_c, and G_f).

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5. CONCLUSIONS

This study provided an evaluation of the innovative guayule bio-based mixtures against major distresses from the perspective of mixture: moisture damage, rutting, fatigue cracking, and thermal cracking. The following observations were made:

1. Guayule was worse than asphalt in resisting moisture damage through the standard (TSR) test. In contrast, guayule-based mixtures presented a high resistance to moisture damage evaluated by the HWT test, found more reliable to address the field performance.
2. Neat guayule mixture had a high resistance to rutting at its high-temperature performance grade. Guayule modification using CRM and partial asphalt replacement by guayule and rubber in ARG(75-20) enhanced the rutting resistance. This was compatible with the binder performance evaluated by the Superpave criteria.
3. Changing parameters (e.g., V_a, rubber addition, and partial asphalt replacement by guayule and rubber) enhanced the guayule-based mixture's resistance to rutting and moisture damage resulting in acceptable performances by TSR, Rut, and HWT tests.
4. The positive impact of the guayule-based binders in mixtures regarding the fracture resistance at the intermediate temperature reflected the great fracture toughness of partial or entire asphalt replacement by guayule. Guayule offered better performance in the mixture than the neat asphalt due to the unavailability of the fracture toughness criterion in binder evaluation by the Superpave criteria. Compared to neat asphalt mixture, guayule-based mixture presented a higher rate of change of strain energy per noth depth, which indicated a tougher material.
5. Guayule (with or without CRM addition) did not offer the desired performance at low temperatures. This could indicate the difficulty of using guayule (as a 100% asphalt replacer) to resist the potential thermal cracking, indicating a worse low-temperature performance than what was expected from the binder evaluation by the Superpave criteria. However, partial asphalt replacement by guayule and CRM resisted the thermal fracture to a great extent.
6. Future works are recommended to enhance the performance of the guayule-based mixtures, particularly at low temperatures. Rejuvenators' addition is a potential material parameter that could significantly improve such a mixture's performance at low temperatures and intermediate temperatures.

Table 4. Summary Table

Mixture	Parameter(s)	TSR Test		Rut Test (APA)	HWT Test				SCB Test	DCT Test			
A-Mix	T [°C]	25		64	50				25	6	0	-6	-12
	V_a [%]	7±0.5%	N/A	7±0.5%	4%		6%		7±0.5%	7±0.5%			
	Outcomes	TSR: 82%		RD: 3.8 mm	10,000 Passes	20,000 Passes	10,000 Passes	20,000 Passes	J_c: 0.46 J/m^2	N/A	N/A	N/A	G_f: 429 J/m^2
					RD: 1.5 mm	RD: 2 mm	RD: 2.2 mm	RD: 3.2 mm					
G-Mix	T [°C]	25		58	45				25	6	0	-6	-12
	V_a [%]	7±0.5%	3.50 %	7±0.5%	4%		6%		7±0.5%	7±0.5%			
	Outcomes	TSR: 40%	TSR: 71 %	RD: 6.3 mm	10,000 Passes	20,000 Passes	10,000 Passes	20,000 Passes	J_c: 0.66 J/m^2	G_f: 232 J/m^2	G_f: 180 J/m^2	N/A	N/A
					N/A	N/A	RD: 1.5 mm	RD: 2.0 mm					
ARG(75-20)-Mix	T [°C]	25		64	50				25	6	0	-6	-12
	V_a [%]	7±0.5%	3.50 %	7±0.5%	4%		6%		7±0.5%	7±0.5%			
	Outcomes	TSR: 86%	TSR: 96 %	RD: 2.6 mm	10,000 Passes	20,000 Passes	10,000 Passes	20,000 Passes	J_c: 0.48 J/m^2	N/A	N/A	Gf: 591 J/m^2	Gf: 409 J/m^2
					N/A	N/A	RD: 1.1 mm	RD: 1.6 mm					
GR(12.5)-Mix	T [°C]	25		58	45				25	6	0	-6	-12
	V_a [%]	7±0.5%	N/A	7±0.5%	4%		6%		7±0.5%	7±0.5%			
	Outcomes	TSR: 50%		RD: 2.3 mm	10,000 Passes	20,000 Passes	10,000 Passes	20,000 Passes	J_c: 0.65 J/m^2	G_f: 305 J/m^2	G_f: 227 J/m^2	N/A	N/A
					RD: 1.1 mm	RD: 1.5 mm	RD: 1.2 mm	RD: 1.8 mm					
GR(25)-Mix	T [°C]	25		64	50				25	6	0	-6	-12
	V_a [%]	7±0.5%	N/A	7±0.5%	4%		6%		7±0.5%	7±0.5%			
	Outcomes	TSR: 73%		RD: 0.4 mm	10,000 Passes	20,000 Passes	10,000 Passes	20,000 Passes	J_c: 0.69 J/m^2	G_f: 263 J/m^2	G_f: 161 J/m^2	N/A	N/A
					RD: 0.8 mm	RD: 1.3 mm	RD: 0.9 mm	RD: 1.3 mm					

*N/A: not available; RD: Rut Depth; T: Test Temperature; V_a: Air Content.

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AUTHOR CONTRIBUTIONS

The authors confirm contribution to the paper as follows: study conception and design: Hemida, Abdelrahman; data collection and lab testing: Hemida; analysis and interpretation of results: Hemida, Abdelrahman; writing – original draft: Hemida; writing – review & editing: Hemida, Abdelrahman. All authors reviewed the findings and approved the final version of the manuscript.

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