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Use of Innovative Recycling Agents for Improving the Sustainability and Durability of Asphalt Pavement

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13. Abstract
The use of reclaimed asphalt pavement (RAP) as a partial replacement for virgin aggregates and asphalt binders has received considerable attention in recent years due to its economic and environmental benefits. These benefits include a potential reduction in asphalt mixture cost and the promotion of sustainability. However, many state Departments of Transportation are cautious in adopting high RAP content (>25%) into their asphalt mixture designs because of cracking and durability issues resulting from the aged RAP binder. This study considered two approaches to mitigate concerns regarding hardened and oxidized aged RAP binders: (1) reagent catalyst (Lewis acid type—iron chloride, $FeCl_3$) to modify the asphalt binder's chemical composition and disrupt the associated molecules formed in the aged RAP binder; and (2) the application of petroleum-based and

bio-derived recycling agents (RAs) to improve the cracking resistance of asphalt mixtures containing aged RAP binder (>25%).

The objective of this study was to assess the effectiveness of the use of $FeCl_3$ and petroleum-based and bio-derived RAs in mitigating cracking in high RAP asphalt mixtures. The additives evaluated included $FeCl_3$ and six RAs (petroleum-derived aromatic oil, soy oil, and four types of tall-oil-derived phytosterol). The dosage rate for $FeCl_3$ was optimized at 2% of RAP asphalt binder content. Further, a binder blending tool was developed to optimize the dosage rate of RAs to obtain conventional target asphalt binders. Three 12.5 mm NMAS asphalt mixtures containing three different levels of RAP were evaluated: 0, 30, and 50%.

The asphalt binder experiment included chemical characterization using Fourier Transform Infrared Spectroscopy (FTIR) and saturates / aromatics / resins / asphaltenes (SARA) analysis, as well as rheological evaluation using Superpave performance grading. The asphalt mixture experiment included tests for linear viscoelastic properties (dynamic modulus), fracture and fatigue resistance (semi-circular bend), rutting resistance and moisture susceptibility (Hamburg wheel tracking), low-temperature cracking resistance (thermal stress-restrained specimen tensile strength), and durability (Cantabro abrasion). The engineering performance acceptance criteria of the studied asphalt mixtures were evaluated within the BMD framework specified by DOTD.

Results obtained from the SCB and other tests showed that incorporating $FeCl_3$ or RAs in asphalt mixtures with high RAP content can enhance cracking resistance. Further, the use of these additives did not adversely impact the asphalt mixtures' permanent deformation. Thus, asphalt mixtures containing high RAP content and $FeCl_3$ or RAs exhibited similar performance to conventional mixtures. It is recommended that high RAP asphalt mixtures be implemented in field sections while incorporating the RAs utilized in this study for further validation, with field performance to be considered in provisional DOTD specifications.

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Use of Innovative Recycling Agents for Improving the Sustainability and Durability of Asphalt Pavement

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Abstract

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The asphalt binder experiment included chemical characterization using Fourier Transform Infrared Spectroscopy (FTIR) and saturates / aromatics / resins / asphaltenes (SARA) analysis, as well as rheological evaluation using Superpave performance grading. The asphalt mixture experiment included tests for linear viscoelastic properties (dynamic modulus), fracture and fatigue resistance (semi-circular bend), rutting resistance and moisture susceptibility (Hamburg wheel tracking), low-temperature cracking resistance (thermal stress-restrained specimen tensile strength), and durability (Cantabro abrasion). The engineering performance acceptance criteria of the studied asphalt mixtures were evaluated within the BMD framework specified by DOTD.

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Thus, asphalt mixtures containing high RAP content and $FeCl_3$ or RAs exhibited similar performance to conventional mixtures. It is recommended that high RAP asphalt mixtures be implemented in field sections while incorporating the RAs utilized in this study for further validation, with field performance to be considered in provisional DOTD specifications.

Acknowledgements

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Implementation Statement

Findings from this research will enhance the sustainability and durability of asphalt pavements by utilizing innovative recycling agents (RAs) to mitigate the cracking issues associated with high RAP content in asphalt mixtures. This study explored two approaches to address the challenges posed by hardened and oxidized RAP binders. The first approach involved using a reagent catalyst (iron chloride, $FeCl_3$) to modify the chemical composition of the asphalt binder and disrupt molecular associations in aged RAP binder. The second approach utilized six types of RAs, including petroleum-derived aromatic oil, soy oil, and four types of tall-oil-derived phytosterol (industrial by-product, intermediate, purified, and fatty-acid-based), to improve the cracking resistance of asphalt mixtures containing high RAP content. Specific areas of implementation include:

Design

- Revise DOTD Table 502-6 of the *Specifications for Roads and Bridges* to allow RAP content up to 50% for eligible mixes, excluding airport pavements and SMA.
- Follow the Louisiana BMD framework for the design of asphalt mixtures containing up to 50% RAP, including the conduction of LWT and SCB tests.
- Use the binder blending tool developed in this research to confirm that the base binder + RA meets the target binder PG grade.

Production Verification

- Periodically confirm that the RAP quality remains consistent with the material used in the design phase.
- Periodically conduct LWT and SCB tests, with the SCB test performed on long-term aged mixtures (loose mixture, 135°C for 6 hrs).

Field Test Sections

- Construct field test sections to evaluate performance under actual traffic and environmental conditions.
- Monitor field performance over time to verify the research findings.

The use of this recommended implementation plan will improve the durability and sustainability of Louisiana's asphalt pavements.

Table of Contents

List of Tables

List of Figures

Introduction

Asphalt mixtures, a key material in flexible pavement construction, are facing increased costs, prompting pavement agencies to explore cost-effective alternatives without sacrificing performance [1, 2, 3]. One such sustainable method is the use of recycled materials to compensate for part of the virgin materials. Using reclaimed asphalt pavement (RAP), which is generated from the milling of old pavements, can potentially protect the environment and conserve resources; however, incorporating RAP into virgin asphalt can be challenging. The aged asphalt binder from RAP materials (subsequently referred to as RAP binder) generally cannot be used as a direct substitute for virgin asphalt binder because of its aging during service life and the resulting changes in its chemical composition and properties [4, 5, 6, 7].

The asphalt binder's chemical composition has a delicate balance of polar to non-polar molecules, small to large molecules, and aromatic to paraffinic compounds. When there is an imbalance in this composition, incompatibility between the components may occur, resulting in poor engineering performance. Such an imbalance can originate from the aging of the asphalt binder. The blending process of RAP binder with virgin asphalt binder is not yet well understood due to the complex chemical composition of asphalt binder, which makes it difficult to analyze and predict the characteristics of the blend.

RAP materials have been utilized with virgin aggregates and asphalt binders in Louisiana and across the U.S. for decades [4]. However, there are many concerns related to the cracking performance when a high RAP level is used in asphalt mixtures. High RAP content was defined as 25-50% or higher according to National Cooperative Highway Research Program (NCHRP) Report 752 [6]. This is due to the fact that aged RAP binder is unable to be utilized as a direct replacement for virgin asphalt binder. Consequently, RAP binders frequently include molecules with large molecular weights that raise issues with durability and cracking [7]. Therefore, increased RAP content in asphalt mixtures could negatively impact the cracking performance of asphalt pavements, which would ultimately raise the cost of pavement maintenance and repairs [4].

State Departments of Transportation and contractors are continually looking for better ways to incorporate greater amounts of recycled materials into asphalt mixtures without adversely affecting pavement performance, specifically cracking performance. Many methods are available for rejuvenating aged binders from RAP materials. Recent studies presented using softer virgin binders (e.g., warm mix asphalt additives) or increasing the virgin asphalt binder content as possible solutions to produce a higher recycled binder ratio (RBR, defined as the percentage of recycled asphalt binder to the total asphalt binder content of an asphalt mixture). The concept of using additives in asphalt mixtures to improve short- and long-term performance is not new. Specifically, the use of additives to restore the rheology of aged binders has been studied extensively [1]. Since 2012, research has been conducted to find remedies to disband the associated asphaltenes in RAP binders.

Literature Review

RAs can be categorized as softening or rejuvenating agents. Rejuvenating agents, primarily organic oils (e.g., BituTech RAP, SonneWarmix RJT, SonneWarmix RJ, Cyclogen, organic oils, etc.), are rich in maltenes that disperse the aged asphaltenes and rejuvenate the asphalt binder's chemical and physical characteristics [1]. Softening agents (e.g., lube stock, asphalt flux, slurry oils, etc.) primarily decrease the aged asphalt binder's viscosity to yield suitable workability for mixing high RAP in asphalt mixtures; their role is predominantly focused on altering the physical characteristics of the RAP binder [1]. It has been highlighted that the effectiveness of any rejuvenators should be evaluated by assessing their ability to reverse the chemical composition of aged binders and recover it to that of virgin asphalt, or to decrease the asphaltene content and increase the aromatic content in aged asphalt binders [2].

Table 1 compiles a range of RAs from existing literature, detailing their results and impacts. Findings show discrepancies relative to the effectiveness of RAs on cracking performance. Specifically, a DOTD study reported that the addition of RAs resulted in a reduction in cracking resistance compared to similar asphalt mixtures with no RAs [1].

Table 1. Literature summary of RAs' effectiveness on RAP binder and RAP asphalt mixtures

RA Type and Components	Tests Performed	Findings
Resin produced from cashew nut shells Vegetable oil, naphthenic oils	Softening point, penetration, DSR, FTIR	RAs were effective in decreasing the PG of RAP asphalt binder [3] RAs expedited the aging process when added to virgin asphalt binder
Aromatic extract polar Waste vegetable oil non-polar	DSR, BBR, AFM, SARA	RAs were effective in decreasing high and low PG of RAP asphalt binder [3]
Hydrogreen Road Science rejuvenator Arizona chemical	HWT, OT	Enhanced cracking resistance Concerns with rutting resistance [4]
Waste vegetable grease Organic oil Aromatic extract	DSR, BBR, RV, RTFO, HWT, IDT, CAST	All enhanced rutting, moisture, and fatigue cracking resistance Only the Aromatic type enhanced low-temperature cracking resistance [5]
Waste vegetable oil		Enhanced rutting, fatigue cracking Concerns with moisture susceptibility
Distilled tall oil		Enhanced rutting and fatigue resistance Concerns with low-temperature cracking resistance
Waste engine oil		Enhanced rutting resistance and decreased cracking resistance [5]
BituTech SonneWarmix RJT SonneWarmix RJ	DSR, BBR, LAS, MSCR, OT, TSRST	Enhanced intermediate- and low-temperature cracking resistance, especially BituTech Concerns were related to rutting and moisture susceptibility [6]
Hydrogreen Cyclogen-L Asphalt Flux Soft binder PG 58-28	DSR, BBR, LAS, MSCR, HWT, SCB, TSRST	Additives showed negative effects on the intermediate and low-temperature properties of the asphalt mixtures, failed to improve cracking resistance [1]

Note: RA: Recycling Agent, DSR: dynamic shear rheometer test; FTIR: Fourier Transform Infrared Spectroscopy; BBR: bending beam rheometer; AFM: atomic force microscopy analysis; SARA: saturates, asphaltenes, resins, and aromatics analysis; IDT: Superpave indirect tension; TSR: tensile strength ratio; APA: asphalt pavement analyzer; HWT: Hamburg wheel tracking test; SCB: Semi-circular Bending test; OT: overlay tester; CAST: coaxial shear test; LAS: linear amplitude sweep test. I-FIT: Illinois flexibility index test; IDEAL-CT: IDEAL cracking tolerance test; S-VECD: simplified viscoelastic continuum damage test, IDT; indirect tensile creep compliance and strength tests.

Chemical Recycling Agent

Catalysts are commonly associated with clean energy and green chemistry. They have the potential to be a new generation of asphalt binder rejuvenators. Lewis acid catalyst (iron chloride—$FeCl_3$) is known to catalyze the conversion of coal to liquid product [7]. Using a catalyst as a rejuvenator is a promising new approach for restoring the properties of aged RAP binders, as it disrupts the associated molecules formed during aging [8]. Spivak and Balamurugan [9] found that the incorporation of 2% $FeCl_3$ by weight of the RAP binder could significantly reduce the content of asphaltenes (i.e., high molecular weight compounds) in the aged RAP binder. A 56% reduction in the carbonyl index, as measured by Fourier-Transform Infrared Spectroscopy (FTIR), was also observed [9]. The carbonyl index is a well-established parameter to assess the aging characteristics of asphalt binders [10, 11].

Petroleum-Based Recycling Agent

Reclamite base oil is a registered trademark of Ergon, Inc. [12]. Reclamite is the original Maltene Replacement Technology (MRT) to restore and preserve the durability of asphalt binders. Reclamite is a cationic petroleum emulsion manufactured from a single source of naphthenic crude stock. Naphthenic crude has excellent natural solvency, allowing it to penetrate, co-mingle, and flux with existing asphalt binders [13]. In 1960, Dr. Fritz Roster discovered the origin of asphalt pavement degeneration and separated the asphalt binder’s soluble, more reactive components, known as maltenes, when exposed to the heat used in processing asphalt-based formulations [14]. Dr. Roster collaborated with Golden Bear’s Richard White and developed a technology that restored the petroleum maltenes of aged asphalt binder, rejuvenated its pliability and resilience, and decreased asphalt binder viscosity [12].

Bio-Derived Recycling Agents

Modified soy oil, a readily available bio-renewable commodity, has been used as a rejuvenator. This bio-based alternative to petroleum-derived rejuvenators can restore binder flexibility and workability by altering its chemical composition [15]. Soy oil can react with asphaltene molecules, adding relatively long hydrocarbon chains and increasing the stability of asphaltenes within the maltene matrix [15]. Studies indicate that using soybean oil in RAP mixtures can improve fatigue resistance, lower the binder stiffness, and potentially lead to longer-lasting pavements [16]. However, challenges remain, such as optimizing dosage and addressing concerns related to potential impacts on moisture damage and rutting resistance [17].

Tall oil has been produced in the industry by products purified to different levels and used as an RA in asphalt mixtures with high RAP content. Studies have shown that tall oil is effective in improving cracking resistance, softening asphalt binder, and enhancing rutting resistance [18, 6]. In a related study, Reinke et al. [19] evaluated the use of phytosterols (sterols), obtained from seed oils and tall oil, to retard aging in asphalt mixtures. The sterol rejuvenator blend consisted of 40-60% beta-sitosterol ($C_{29}H_{50}O$), 20-40% campesterol ($C_{28}H_{48}O$), and approximately 5% stigmasterol ($C_{29}H_{48}O$). These sterols, when used as bitumen additives, effectively mitigated the negative impacts of bitumen aging and improved low-temperature cracking resistance [19]. Further, Ingevity has developed EvoFlex CA, a family of engineered additives derived from forestry products. EvoFlex CA has been proven to improve crack resistance [5, 20, 21, 22], enhance the interaction between RAP binder and virgin asphalt binder [23], and stabilize aged RAP binder, reducing the need for virgin asphalt binder [24].

Objective

The objectives of this study were to:

1. Assess the effectiveness of using a reagent catalyst in improving the engineering performance of asphalt mixtures containing high RAP content;
2. Predict the long-term performance of asphalt pavements containing mixtures with high RAP content and a Lewis acid catalyst;
3. Develop a binder blending tool to optimize the dosages of RAs and increase RBR up to 50%; and
4. Investigate the effectiveness of using new RAs, both petroleum-based and bio-derived, to mitigate cracking susceptibility in asphalt mixtures containing 30% and 50% RAP.

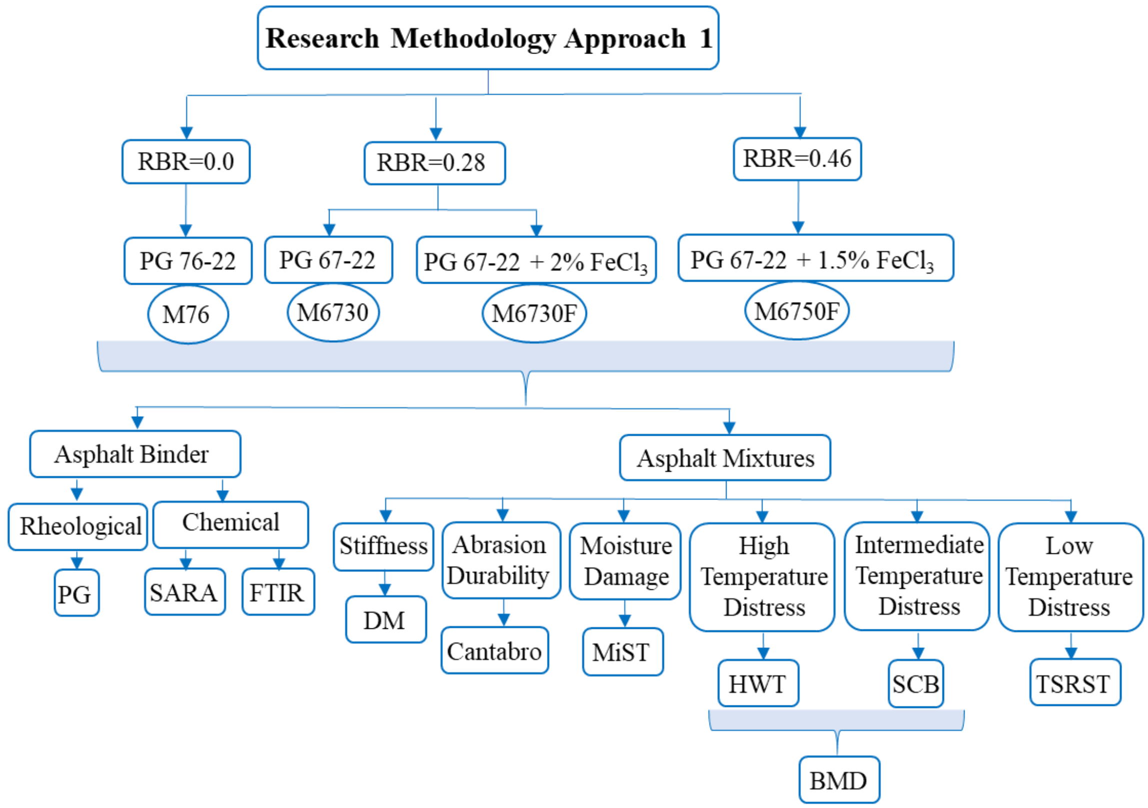
Scope

Three 12.5 mm NMAS asphalt mixtures containing three levels of RAP were evaluated: 0, 30, and 50%. These RAP levels yielded RBRs of 0, 0.28, and 0.46, respectively. The asphalt binders were extracted and recovered from the studied asphalt mixtures. This study considered two approaches to mitigate concerns of hardened and oxidized aged RAP binders: (1) reagent catalyst (Lewis acid type–$FeCl_3$), and (2) petroleum-derived aromatic oil, soy oil, and four types of tall-oil-derived phytosterol RAs. The dosage rate for $FeCl_3$ was optimized at 2% of RAP asphalt binder content based on a previous study [9]. However, a binder blending tool was developed to optimize the dosage rate of RAs to obtain conventional target asphalt binders.

Figure 1 presents the research methodology for the first approach. It includes a control mixture (M76) prepared with SBS-modified PG 76-22 asphalt binder, two mixtures prepared with 30% RAP and unmodified PG 67-22 asphalt binder (M6730 and M6730F), and one mixture (M6750F) prepared with 50% RAP and unmodified PG 67-22 asphalt binder. The M6730F and M6750F mixtures contain 2% and 1.5% $FeCl_3$ by weight of RAP binder, respectively. Figure 2 presents the research methodology for the second approach. It includes a control mixture (M76) prepared with SBS-modified PG 76-22 asphalt binder and seven mixtures prepared with 50% RAP (RBR = 0.46) and unmodified PG 67-22 asphalt binder, each incorporating a different rejuvenating agent (Mix 1 to Mix 7).

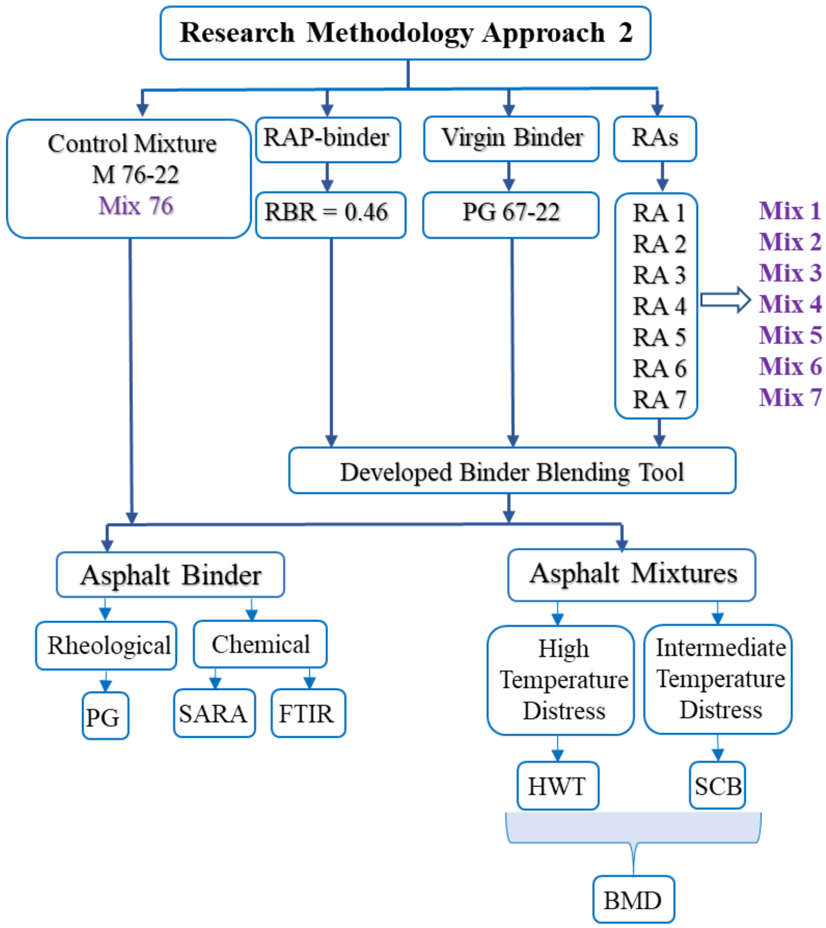
Asphalt binders were chemically analyzed using saturates / aromatics / resins / asphaltenes (SARA) analysis and Fourier Transform Infrared Spectroscopy (FTIR). Their performance grades were also determined through rheological characterization. Additionally, asphalt mixtures were subjected to a suite of mechanical tests to evaluate their performance: dynamic modulus (DM) test for linear viscoelastic properties; Semi-circular Bend (SCB) test for fracture and fatigue resistance; Hamburg wheel tracking (HWT) test for rutting resistance; Moisture-induced Stress Tester (MiST) conditioning method followed by HWT for moisture susceptibility; Thermal Stress Restrained Specimen Tests (TSRST) for low-temperature cracking resistance; and Cantabro abrasion test for durability. The engineering performance acceptance criteria of the studied asphalt mixtures was evaluated within the BMD framework specified by DOTD [25].

Figure 1. Research Methodology Approach 1



Note: RBR: Recycled binder ratio (percentage of RAP binder to total binder content); RA: Recycling agent; PG: Performance grading of extracted binder; SARA: Saturates, Aromatics, Resins, and Asphaltenes Analyses; FTIR: Fourier Transform Infrared Spectroscopy; DM: Dynamic modulus test; HWT: Hamburg wheel tracking test; MiST: Moisture induced Stress Tester; SCB: Semi-circular Bend test; TSRST: Thermal Stress Restrained Specimen Test; BMD: Balanced mixture design criteria at DOTD.

Figure 2. Research Methodology Approach 2



Note: RBR: Recycled binder ratio (percentage of RAP binder to total binder content); RA: Recycling agent; PG: Performance grading of extracted binder; SARA: Saturates, Aromatics, Resins, and Asphaltenes Analyses; HWT: Hamburg wheel tracking; SCB: Semi-circular Bend test; BMD: Balanced mixture design criteria at DOTD.

Methodology

In this study, the engineering performance of high RAP asphalt mixtures was evaluated by:

1. Determining the rheological properties of RAP binder and unmodified asphalt binder blended with RAs;
2. Developing a novel asphalt binder blending tool to optimize RAs' dosages based on the target asphalt binder of an asphalt mixture and its component materials;
3. Preparing asphalt mixtures containing high RAP levels (30% and 50%), then conducting engineering performance tests on those asphalt mixtures and further comparing the engineering performance of high RAP asphalt mixtures to DOTD conventional mixtures utilizing Louisiana's balanced mix design (BMD) framework; and
4. Investigating the effect of using new RAs on the engineering performance of asphalt mixtures containing high RAP content.

Materials

Limestone and coarse natural sand, which are both commonly used in Louisiana, were utilized in this study. Superpave asphalt mixtures were prepared using styrene-butadiene-styrene (SBS) polymer-modified asphalt binders (PG 76-22M and PG 70-22M) and unmodified PG 67-22 asphalt binder. Three RAP percentages (0, 30, and 50% by weight of the total mixture) were incorporated into the asphalt mixtures. These RAP levels yielded RBRs of 0, 0.28, and 0.46, respectively.

Recycling Agents

A reagent catalyst Lewis acid type, iron chloride ($FeCl_3$), was used in this study as a chemical RA. $FeCl_3$ was added at a dosage of 2% by weight of the RAP binder, based on the findings of Spivak and Balamurugan [9], who reported a 56% reduction in the carbonyl index, indicating reduced aging. $FeCl_3$ was first blended into the virgin asphalt binder at 325°F (163°C) using a high-shear mixer (3,000 rpm) for 30 min. Additionally, this study investigated the effectiveness of petroleum-based and bio-derived RAs in enhancing the cracking resistance of high RAP asphalt mixtures. Six types of RAs were considered: petroleum-derived aromatic oil, soy oil, and four types of tall-oil-derived phytosterol (industrial by-product, intermediate, purified, and fatty-acid-based). Table 2 and Figure 3 list the RAs considered in this report.

Table 2. Recycling agents considered in this study

RA Designation	RA	RA Category
F	Iron chloride ($FeCl_3$)	Chemical reagent catalyst
RA1	Petroleum-derived aromatic oil using maltene blend (Reclamite B)	Petroleum-based oil
RA2	Modified soy-based oil	Bio-derived oils
RA3	Modified soy-based oil + tall oil-derived phytosterol containing industrial by-product	
RA4	Modified soy-based oil + tall oil-derived phytosterol intermediate	
RA5	Modified soy-based oil + tall oil-derived purified phytosterol	
RA6	Tall oil-derived fatty acid-based oil (Evoflex CA-7)	
RA7	Tall oil-derived phytosterol containing industrial by-product	

Figure 3. Recycling agents used in this study

Binder Blending Tool

The rejuvenation process is influenced by four factors:

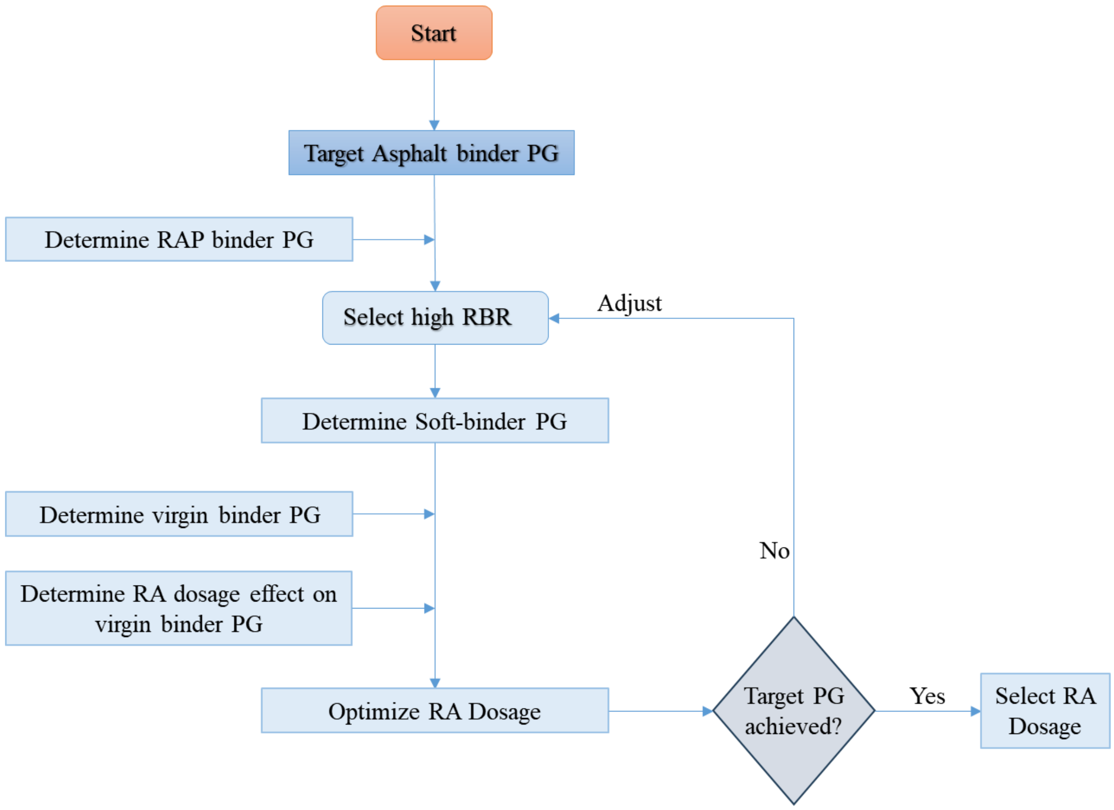
1. RAP binder performance grade (PG);
2. Soft (or rejuvenated) asphalt binder PG;
3. RBR level; and
4. Targeted asphalt binder PG.

Three of the four factors must be predetermined or preselected to use the blending tool. For example, if the RAP binder PG, target asphalt binder PG, and RBR level are known, the soft asphalt binder PG can be calculated. As illustrated below, the Excel-based blending tool was developed to estimate the target asphalt binder PG incorporated in asphalt mixtures containing RAP materials and rejuvenating agents.

Target asphalt binder → RAP asphalt binder + soft asphalt binder (RA modified)

In other words, RA dosage could be determined if the target asphalt binder PG and RBR levels were preselected. In this study, RA was first blended with a virgin asphalt binder, then introduced into the RAP binder to obtain a target asphalt binder. The virgin asphalt binder was used as a carrier for the RA to be blended with the RAP binder to obtain a specific target asphalt binder. The binder blending tool followed the framework presented in Figure 4.

Figure 4. Framework for the developed Excel-based binder blending tool



The developed procedure involved identifying the critical temperatures at which an asphalt binder is likely to exhibit specific distresses based on DOTD specifications [25] and AASHTO M 320 standard criteria [26]. By knowing the critical temperature gradings for the RAP binder, the target asphalt binder, and the selected RBR level, the critical temperatures for the soft asphalt binder were interpolated and determined.

High-Temperature Performance Grading

At high-temperature PG, rutting is a critical distress for asphalt pavements. To address this, a minimum rutting factor of $G^*/\sin(\delta)$ was established based on AASHTO M 320 criteria [26]. Minimum rutting factor values of 1.00 KPa and 2.20 KPa were set for original and short-term aged asphalt binders, respectively; see Equations 1 and 2.

$$\frac{G^*}{\sin(\delta)} \geq 1.0\ KPa \ \rightarrow \ T_c\ (high) = \left(\frac{\log(1.00) - \log(G_1)}{a}\right) + T_1 \qquad (1)$$

$$\frac{G^*}{\sin(\delta)} \geq 2.20\ KPa \rightarrow T_c\ (high) = \left(\frac{\log(2.20) - \log(G_1)}{a}\right) + T_1 \qquad (2)$$

where,
G^* = complex shear modulus;
δ = phase angle;
T_c (high) = high-critical temperature;
G_1 = value of $G^*/\sin(\delta)$ at temperature T_1;
T_1 = recommended to be the closest temperature to the criteria; and
a = slope of stiffness-temperature curve = $\Delta \log (G^*/\sin(\delta))/\Delta T$.

Intermediate-Temperature Performance Grading

At intermediate-temperature PG, intermediate-temperature cracking is a critical distress for asphalt pavements. To address this, a maximum fatigue factor of $G^*\sin(\delta)$ was established based on AASHTO M 320 criteria [26]. A maximum cracking factor of 5,000 kPa was set for long-term aged asphalt (pressure aging vessel, PAV-conditioned) binder; see Equation 3.

$$G^*.\mathrm{Sin}(\delta) \leq 5000\ KPa \rightarrow T_c\ (Intermediate) = \left(\frac{\log(5000) - \log(G_1)}{a}\right) + T_1 \qquad (3)$$

where,
Tc (intermediate) = intermediate-critical temperature;
G1 = value of $G^*. \mathrm{Sin}(\delta)$ at temperature T1; and
a= slope of stiffness-temperature curve = $\Delta\log (G^*. \mathrm{Sin}(\delta))/ \Delta T$.

Low-Temperature Performance Grading

At low-temperature PG, low-temperature cracking is a critical distress for asphalt pavements. To resist low-temperature cracking, the asphalt binder should exhibit decreased stiffness and increased relaxation. As such, a maximum creep stiffness of 300 kPa and a minimum relaxation or m-value of 0.300 were set based on AASHTO M 320 criteria [26]. Next, the critical temperature values for the maximum creep stiffness and the minimum relaxation values were determined; see Equations 4 and 5. The higher of the two critical temperature values was selected as the low-temperature PG grade. Asphalt binders were short- and long-term aged using the Rolling Thin Film Oven (RTFO) and Pressure Aging Vessel (PAV) methods, respectively. The aged binders were then graded using the Bending Beam Rheometer (BBR) test following the AASHTO T 313 standard [27].

$$T_c\ (S) = \left(\frac{log(300) - log(S_1)}{a_S}\right) + T_1 \quad (4)$$

$$T_c\ (m) = \left(\frac{0.300 - m_1}{a_m}\right) + T_1 \quad (5)$$

where,

T_c (S) = critical low-temperature obtained at stiffness;

T_c (m) = critical low-temperature obtained from *m*-value;

S_1 = the S-value at temperature T_1;

m_1 = the *m*-value at temperature T_1;

T_1 = recommended to be the closest temperature to the criteria;

a_s = slope of stiffness-temperature curve = Δ log (S)/ΔT; and

a_m = slope of *m*-value-temperature curve = Δ*m*-value/ΔT.

Target Asphalt Binder Performance Grading

Based on the primary concept of equations introduced in NCHRP Report 452 [28], Equations 6, 7, 8, and 9 were developed to compute the high-, intermediate-, and low-temperature performance gradings for the target asphalt binders, respectively.

$$log\left(\frac{G^*}{sin(\delta)}\right)_{Bended\ Binder} = RBR * log\left(\frac{G^*}{sin(\delta)}\right)_{RAP-binder} + (1 - RBR) * log\left(\frac{G^*}{sin(\delta)}\right)_{Soft\ Binder} \quad (6)$$

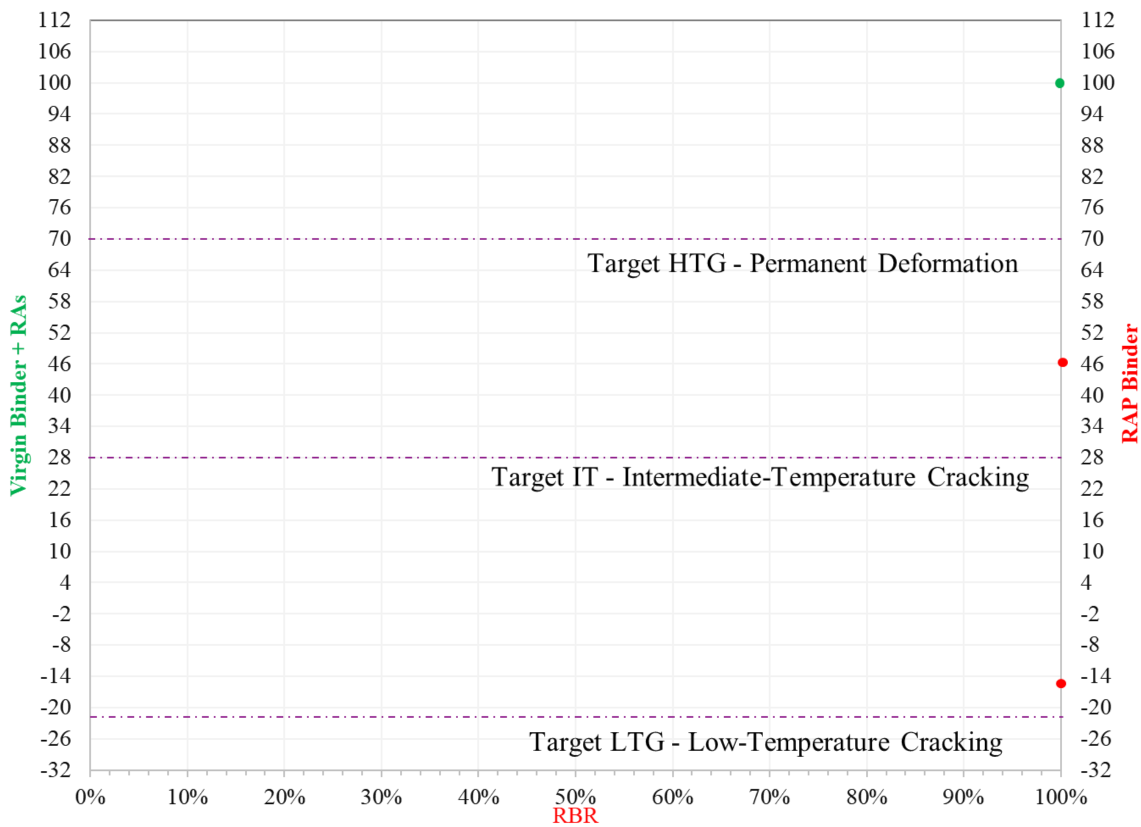
$$log(G^*.Sin\delta)_{Bended\ Binder} = RBR * log(G^*.Sin\delta)_{RAP-binder} + (1 - RBR) * log(G^*.Sin\delta)_{soft\ Binder} \quad (7)$$

$$log(S)_{Bended\ Binder} = RBR * log(S)_{RAP-binder} + (1 - RBR) * log(S)_{Soft\ \ Binder} \quad (8)$$

$$m - value_{Blended\ Binder} = RBR * m - value_{RAP-binder} + (1 - RBR) * m - value_{Soft\ Binder} \quad (9)$$

All of the previous equations have been incorporated into an Excel-based binder blending tool for asphalt binders. Figure 5 presents the blending tool interface, which entails two parallel Y-axes and an X-axis. The left Y-axis represents the PG of the soft asphalt binder (i.e., virgin asphalt binder blended with an RA), the right Y-axis represents the PG of the RAP asphalt binder, and the X-axis represents the RBR level or the percentage of RAP binder in the asphalt mixture. The target asphalt binder PG is graphed horizontally across the blending chart at high-, intermediate-, and low-temperature gradings; see Figure 5.

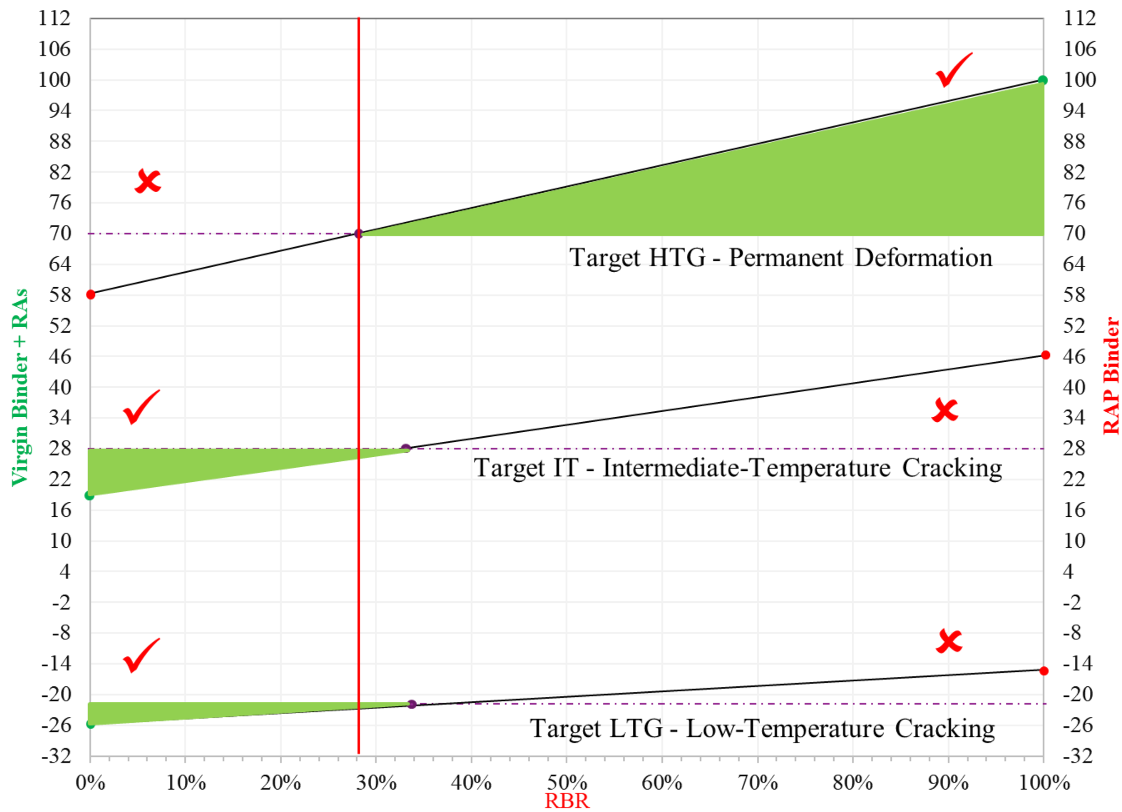
Figure 5. Excel-based tool for blending asphalt binders



After determining the RAP-binder PG and target asphalt binder PG, an RBR level of 0.28 (corresponding to 30% RAP content in the asphalt mixture) was selected. This RBR level is presented by a red vertical line intersecting the X-axis on the blending tool chart; see Figure 6. Three lines were then drawn from the RAP binder PG points on the right Y-axis to the intersection points corresponding to the target asphalt binder PG for low-, intermediate-, and high-temperature gradings and the RBR vertical line. These lines were extended to the left Y-axis to determine the required soft asphalt binder PG. In this example, the required soft asphalt binder was PG 58-28; see Figure 6. The unmodified

PG 67-22 virgin asphalt binder was softened to PG 58-28 asphalt binder by blending it with optimized dosages of RAs. It is noted that the three green triangles on the blending chart represent the regions where the target asphalt binder PG can be achieved. The RBR level can be adjusted within these acceptable regions, as demonstrated by the RBR of 0.28 in Figure 5. While an RBR of 0.3 could have been selected, 0.28 was chosen as a more conservative value to ensure optimal cracking performance.

Figure 6. RAs dosage optimization using asphalt binder blending tool



To simplify this blending tool for practitioners, the following input-output interface was developed. Table 3 presents an example of a 30% RAP mixture blend:

- Inputs: RAP-binder PG of 100-16, RBR of 0.28, and target binder of PG 70-22
- Output: Blend of virgin binder and RAs with PG 58-28

It is noted that this blending tool is developed to compute the PG of the resulting asphalt binder blend and does not directly predict asphalt mixture performance. To evaluate asphalt mixture performance, mechanical tests should be conducted and assessed against the BMD framework criteria.

Table 3. Binder blending tool example

Input parameters	
RAP binder (PG)	PG 100-16
RBR, %	0.28
Target binder (PG)	PG 70-22
Output results	
Blend of virgin binder and RA (PG)	PG 58-28

Experimental Program

Table 4 shows the asphalt mixtures considered in this study for design, preparation, and testing. The first three mixtures (M67, M70, and M76) contained conventional asphalt binders PG 67-22, PG 70-22 (SBS-modified), and PG 76-22 (SBS-modified), respectively. Mixture M6730 contained PG 67-22 asphalt binder and 30% RAP materials without RAs. Mixture M6730F contained PG 67-22 asphalt binder, 30% RAP, and 2% $FeCl_3$. Mixes M1 to M7 contained PG 67-22 asphalt binder blended with 30% RAP and RAs 1 to 7, respectively. It is noted that the RAP materials considered in this study contained 4.9% asphalt content (AC), and asphalt mixtures were designed to include 5.3% AC. The recycled binder ratio (RBR) was 0.28, calculated using Equation 10. Similarly, M1 to M7 were also prepared with 50% RAP and RAs 1 to 7, respectively.

$$\mathrm{RBR} = \frac{\mathrm{RAP},\% * \mathrm{AC}_{\mathrm{RAP}},\%}{\mathrm{AC}_{\mathrm{total}}},\% \qquad (10)$$

Table 4. Asphalt mixtures and experimental factorial for Approach 1

Mixture ID	Virgin Asphalt Binder	RBR	RA (Table 2)
M76	PG 76-22	--	--
M6730	PG 67-22	0.28	--
M6730F	PG 67-22	0.28	$FeCl_3$
M6750F	PG 67-22	0.46	$FeCl_3$

Note: M: Mixture; PG: Performance grading; RAP: Reclaimed asphalt pavement; RBR: Recycled binder ratio; RA: Recycling agent; $FeCl_3$: Iron chloride.

Table 5. Asphalt mixtures and experimental factorial for Approach 2

Mixture ID	Virgin Asphalt Binder	RBR	RA (Table 2)
M70	PG 70-22	--	--
M76	PG 76-22	--	--
M1	PG 67-22	0.28 & 0.46	RA1
M2	PG 67-22	0.28 & 0.46	RA2
M3	PG 67-22	0.28 & 0.46	RA3
M4	PG 67-22	0.28 & 0.46	RA4
M5	PG 67-22	0.28 & 0.46	RA5
M6	PG 67-22	0.28 & 0.46	RA6
M7	PG 67-22	0.28 & 0.46	RA7

Note: M: Mixture; PG: Performance grading; RAP: Reclaimed asphalt pavement; RBR: Recycled binder ratio; RA: Recycling agent.

Asphalt Mixture Design

Three Superpave asphalt mixtures with a nominal maximum aggregate size (NMAS) of 12.5 mm were designed and evaluated in this study. A Louisiana Level 2 asphalt mixture design was performed following the AASHTO R 35 standard [29] and Section 502 of the 2016 *Louisiana Standard Specifications for Roads and Bridges* [25]. The first mixture (M76) served as the conventional/control, containing PG 76-22M asphalt binder and no RAP. The second mixture (M6730) included PG 67-22 asphalt binder, 30% RAP, without the addition of $FeCl_3$. The third mixture (M6730) and fourth mixture (M6750F) both used

PG 67-22 asphalt binder and 2% $FeCl_3$, with the third containing 30% RAP and the fourth containing 50% RAP. Table 6 presents the job mix formula for the four mixtures evaluated. It is noted that the four mixtures are fine-sided of the maximum density line, dense-graded, and exhibit similar gradation. The design and RAP asphalt binder contents were 5.3% and 4.7%, respectively.

Table 6. Asphalt mixtures—job mix formulas

Mixture Designation		M76/M70/M67	30% RAP Mixes	50% RAP Mixes	DOTD Specs
Asphalt binder type		PG 76-22M/ PG 70-22M/ PG 67-22	PG 67-22	PG 67-22	-
Aggregate blend	LS#78, %	60.0	45.3	30.0	
	LS#11, %	32.0	20.6	16.0	
	CS, %	8.0	4.1	4.0	
Asphalt binder content (include RAP binder), %		5.3	5.3	5.3	
RAP content, %		0.0	30.0	50.0	
RBR		0.0	0.28	0.46	
Number of gyrations in SGC	N_i	7	7	7	7
	N_d	65	65	65	65
	N_f	105	105	105	105
Design volumetric properties	$\%G_{mm}$, N_i	86.2	86.5	87.7	<89
	$\%G_{mm}$, N_f	97.6	97.6	97.7	<98
	AV, %	3.9	3.9	3.7	2.5 - 4.5%
	VMA, %	14.8	13.6	15.7	≥13.5
	VFA, %	74.2	78.4	76.5	69 - 80
Passing	25.0mm	100.0	100.0	100.0	-
	19.0mm	100.0	100.0	100.0	100
	12.5mm	96.3	96.6	94.8	90 -100
	9.5mm	86.2	87.2	86.4	≤89
	4.75mm	43.0	46.3	44.4	-
	2.36mm	31.3	32.0	31.6	29-58
	1.18mm	22.5	24.6	23.6	-
	0.600mm	16.3	19.2	17.9	-
	0.300mm	9.2	12.3	10.9	-
	0.150mm	5.2	8.4	6.3	-
	0.075mm	4.0	4.7	4.4	4.0 -10.0

Note: % RAP: Percentage recycled asphalt pavement content in asphalt mixture; LS: Limestone aggregates; CS: Coarse natural sand aggregates; RBR: Recycled binder ratio to the total asphalt content; $FeCl_3$: Iron chloride; N_i: Initial number of gyrations; N_d: Design number of gyrations; N_f: Final number of gyrations; SGC: Superpave gyratory compactor; G_{mm}: Maximum Specific Gravity; $\%G_{mm}$, N_i: percent of G_{mm} at N_i; $\%G_{mm}$, N_f: percent of G_{mm} at N_f;AV; Air voids; VMA: Volume of mineral aggregates; VFA: Voids filled with asphalt.

It is noted that $FeCl_3$ was initially blended into the virgin asphalt binder at 325°F (163°C) using a high-shear mixer at 3,000 rpm for 30 min [30]. The following steps were then followed to prepare the asphalt mixtures containing RAP materials [31]:

1. 5% moisture content was added to the RAP. It was stirred thoroughly to ensure water did not collect with fines at the bottom of the pan, then soaked overnight.
2. Virgin aggregates were superheated to a minimum temperature of 383°F (195°C) for 3 hrs., while the mixing tools were heated to 325°F (163°C).
3. The moisture-laden RAP was placed at the bottom of the heated mixing bucket, and the superheated virgin aggregates were placed on top. These materials were then mixed, resulting in steaming. Mixing continued until the steam ceased and the RAP's dark color disappeared, indicating the detachment of the RAP binder from the RAP aggregates.
4. The blended aggregates and RAP materials were heated to 325°F (163°C) to reach the mixing temperature with the asphalt binder.
5. The heated asphalt binder and blended aggregates were mixed in a heated mixing bucket. After mixing, the mixture was spread in a pan and short-term oven-aged for 2 hrs. at 275°F (135°C).
6. Finally, compacted cylindrical specimens were prepared to the specified dimensions for each test procedure using the Superpave gyratory compactor (SGC).

Laboratory Testing

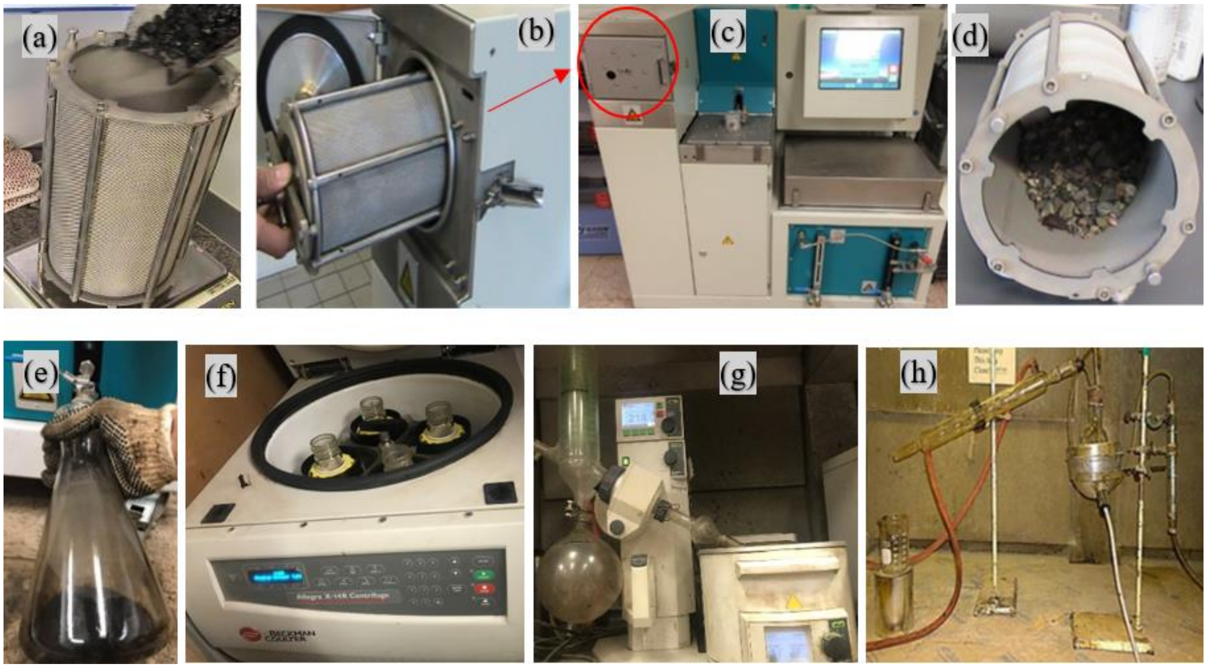
Asphalt Binder Testing

Three grades of asphalt binders were evaluated: PG 76-22, PG 70-22, and PG 67-22, all meeting Louisiana specifications [25]. The asphalt binders PG 76-22 and PG 70-22 binders were produced by blending 3.5% and 1.5% polystyrene-butadiene-styrene (SBS) by weight, respectively, into unmodified asphalt binder PG 67-22.

Asphalt Binder Extraction

Asphalt binders were extracted using trichloroethylene (TCE) solvent, following the ASTM D 8159 standard [32]. An automated asphalt analyzer was used for this process. Approximately 2 kg of loose asphalt mixture was placed into the machine's drum; see Figure 7(a). The drum was then installed in the machine; see Figure 7(b). Inside the drum, the asphalt mixture was washed and dried automatically through multiple cycles using the TCE solvent (see Figure 7(c)), resulting in the separation of aggregates (see Figure 7(d)) and unrecovered asphalt binder (see Figure 7(e)). An auto-centrifuge (Allegra X-14R) was then used to remove any remaining fillers and fines by spinning at 770 rotations per min. for 30 min.; see Figure 7(f). Subsequently, an auto-evaporator was used to condense most of the TCE; see Figure 7(g). To further separate the TCE from the extracted asphalt binder, the Abson distillation process was followed, following the ASTM D 1856 standard [32]. Any remaining traces of TCE were then removed by introducing carbon dioxide gas. The Abson method was conducted as stipulated by the AASHTO R 59 standard [33]. The setup for the Abson Method is shown in Figure 7(h). Finally, the extracted asphalt binder and aggregates were used for material testing and characterization.

Figure 7. Asphalt binder extraction



Rheological Characterization

Performance Grading. The rheological properties of asphalt binders are crucial to the performance of asphalt mixture pavements. These properties change during production and over time due to aging caused by oxidation and environmental factors. If these changes are not properly addressed before production, pavement distresses such as raveling, cracking, stripping, and rutting may occur. To ensure that asphalt binders meet the criteria to minimize pavement distress, their properties must be tested. Specifications have been developed to limit the contribution of asphalt binders to durability, rutting, fatigue cracking, and low-temperature cracking.

In this study, asphalt binders (RAP binders, virgin asphalt binders, asphalt binders containing RAs, and extracted asphalt binders from asphalt mixtures) were tested and characterized according to the AASHTO R 29 standard [34] and DOTD specifications [25]. To evaluate the rheological properties of these binders, short- and long-term aging was simulated using the Rolling Thin Film Oven (RTFO) and the Pressure Aging Vessel (PAV), respectively. The RTFO test, as stipulated in AASHTO T 240 [35], simulates the aging that occurs during production and construction, while the PAV test, administered according to AASHTO R 28 [36], simulates the long-term aging experienced during service life.

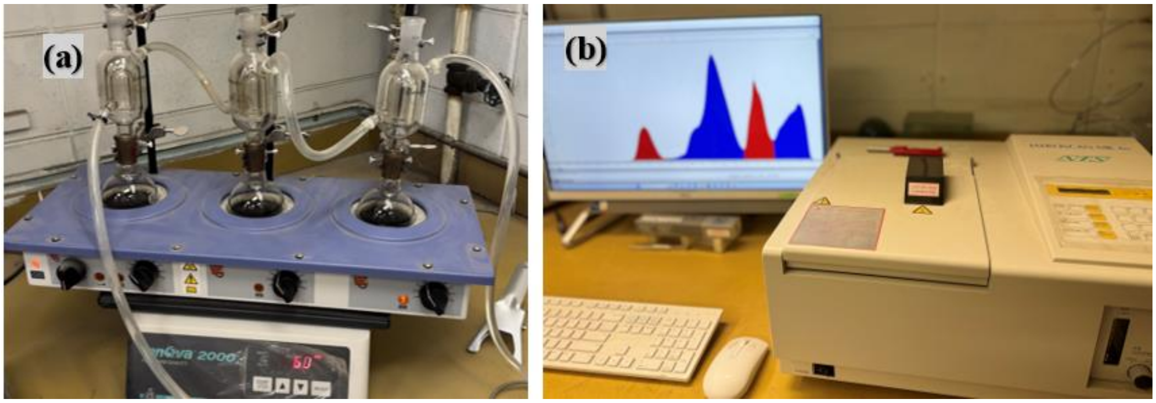
Following the short and long-term aging simulations, various rheological tests were conducted. The Rotational Viscometer (RV) test, following AASHTO T 316 [37], measures the binder viscosity at high construction temperatures (135°C) to ensure it can be pumped and handled easily. Further, the dynamic shear rheometer (DSR) device, utilized according to AASHTO T 315 [38], measured the binder properties at high and intermediate temperatures encountered during pavement service to determine its resistance to rutting and fatigue cracking.

In addition to high and intermediate temperature performance, the low-temperature characteristics of the binders was assessed using the bending beam rheometer (BBR) device, which measures the binder properties during cold weather to determine its resistance to low-temperature cracking as described in the AASHTO T 313 standard [27]. Finally, the performance grading of the tested asphalt binders was determined by analyzing the results from these rheological tests according to AASHTO M 320 [26].

Chemical Composition

SARA Analysis. The chemical composition of asphalt binders was determined through SARA analysis, which stands for saturates, aromatics, resins, and asphaltenes. This method, also known as column chromatography, separates the asphalt binder into asphaltenes (insoluble in n-heptane) and maltenes (soluble in n-heptane). The maltenes are further separated into saturates, resins, and aromatics using column chromatography. SARA analysis was conducted following the ASTM D 3279 standard [39]; see Figure 8(a). The maltenes fractionation was conducted using an Iatroscan TH 10 Hydrocarbon Analyzer to isolate saturates, aromatics, and resins; see Figure 8(b). N-pentane was used to elute saturates, while a 90/10 toluene/chloroform mixture isolated aromatics. Resins remained at the origin and were not eluted. It is noted that the combination of saturates, aromatics, and resins constitutes the maltenes fraction.

Figure 8. SARA analysis devices

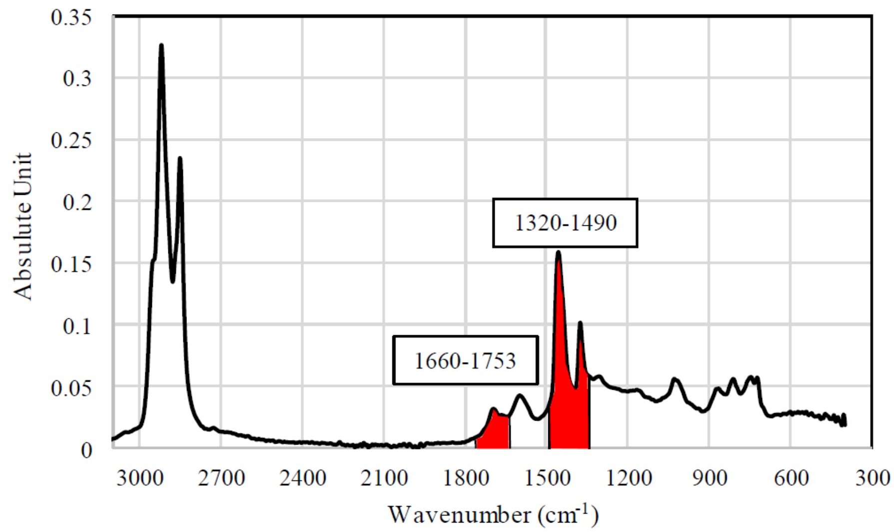


Asphaltenes and maltenes are two crucial components of asphalt binder, each with distinct properties that significantly affect its performance. Asphaltenes, defined as the fraction insoluble in pentane or heptane, consist of complex polar molecules known to increase viscosity [40]. During aging, oxidation leads to the formation of ketones, which influence polarity and solubility, causing aromatic components to clump together into asphaltenes [41]. This increase in asphaltenes is a primary driver of the observed rise in viscosity as the binder ages [41].

Fourier Transform Infrared Spectroscopy (FTIR). The FTIR test was conducted according to the ASTM E 1252 standard [42] to identify and quantify the functional groups present in asphalt binders. This approach follows the principle that molecules absorb infrared light at resonant frequencies characteristic of their covalent bonds. By analyzing the position, shape, and intensity of peaks in the resulting infrared spectrum, details about the molecular structure of the asphalt can be revealed [41]. In this study, the carbonyl index (C=O, a carbon atom double-bonded to an oxygen atom) was evaluated in relation to aging and cracking resistance. The underlying rationale is that highly polar and strongly interacting oxygen-containing functional groups, including carbonyl, are formed during the oxidative aging process. When the concentration of such polar functional groups becomes sufficiently high to cause molecular immobilization through increased intermolecular interaction forces, cracking will occur [43, 44, 45]. The carbonyl index (CI) is defined by the ratio indicated in Equation 11. Figure 9 shows a sample of FTIR test results for the recovered asphalt binder.

$$CI = \frac{\text{Area of carbonyl band centered around 1700 cm} - 1}{\Sigma \text{Areas of spectral bands between 1320 and 1490 cm} - 1} \tag{11}$$

Figure 9. Sample FTIR spectrum



Asphalt Mixture Testing

Table 7 presents the details of the mechanical tests conducted on asphalt mixtures. All specimens were prepared to achieve an air void level of 7 ± 0.5%. Specimens were then long-term oven aged at 85°C for 120 hrs., following the AASHTO R 30 standard [46] before testing, except for the HWT test, which was conducted directly after short-term aging conditioning.

Table 7. List of mechanical tests conducted on asphalt mixtures

Test designation	Testing temperatures (℃)	No. of replicates / _ Sample Size: Dia. (mm) x Height (mm) x Width (mm)	Engineering properties	Protocols / standards
Dynamic Modulus	4.4, 25, 37.8, & 54	3/D100 x H150	Stiffness	AASHTO T 342 [47]
HWT	50	4/D150 x H60	Rutting resistance	AASHTO T 324 [48]
MiST + HWT	60	4/D150 x H60	Moisture damage	ASTM D 7870 [49]; AASHTO T 324 [48]
SCB	25	4/D150 x H57	Intermediate temperature cracking resistance	ASTM D 8044 [50]
Cantabro	25	3/D150 x H115	Durability	AASHTO T 401 [51]
TSRST	5 & -10/hr.[1]	3 / T50 x W50 x H250	Low temperature cracking resistance	AASHTTO TP 10 [52]

Note: HWT: Hamburg wheel tracker; MiST: Moisture-induced Stress Tester; SCB: Semi-circular Bending test; TSRST: Thermal Stress Restrained Specimen Tensile Strength test. [1] The test temperature starts at 5℃, then gets colder at a rate of -10℃ per hour.

Dynamic Modulus Test

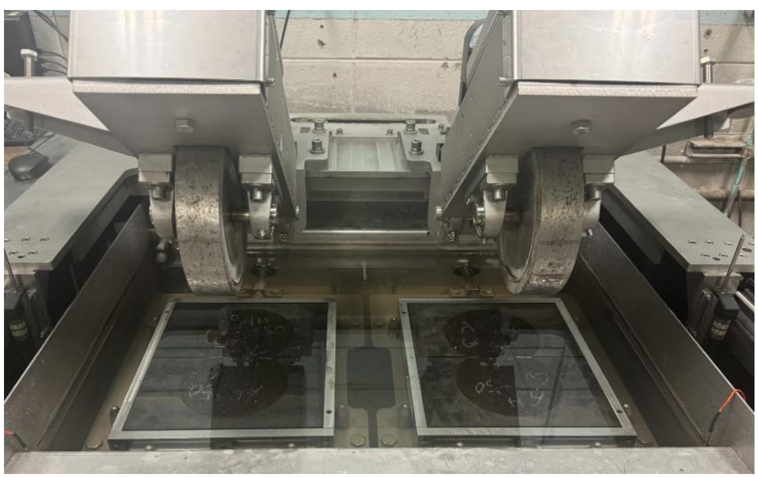
The dynamic modulus (DM) test is a triaxial compression test that represents asphalt mixture stiffness and is conducted following the AASHTO T 342 standard [47]. A uniaxial sinusoidal (haversine) axial compressive stress with different loading frequencies (0.1, 0.5, 1, 5, 10, and 25 Hz) was applied to the sample at specific temperatures (4.4, 25, 37, and 54˚C) [53]. The applied stress and the resulting strain response of the specimen were measured continuously during the test using a data acquisition system. The stress-to-strain relationship under a continuous sinusoidal loading for linear viscoelastic materials is denoted as a complex modulus often called E star (E*). The absolute value of the complex modulus |E* | is the dynamic modulus. The dynamic modulus is mathematically defined as the maximum or peak dynamic stress divided by the peak recoverable strain; see Equation 12.

$$\left|E^*\right| = \frac{\sigma_o}{\epsilon_o} \tag{12}$$

Hamburg Wheel Tracking (HWT) Test

The critical distress in asphalt pavement in high-temperature climates is permanent deformation, often known as rutting distress. The HWT test was conducted following the AASHTO T 324 standard [54] to evaluate the rutting performance of the studied asphalt mixtures; see Figure 10. In this test, specimens are subjected to a steel wheel that repeatedly rolls across their surface at a speed of 1.1 km/hr. and a passing rate of 52±2 passes/min. while submerged in 50°C hot water. Each wheel rolls 230 mm (9.1 in.) before reversing direction. The test wheel weighs 703 N (158 lbs.), has a diameter of 203.5 mm (8 in.), and has a width of 47 mm (1.85 in.). Before testing, the laboratory specimens were conditioned by being submerged in hot water for 45 min. at 50°C. The test completion time is predicated upon test specimens being subjected to a maximum of 20,000 cycles or attainment of 20 mm deformation, whichever is reached first. Upon completion of the test, the average rut depth for the tested samples is recorded. To accurately measure permanent deformation, two Linear Variable Displacement Transducers (LVDTs) were utilized, and the subsequent test results (rut depths, number of passes, water bath temperature) were collected and recorded in an automatic data recording system associated with the HWT device used in this study. Lower rut depth values are desirable for rut-resistant mixtures.

Figure 10. Hamburg loaded wheel tracking device



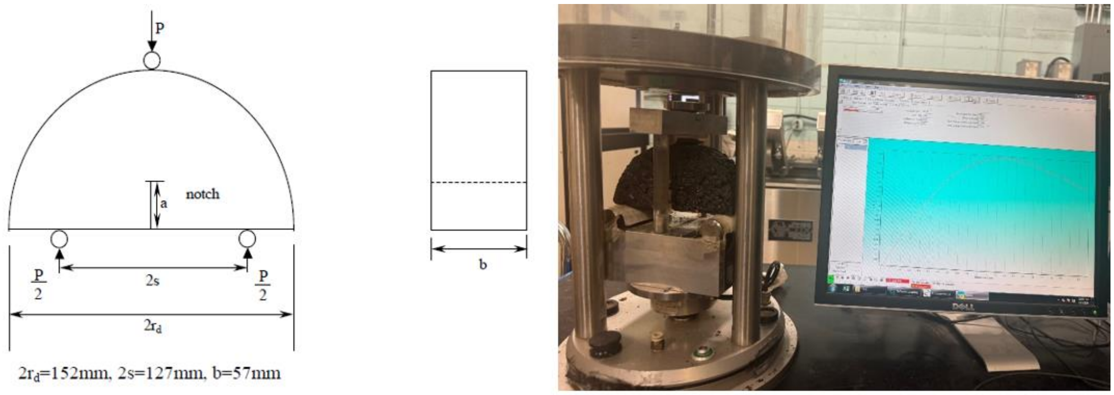
Moisture-induced Stress Tester (MiST)

Moisture damage is a major concern for asphalt pavements, as it can significantly reduce their lifespan and performance. When water infiltrates the pavement, it weakens the bond between the asphalt binder and the aggregate particles, leading to a variety of distress mechanisms, such as cracking, potholes, and raveling. The MiST conditioning protocol is a laboratory technique used to evaluate the susceptibility of asphalt mixtures to moisture damage. MiST conditioning is conducted following the ASTM D 7870 standard [49] to simulate the combined effects of water, stress, and elevated temperature that pavements experience in real-world conditions. Specimens used in this test had dimensions of 60 mm in height and 150 mm in diameter. After MiST conditioning, four specimens were then tested using the HWT device to better characterize their resistance to moisture damage.

Semi-Circular Bending (SCB) Test

The cracking potential of the asphalt mixtures was evaluated using the SCB test procedure, which is based on fracture mechanics principles [55, 56]. The SCB test was conducted according to ASTM D 8044 standards [50]. This test simulates the cracking propagation resistance of asphalt mixtures under loading conditions. Figure 11 presents the specimen dimensions and device setup. In this study, semi-circular specimens with a thickness of 57 mm and two notch depths, 25.4 mm and 38.1 mm, were evaluated. The semi-circular specimens were loaded monotonically until fracture failure under a constant crosshead deformation rate of 0.5 mm/min. in a three-point bending load configuration. The load and deformation were continuously recorded. This test was performed at a temperature of 25°C. The area under the load-deformation curves, up to the maximum load measured for each notch depth, represents the strain energy to failure (U). The average values of U were then plotted against the different notch depths to compute a regression line slope, which yields the value expression dU/da. The J_c was computed by dividing the dU/da value by the specimen thickness. Higher J_c values are desirable for fracture-resistant mixtures.

Figure 11. SCB test specimen dimensions and device setup



The critical strain energy release rate, also called the critical value of J-integral (J_c), was used to describe the mixture's resistance to fracture; see Equation 13.

$$J_c = -\left(\frac{1}{b}\right) * \frac{dU}{da} \qquad (13)$$

where,
J_c= critical strain energy release rate (kJ/m^2);
b = sample thickness (m);
a = notch depth (m);
U = strain energy to failure (kJ); and
dU/da = change of strain energy with notch depth (kJ/m).

In this study, four test replicates were used for each notch depth. DOTD specified that the SCB J_c should be higher than 0.6 kJ/m^2 for Level 2 mixtures (i.e., roads with traffic higher than 3 million ESALs) [25].

Cantabro Test

The Cantabro test was conducted according to the AASHTO T 401 standard [51] to evaluate asphalt mixtures' durability and abrasion resistance. The test utilizes a Los Angeles Abrasion machine, a rotating drum that simulates the grinding and wear an asphalt pavement experiences under traffic. The specimens were weighed before and after testing, and the weight loss (Cantabro loss) was calculated as a percentage of the original weight. A lower Cantabro loss indicates a more durable asphalt mixture that can better resist asphalt pavement degradation from traffic and environmental conditions.

Thermal Stress Restrained Specimen Tensile Strength Test (TSRST)

The TSRST was conducted according to the AASHTO TP 10 standard [52] to evaluate asphalt mixtures' resistance to low-temperature cracking. This method determines the tensile strength and the fracture temperature of an asphalt mixture by measuring the tensile load in a specimen that is cooled at a constant rate while being constrained from contraction. The data acquired from this test allows for the determination of the temperature versus stress relationship of the asphalt mixture. For each asphalt mixture studied, a rectangular slab (300 mm wide x 400 mm long x 50.8 mm thick) was compacted using a linear kneading compactor. After compaction, the rectangular slabs were cooled to room temperature and then checked to have 7±0.5% air voids. The slabs were then sawn into four replicate test beams with dimensions of 50±5 mm square and 250±5 mm long. The sawed beams were long-term aged, glued to platens at both ends, conditioned at 5±1°C for 4 hrs., and cooled at a rate of 10.0±1°C per hr. until tensile fracture occurred. The thermal contraction along the long axis of the specimen was monitored electronically, and the initial length of the asphalt mixture's beam specimen was held constant. Before the failure temperature was reached, the tensile strength slope (dS/dT) was computed to assess the stress build-up during testing. A lower dS/dT is desired and associated with better low-temperature cracking resistance [57].

Discussion of Results

Laboratory test data underwent statistical analysis using the Analysis of Variance (ANOVA) procedure implemented in the Statistical Analysis System (SAS) software (Version 9.4, SAS Institute Inc., Cary, NC) [58]. A multiple comparison test was conducted on the means at a significant level of $\alpha = 0.05$. Tukey's Honestly Significant Difference (HSD) test was chosen due to its ability to control Type I errors and its suitability for comparisons involving large datasets ($n \geq 6$). The groupings reflected the mean test results categorized by mixture type. The results are reported using a letter notation (A, B, C, etc.), with A assigned to the highest mean and subsequent letters following in descending order. A single letter designation (e.g., A vs. B) indicates a statistically significant difference between the corresponding means. Conversely, a double or multiple-letter designation (e.g., A/B or A/B/C) signifies that the mean differences are not statistically clear, suggesting that the mean in question is not definitively higher or lower than the designated comparison groups.

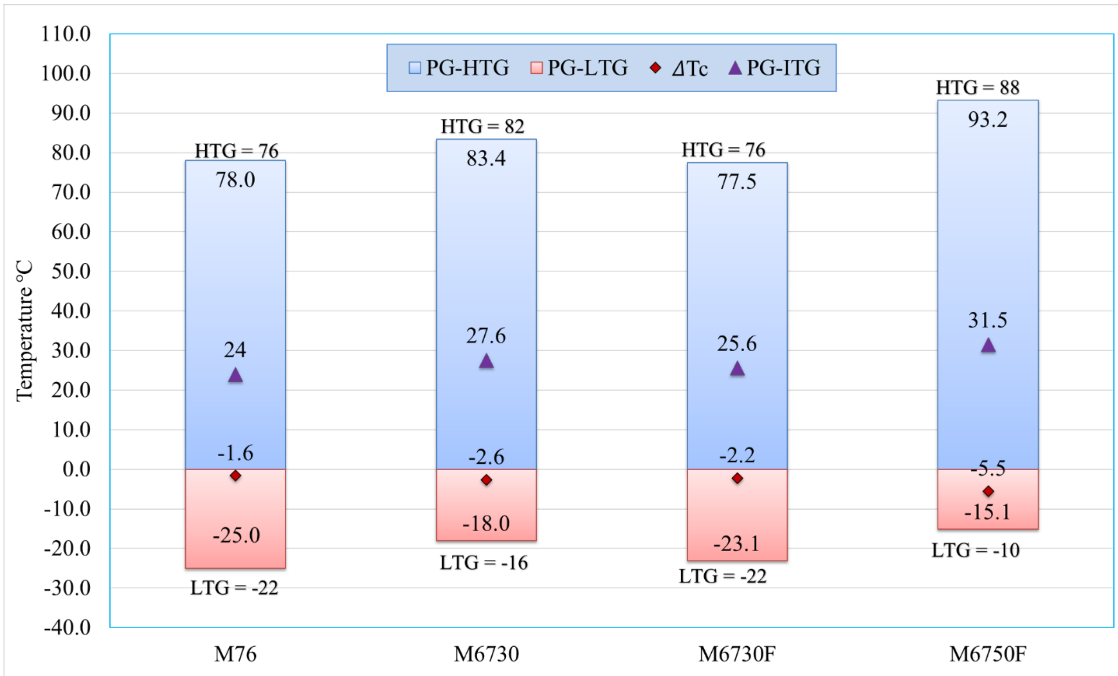
Approach 1: Reagent Catalyst

Asphalt Binder Test Results

Performance Grading (PG)

Figure 12 presents the PGs of asphalt binders extracted from the studied mixtures. As expected, the control mixture contained asphalt binder PG 76-22 with no RAP or additives. Asphalt mixture M6730 contained virgin asphalt binder PG 67-22 and 30% RAP (RBR = 0.28), which yielded a PG 82-16 blended asphalt binder. The inclusion of 2% $FeCl_3$ in mixture M6730F reduced the PG by one grade, which demonstrates the reduction in the stiffness of the binder contributed by the RAP materials. For ΔT_c, the addition of 2% $FeCl_3$ in mixture M6730F increased the temperature from -2.6°C to -2.2°C, reflecting an enhancement of the balance between stiffness and relaxation of blended asphalt binder, which is expected to enhance low temperature cracking resistance. However, increasing the RAP content to 50% with the inclusion of $FeCl_3$ in mixture M6750F resulted in a blended binder with PG 88-10. It is noted that the RAP binder was also extracted and graded as PG 100-16.

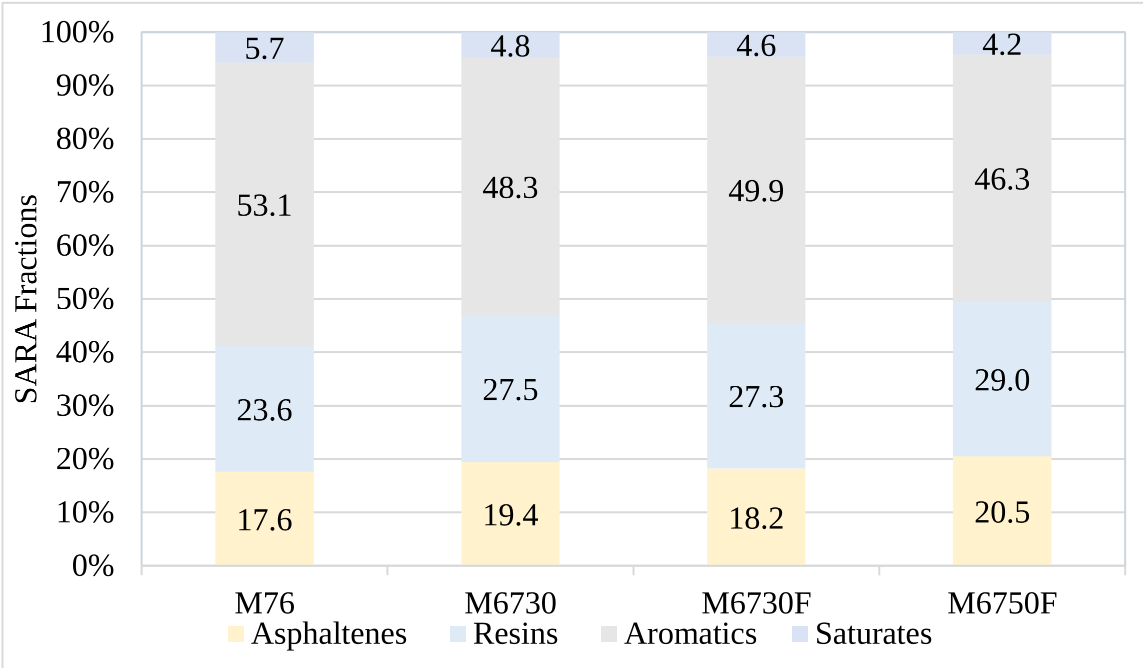
Figure 12. Asphalt binder performance grading (reagent catalyst)



SARA Analysis

Figure 13 presents the SARA fractions for the studied mixtures. Asphaltene and resin fractions are expected to increase, while aromatics and saturates decrease with the presence of oxidized and aged binders (RAP binders). The inclusion of $FeCl_3$ in the asphalt mixture M6730F reduced the asphaltene fractions from 19.4% to 18.2% when compared to the mixture without $FeCl_3$ (M6730); see Figure 13. For the mixture with 50% RAP (M6750F), the RAP binder increased the asphaltene fraction.

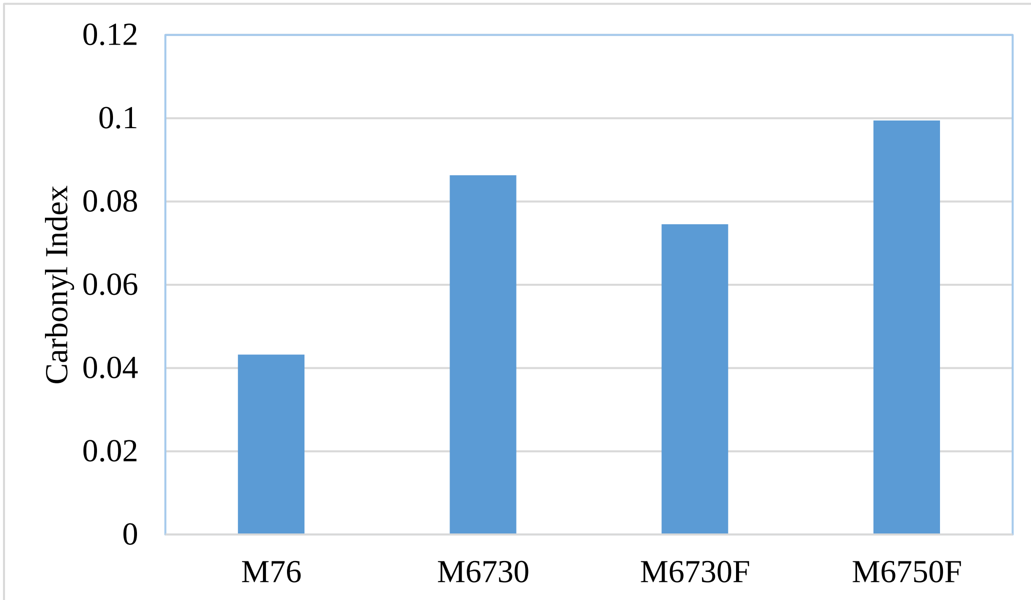
Figure 13. SARA fractions of the studied asphalt binders



Fourier Transform Infrared Spectroscopy (FTIR)

Figure 14 presents the carbonyl index (CI) values for the extracted asphalt binders from the studied asphalt mixtures. In this study, the carbonyl index (C=O, a carbon atom double-bonded to an oxygen atom) was evaluated in relation to aging and cracking resistance. The CI increases with increasing RAP content in the asphalt mixture; however, the inclusion of $FeCl_3$ in the asphalt mixture M6730F decreased CI when compared to mixture M6730, which does not have $FeCl_3$. As such, including $FeCl_3$ in asphalt mixtures with high RAP content is expected to enhance the asphalt mixtures' cracking resistance.

Figure 14. Carbonyl index of the extracted asphalt binders

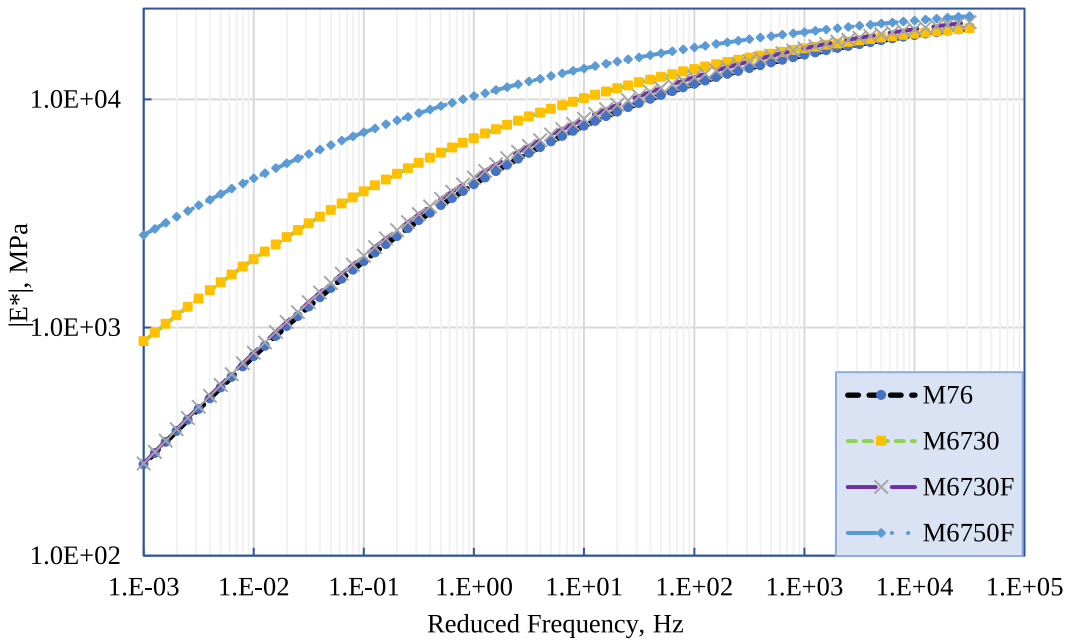


Asphalt Mixture Test Results

Dynamic Modulus

Figure 15 presents the dynamic modulus master curves of the studied mixtures at a reference temperature of 21.1°C, using the time-temperature superposition principle. The mixture M6730 showed higher stiffness than the conventional mixture M76. This implies that the addition of the RAP materials could stiffen the mixture even though a softer asphalt binder was used. By comparing mixture M6730 with M6730F, it can be observed that the incorporation of $FeCl_3$ significantly decreased the stiffness of the mixture, and that mixture M6730F showed similar stiffness as the conventional mixture M76. It is noted that mixture M6750F had the highest stiffness among the studied mixtures.

Figure 15. Dynamic modulus master curves



Hamburg Wheel Tracking (HWT) Test

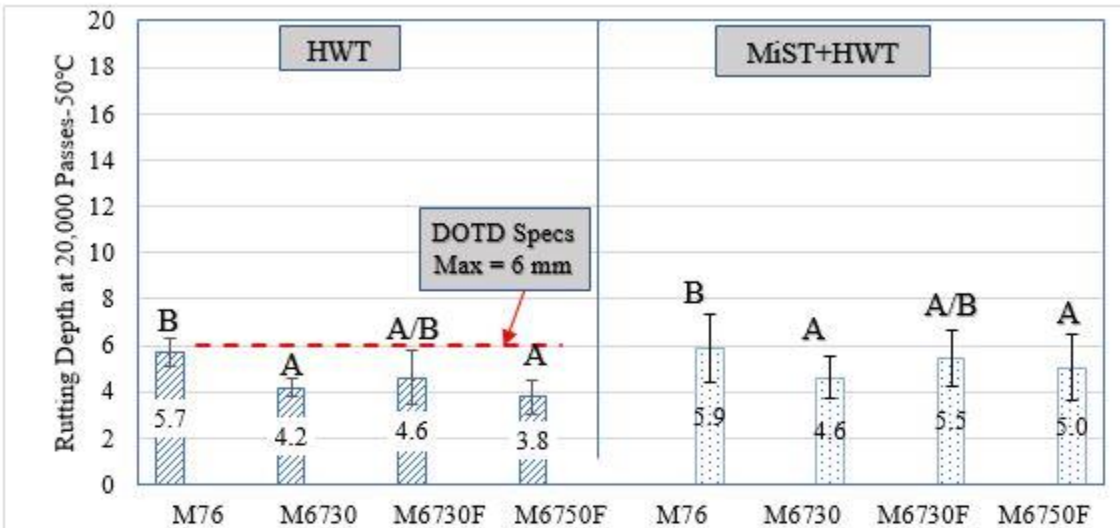
Figure 16 presents HWT test rut depths at 20,000 passes for the mixtures evaluated. The coefficient of variation (CoV) of the rut depth varied between 10-25%, with an overall average of 16%. As shown, the mixture containing the highest RAP content (M6750F) exhibited the lowest rut depth, followed by mixtures M6730, M6730F, and M76. It is noted that the rut depths met DOTD criteria of 6.0 mm for all mixtures [25]. Incorporating $FeCl_3$ in M6730F decreased the rutting resistance compared to M6730, yet it was still better than the conventional mixture M76. Increasing RAP content to 50% in M6750F enhanced the rutting resistance to be similar to that of M6730, despite the incorporation of $FeCl_3$.

Moisture-induced Stress Tester (MiST)

Figure 16 also illustrates the average rutting depths for samples conditioned using the Moisture-induced Stress Tester (MiST). The CoV of the rut depths varied between 13-28%, with an overall average of 22%. The 6 mm rutting depth threshold specified by DOTD [25] was set for unconditioned HWT rutting depth. However, all MiST conditioned HWT rutting depths were still able to achieve this threshold for all mixtures. M6730 achieved a lower rutting depth than the conventional mixture M76. Adding $FeCl_3$

in M6730F reduced the stiff RAP-binder rigidity, which increased the rutting depth when compared to M6730. Increasing RAP content to 50% with $FeCl_3$ in M6750F enhanced the moisture damage resistance.

Figure 16. HWT (MiST + HWT) rutting depths



Semi-Circular Bending (SCB) Test

Figure 17 presents the SCB critical strain energy release rates (J_c) results for the mixtures evaluated. The CoV of the J_c varied between 2-8%, with an overall average of 5%. DOTD specified [25] that the SCB J_c should be higher than 0.5 and 0.6 kJ/m^2 for Level 1 and Level 2 mixtures, respectively. The Level 1 mixture is designed for roads with traffic less than 3 million ESALs, and the Level 2 mixture for roads with traffic higher than 3 million ESALs [25]. As shown in Figure 17, mixture M6730F exhibited a higher J_c than mixture M6730, indicating that the $FeCl_3$ could improve the cracking resistance of the asphalt mixture containing 30% RAP. Mixture M6730F showed a comparable J_c to conventional mixture M76 and met the threshold of DOTD's Level 2 mixture design. Although mixture M6750F had a lower J_c than conventional mixture M76, it passed the criterion for DOTD's Level 1 mixture design.

Figure 17. SCB test J_c results

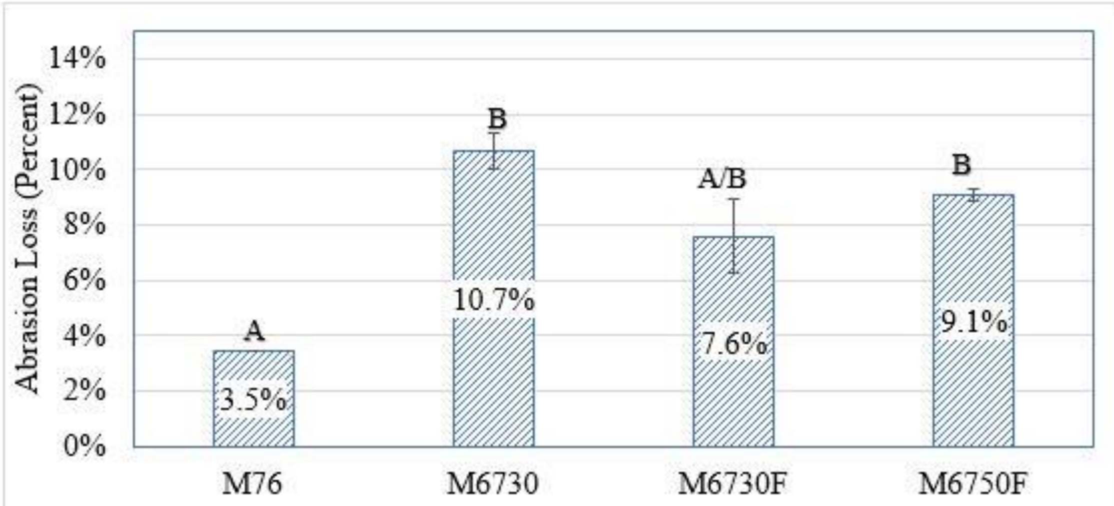
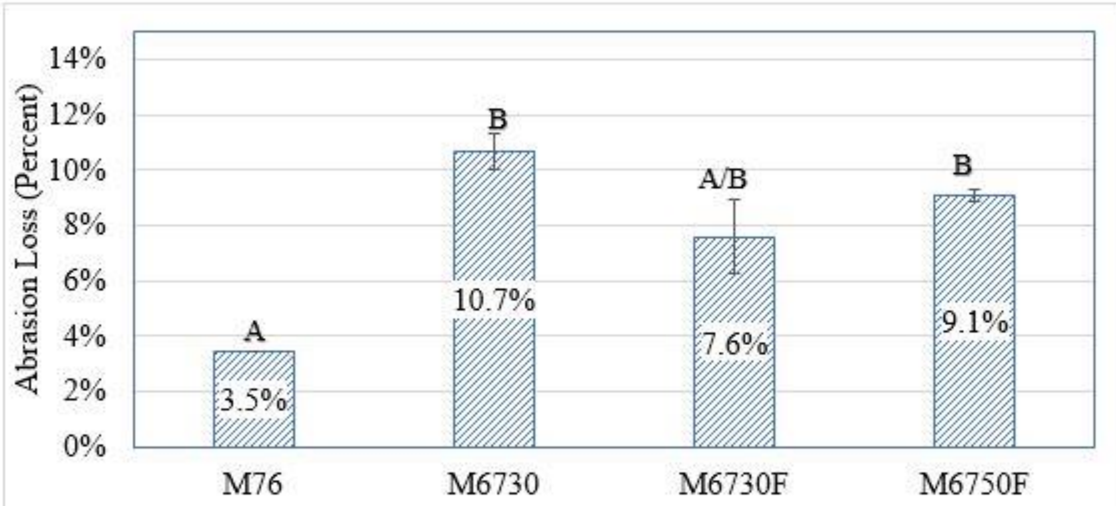


Figure 18 shows the Cantabro test results for all mixtures to evaluate their durability. The specimens were subjected to 300 revolutions within the Los Angeles abrasion machine's drum, and the loss of weight was recorded as abrasion loss. The lower the abrasion loss percentage of a mixture, the better durability it exhibits. As shown in Figure 18, mixture M6730 exhibited lower durability than the conventional mixture M76. Incorporating $FeCl_3$ in M6730F enhanced the mixture's durability compared to M6730 and resulted in similar performance to the conventional mixture M76. Increasing the RAP content to 50% in M6750F utilizing $FeCl_3$ marginally reduced the durability, yet it was still similar to M6730F.

Figure 18. Cantabro test results–abrasion loss percentages



Thermal Stress Restrained Specimen Tensile Strength Test (TSRST)

The TSRST test was conducted according to AASHTO TP 10 [52]. A 10°C temperature drop per hour was applied, starting at 5°C. Figure 19 presents the testing temperature versus the resulting tensile stress induced in the asphalt mixtures evaluated. Before the failure temperature, the tensile strength slope (dS/dT) was computed to assess the stress build-up during testing. A lower dS/dT is desired and associated with better low-temperature cracking resistance [57]. The addition of $FeCl_3$ to M6730F and M6750F improved low-temperature cracking resistance, resulting in a lower dS/dT compared to mixture M6730 without the Lewis acid catalyst; see Figure 20. As expected, the conventional mixture M76 exhibited the lowest slope (dS/dT) among the mixtures evaluated. It is noted that mixtures M6730F, M6750F, and M76 showed statistically similar dS/dT; see Figure 20. However, mixture M6730 exhibited a significantly higher slope compared to the other mixtures; see Figure 20.

Figure 21 shows the low temperature at which cracking occurs for the asphalt mixtures evaluated. A lower temperature, which is more negative, is desired for low-temperature cracking resistance. The use of $FeCl_3$ was effective in improving low-temperature cracking, such that M6730F showed significantly better resistance to low-temperature cracking than M6730 and had a similar performance to M76. The addition of $FeCl_3$ to the mixture that contains 50% RAP enhanced its low-temperature cracking performance to be similar to that of the mixture containing lower RAP content (M6730F).

Figure 19. Thermal Stress Restrained Specimen Tensile Strength test graph

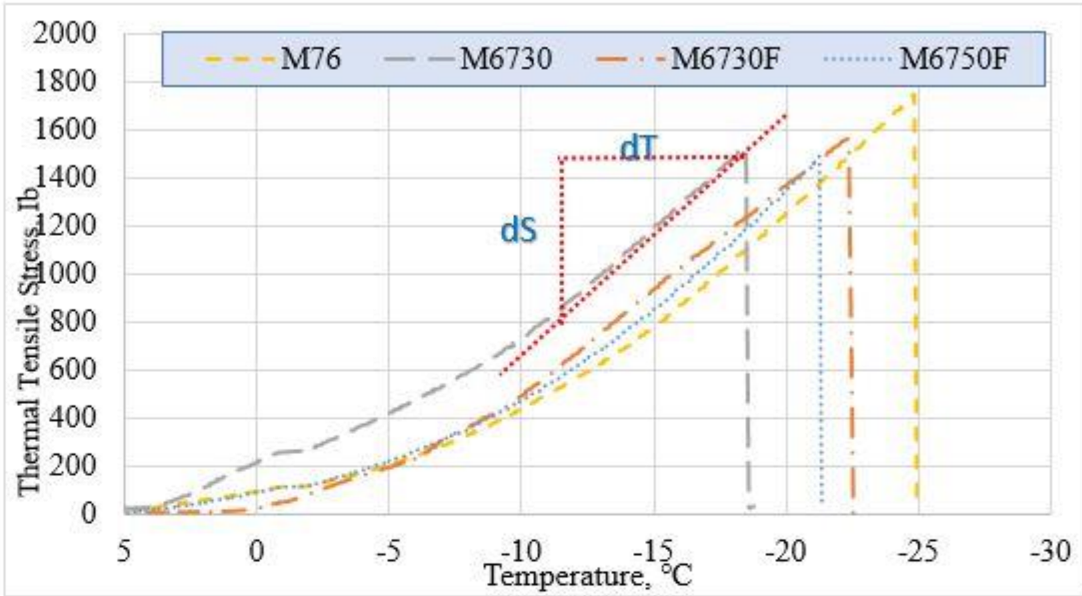


Figure 20. Tensile strength slope for TSRST test

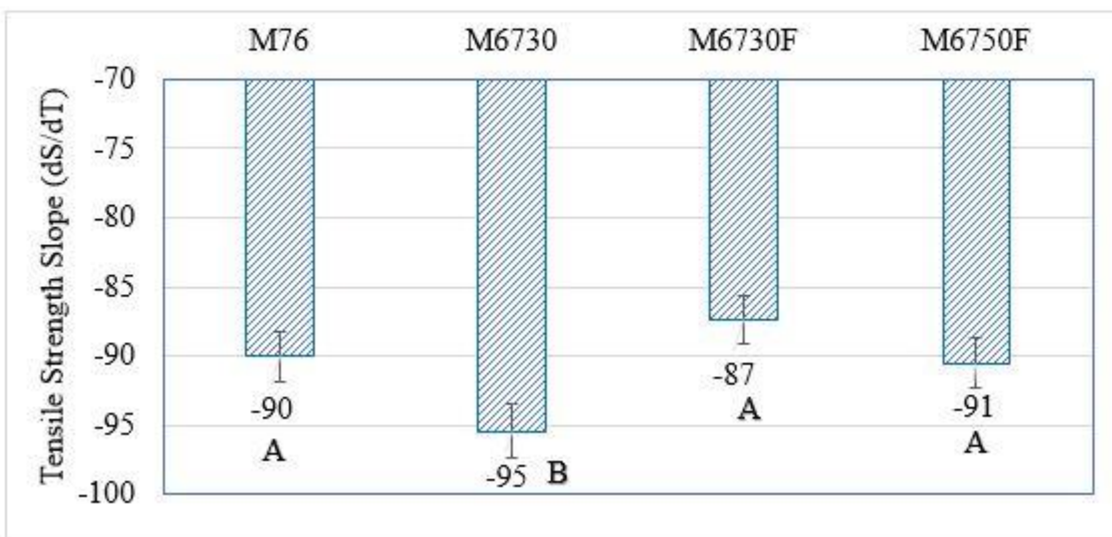
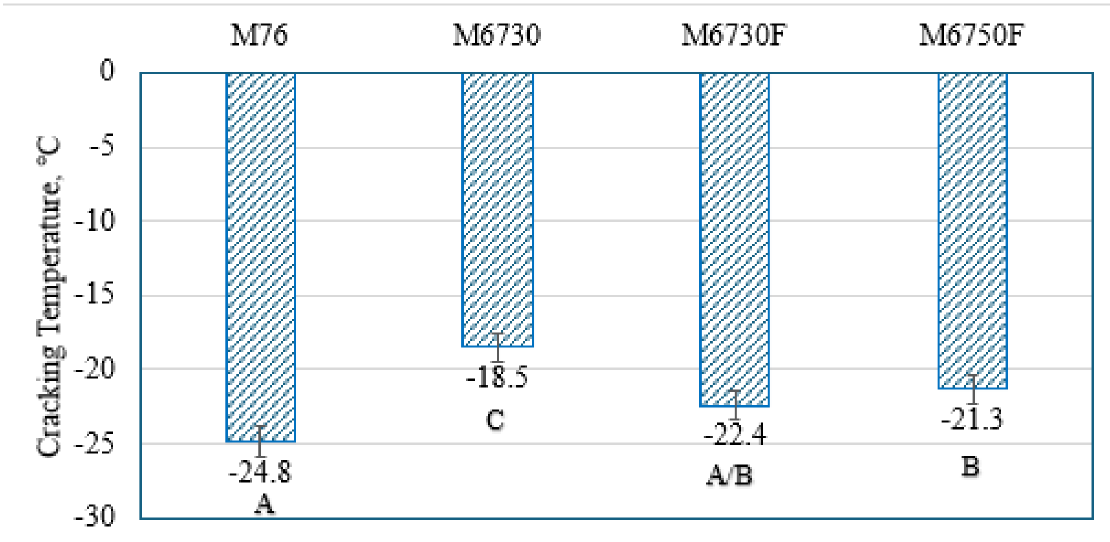


Figure 21. Low-temperature cracking temperature for the TSRST test

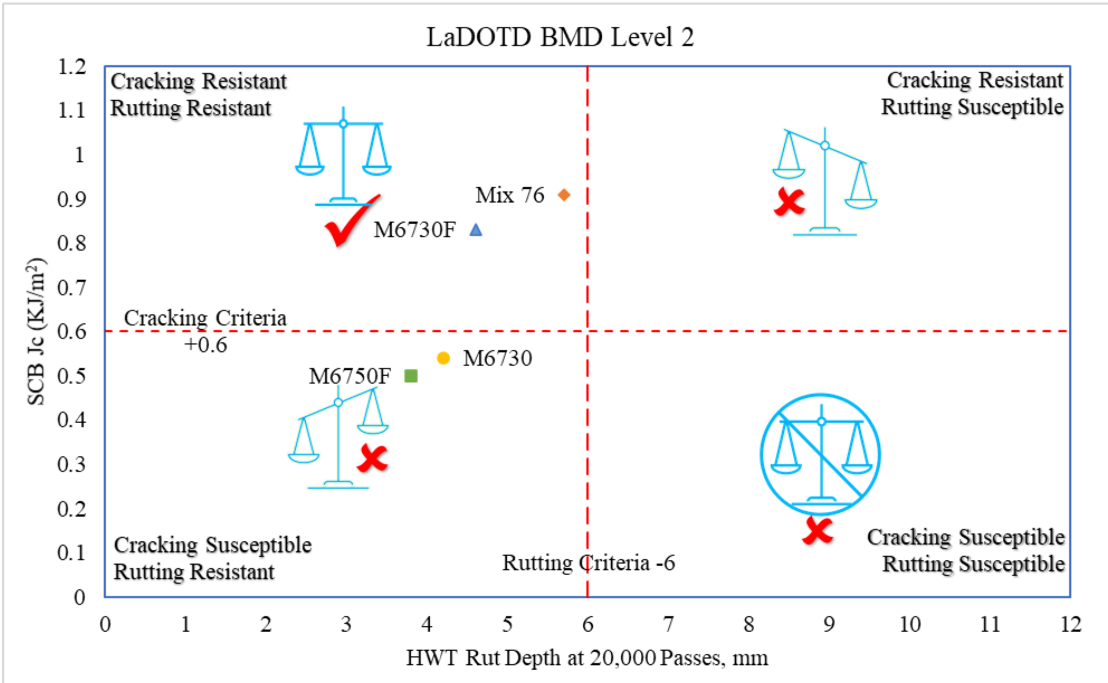


BMD Acceptance Criteria

Figure 22 presents Louisiana's balanced mixture design (BMD) framework. It is noted that Louisiana selected the mechanical tests based on the implementation of DOTD standard specifications for roads and bridges in 2016, which included SCB J_c and the rut depth from HWT [25]. The criteria for those tests, namely a maximum rut depth of 6.0 mm and a minimum SCB J_c value of 0.6 kJ/m^2 for Level 2 mixture design, were determined from field performance data of the Louisiana pavement system [59]. The BMD framework can be divided into four quadrants: the top-left quadrant presents a balanced design region in which asphalt mixtures are rutting and cracking resistant; conversely, the bottom-right quadrant contains rutting and cracking susceptible asphalt mixtures. The bottom-left quadrant includes asphalt mixtures that are rutting resistant but cracking susceptible, whereas the top-right quadrant contains rutting susceptible and cracking resistant mixtures.

As expected, the conventional asphalt mixture M76 performed well and met the BMD criteria. Asphalt mixture containing 30% RAP and virgin asphalt binder PG 67-22 with no $FeCl_3$ (M6730) did not meet the Louisiana DOTD requirements for a balanced mix; see Figure 22. However, adding $FeCl_3$ to asphalt mixtures containing RAP materials at RBR levels of 0.28 and 0.46 improved their cracking performance to meet Louisiana's Level 2 BMD criteria. In summary, mixtures M76, M6730F, and M6750F met Louisiana's BMD criteria and are further considered in the environmental impact analysis [60].

Figure 22. Balanced mix design criteria by DOTD



Summary of Test Results

Table 8 presents statistical comparisons between the mixtures containing high RAP content and the conventional mixture used in Louisiana. Based on the results, an asphalt mixture containing 30% RAP (RBR=0.28), P67-22 asphalt binder, and 2% $FeCl_3$ (M6730F) met the Level 2 traffic loading thresholds specified by DOTD [25]. It is noted that the asphalt mixture containing 50% RAP (RBR=0.46), P67-22 asphalt binder, and 2% $FeCl_3$ (M6750F) achieved a J_c of 0.5 kJ/m^{2,} which is sufficient for the Level 1 traffic loading or non-surface layers specified by DOTD [25]. The incorporation of 2% $FeCl_3$ effectively improved the fracture and fatigue cracking resistance of asphalt mixtures containing 30% of RAP materials, as evidenced by the mechanical testing results of mixtures M6730 and M6730F. Mixture M6730F had comparable mechanical properties to conventional mixture M76.

Table 8. Summary of statistical analysis for results obtained from mechanical tests

Engineering property	Test	Conventional M76	M6730	M6730F	M6750F
Stiffness	Dynamic Modulus	ND	+	ND	+
Permanent Deformation	HWT	ND	+	ND	+
Moisture Damage	MiST + HWT	ND	+	ND	+
Fracture and Fatigue Cracking	SCB	ND	-	ND	-
	Cantabro	ND	-	ND	-
Low-Temperature Cracking	TSRST	ND	-	ND	-

Note: (ND) represents no statistical difference in performance; (+) represents better performance; (-) represents lower performance; HWT: Hamburg Wheel Tracker; MiST: Moisture-induced Stress Tester; SCB: Louisiana Semi-circular Bend test; TSRST: Thermal Stress Restrained Specimen Tensile Strength test.

Approach 2: Recycling Agents

Petroleum-based and bio-derived RAs were utilized to enhance the performance of high-RAP asphalt mixtures. Six types of RAs were considered for this study: petroleum-derived aromatic oil, soy oil, and four types of tall-oil-derived phytosterol (industrial by-product, intermediate, purified, and fatty-acid-based).

Asphalt Binder Test Results

The six RAs were incorporated into asphalt mixtures containing 30% RAP material by total mixture weight. RAs' dosages were optimized using the binder blending tool, based on the properties of the RAP and unmodified virgin binder, to produce a target PG 70-22 asphalt binder when incorporated in asphalt mixtures containing 30% RAP (RBR of 0.28); see Table 9. For reference and comparison, a control mixture was prepared that contained asphalt binder PG 70-22 (SBS modified) was included. It is noted that all mixtures were designed to have similar volumetrics within Louisiana DOTD specifications' tolerances [25].

For each mixture, a suite of mechanical tests was conducted to evaluate high RAP asphalt mixture performance containing RAs. Further, balanced mixture design (BMD) criteria specified by DOTD [25] were utilized as a threshold for accepting asphalt mixture engineering performance.

Table 9. Asphalt mixtures containing 30% RAP and RAs' dosages (target PG 70-22)

Mix ID	Mix Code	Virgin Asphalt Binder	RBR	Recycling Agent	RA dosage, %
Mix 70	M70	PG 70-22	0	None	None
Mix 1	M6730RA1	PG 67-22	0.28	Reclamite B	12
Mix 2	M6730RA2	PG 67-22	0.28	Soy oil	4
Mix 3	M6730RA3	PG 67-22	0.28	Soy oil + Tall oil (byproduct)	2.5 +10
Mix 4	M6730RA4	PG 67-22	0.28	Soy oil + Tall oil (intermediate)	3 +7.5
Mix 5	M6730RA5	PG 67-22	0.28	Soy oil + Tall oil (Sterols)	4 +5
Mix 6	M6730RA6	PG 67-22	0.28	Tall oil-derived fatty-acid-based oil	3.9

Note: RBR: Recycled Binder Ratio; RA: Recycling agent; M: Asphalt mixture; PG: Performance grade of asphalt binder.

For each asphalt mixture studied, the properties of the component materials in Table 9 were incorporated into the developed binder-blending tool to determine the required dosage of each RA to be blended with PG 67-22 asphalt binder, yielding a PG 58-28 asphalt binder. In other words, the dosages of an RA were selected to yield a PG 58-28 asphalt binder when blended with the unmodified PG 67-22 asphalt binder. The PG 58-28 asphalt binder was then blended with the RAP binder (RBR = 0.28) to yield a target asphalt binder of PG 70-22. Similarly, the previous six asphalt mixtures were prepared with 50% RAP content and RAs’ dosages to obtain a blended asphalt binder of PG 76-22 [61]; see Table 10.

Table 10. Asphalt mixtures containing 50% RAP and RAs' dosages (target PG 76-22)

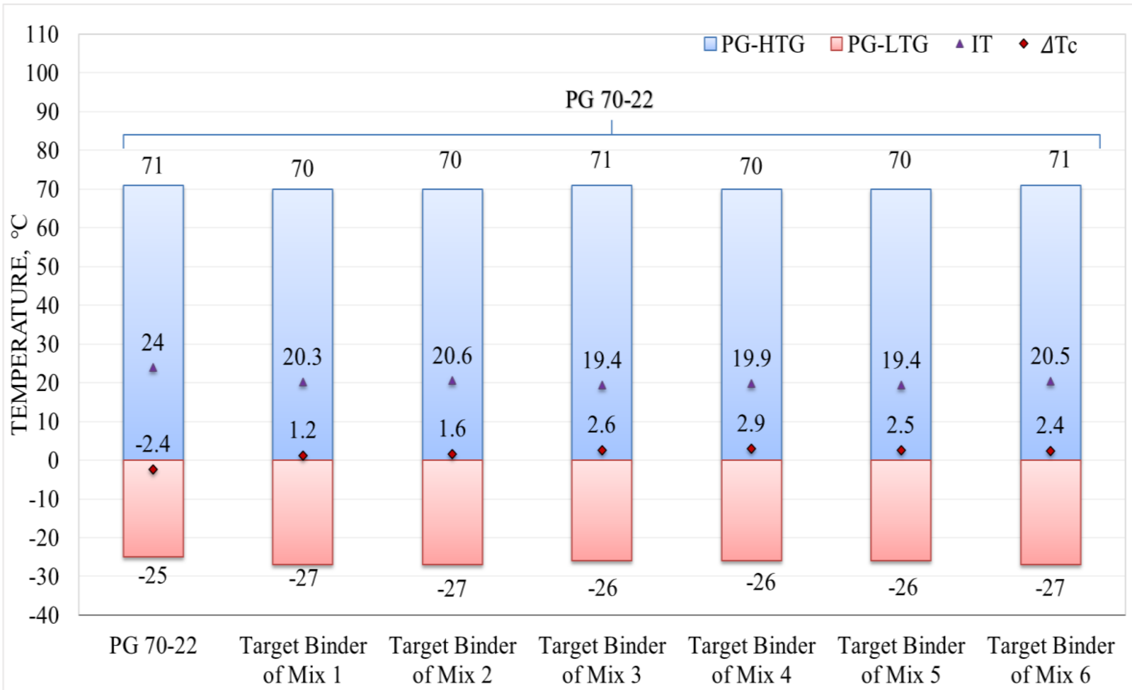
Mix ID	Mix Code	Virgin Asphalt Binder	RBR	Recycling Agent	RA dosage, %
Mix 76	M76	PG 76-22	0	None	None
Mix 1	M6750RA1	PG 67-22	0.46	Reclamite B	12
Mix 2	M6750RA2	PG 67-22	0.46	Soy oil	4
Mix 3	M6750RA3	PG 67-22	0.46	Soy oil + Tall oil (byproduct)	2.5 +10
Mix 4	M6750RA4	PG 67-22	0.46	Soy oil + Tall oil (intermediate)	3 +7.5
Mix 5	M6750RA5	PG 67-22	0.46	Soy oil + Tall oil (Sterols)	4 +5
Mix 6	M6750RA6	PG 67-22	0.46	Tall oil-derived fatty-acid-based oil	3.9

Note: RBR: Recycled Binder Ratio; RA: Recycling agent; M: Asphalt mixture; PG: Performance grade of asphalt binder.

Performance Grading (PG)

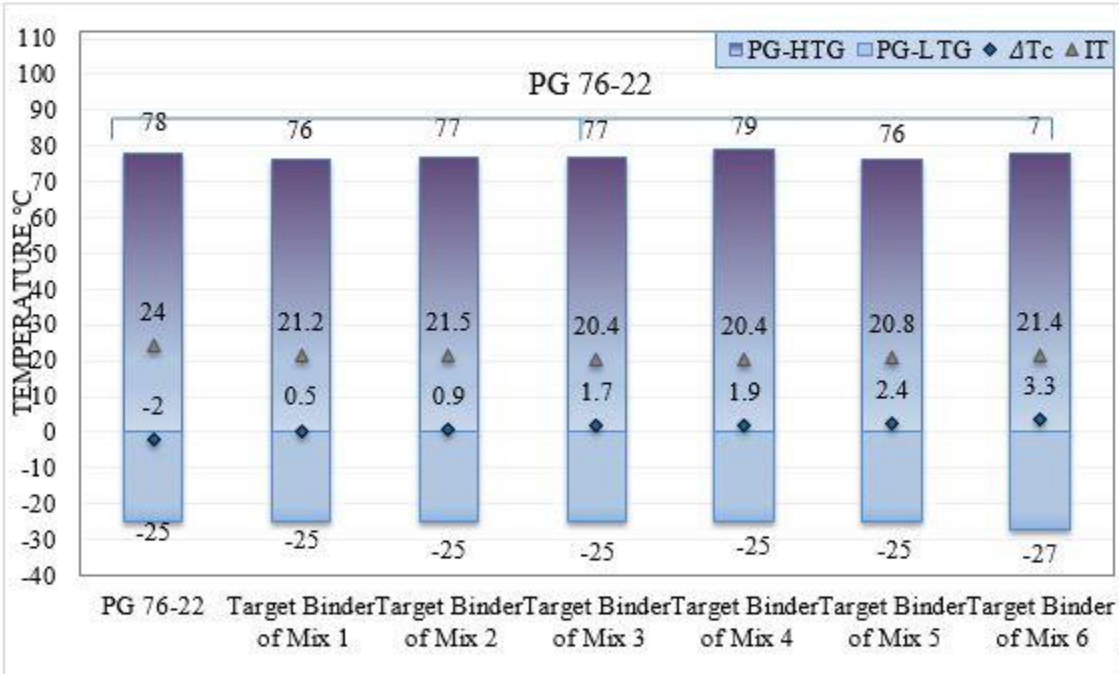
Figure 23 presents the PG of asphalt binders extracted from the control mixture Mix 70 and Mix 1 to Mix 6, which achieved similar target asphalt binder PGs. All blended asphalt binders yielded a PG 70-22; however, the intermediate temperatures in RAP mixtures were found to be less than the conventional mixture containing PG 70-22 asphalt binder without RAP or RAs. Additionally, ΔTc showed that relaxation in RAP mixtures, or m-value, was more critical than stiffness in terms of resisting low temperature cracking, contrary to the control mixture.

Figure 23. Target asphalt binders in 30% RAP mixtures (binder blending tool)



Asphalt mixtures with 50% RAP had a similar target asphalt binder of PG 76-22 by optimizing the RAs' dosages; see Figure 24. All blended asphalt binders yielded a PG 76-22; however, the intermediate temperatures in RAP mixtures were found to be less than the conventional mixture containing PG 76-22 asphalt binder without RAP or RAs. Additionally, ΔTc showed that relaxation in RAP mixtures, or m-value, was more critical than stiffness in terms of resisting low temperature cracking, contrary to the control mixture.

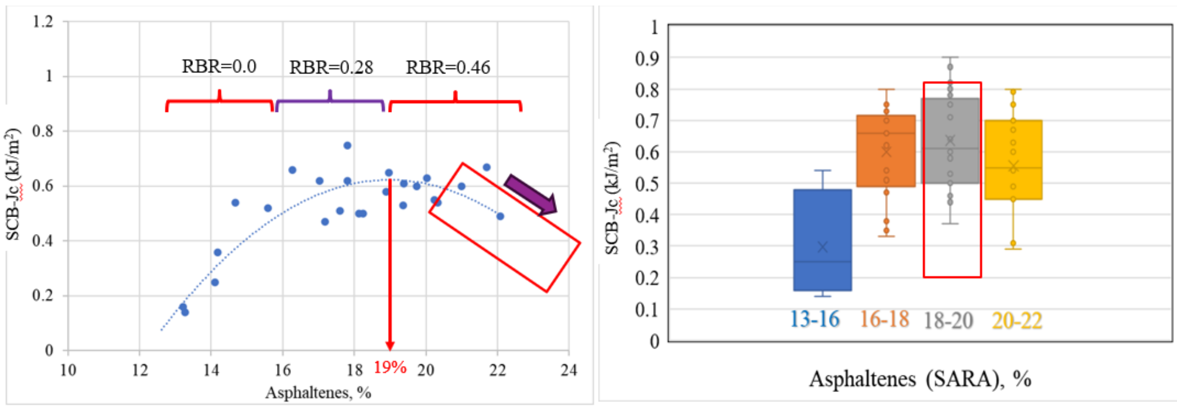
Figure 24. Target asphalt binders in 50% RAP mixtures (binder blending tool)



SARA Analysis

Each SARA fraction for all extracted asphalt binders was plotted against the cracking resistance (SCB J_c) of associated asphalt mixtures. The asphaltenes fraction was the only fraction that showed a strong correlation with the cracking resistance of the asphalt mixture; see Figure 25. The asphaltene fraction showed a proportional correlation with cracking resistance of asphalt mixtures up to 18%, followed by a plateau region (18–20 %) then a reduction. Notably, for virgin asphalt mixtures (i.e., those containing no RAP) and 30% RAP mixtures with RBR up to 0.28, a positive trend between asphaltenes and cracking resistance was observed. However, the trend is negative for 50% RAP mixtures (RBR = 0.46), where the asphaltene fraction is higher than 20%; see Figure 25. A similar negative trend was also observed in a previous study with asphaltene levels of more than 22% [62]. The optimal asphaltene content, ranging from 18-20% as shown in the boxplot, yielded the highest asphalt mixture cracking resistance for the studied mixtures. These observations suggest that a soft or virgin asphalt binder with a low percentage of asphaltenes would not be stiff enough to withstand load-related cracking. Conversely, a higher percentage of asphaltenes would result in a more brittle binder, making it more prone to cracking.

Figure 25. Effect of asphaltene percentage on cracking resistance



Fourier Transform Infrared Spectroscopy (FTIR)

Figure 26 and Figure 27 present the carbonyl index (CI) values for the extracted asphalt binders from the studied asphalt mixtures. In this study, the carbonyl group (C=O, a carbon atom double-bonded to an oxygen atom) was evaluated in relation to aging and cracking resistance. A higher CI value indicates a higher asphalt binder aging level, which suggests the cracking susceptibility of the associated asphalt mixture. Figure 26 shows that all extracted asphalt binders exhibited significantly lower CI (Mix 1) or equivalent CI (Mixes 3, 4, and 6) compared to the conventional binder extracted from Mix 70, with the exceptions of Mix 2 and Mix 5. Thus, all RAs showed positive effects in terms of reducing the RAP binder brittleness, except for RA 2 and RA 5. Similarly, Figure 27 showed the same findings with asphalt binders extracted from 50% RAP asphalt mixtures.

Figure 26. Carbonyl index for asphalt mixtures containing 30% RAP

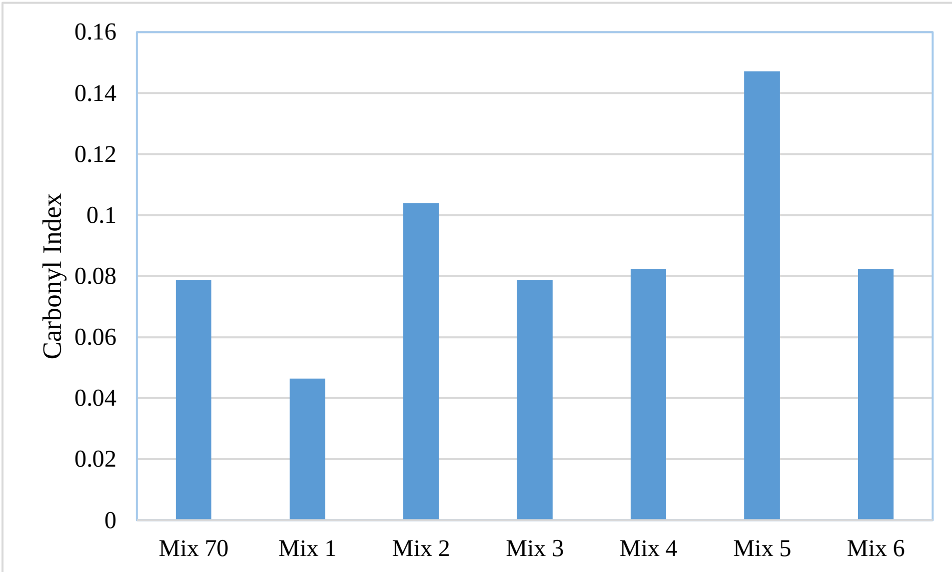
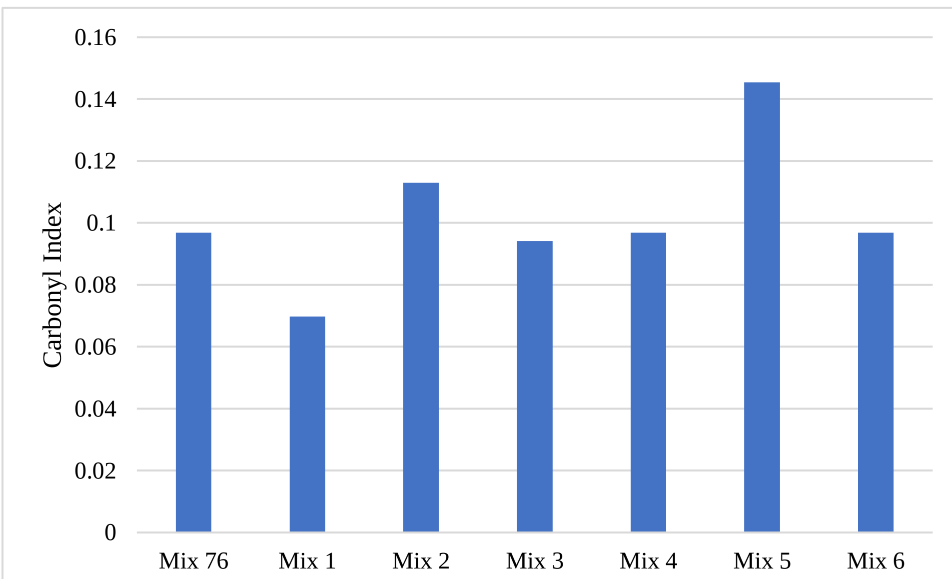


Figure 27. Carbonyl index for asphalt mixtures containing 50% RAP



Asphalt Mixture Test Results

Permanent Deformation

The HWT rut depths at 20,000 passes for the assessed 30% RAP mixtures are shown in Figure 28. The rut depth's coefficient of variation (CoV) ranged from 7-22%, with an overall average of 12.9%. All mixtures evaluated met the maximum HWT rut depth requirement. However, the control mixture Mix 70 showed statistically higher rut depth compared to other RAP mixtures evaluated. Further, Mix 4, containing RA 4, exhibited statistically better rutting resistance when compared to other RAP mixtures. These findings indicate that the addition of RAP materials could stiffen asphalt mixtures, even though a soft asphalt binder (PG 67-22) and RAs were used. It also implies that the use of RAs did not negatively impact the permanent deformation resistance. The studied asphalt mixtures exhibited stripping inflection point values beyond 20,000 passes, indicating that those mixtures were moisture-damage-resistant.

Figure 28. HWT rutting depths for 30% RAP mixtures

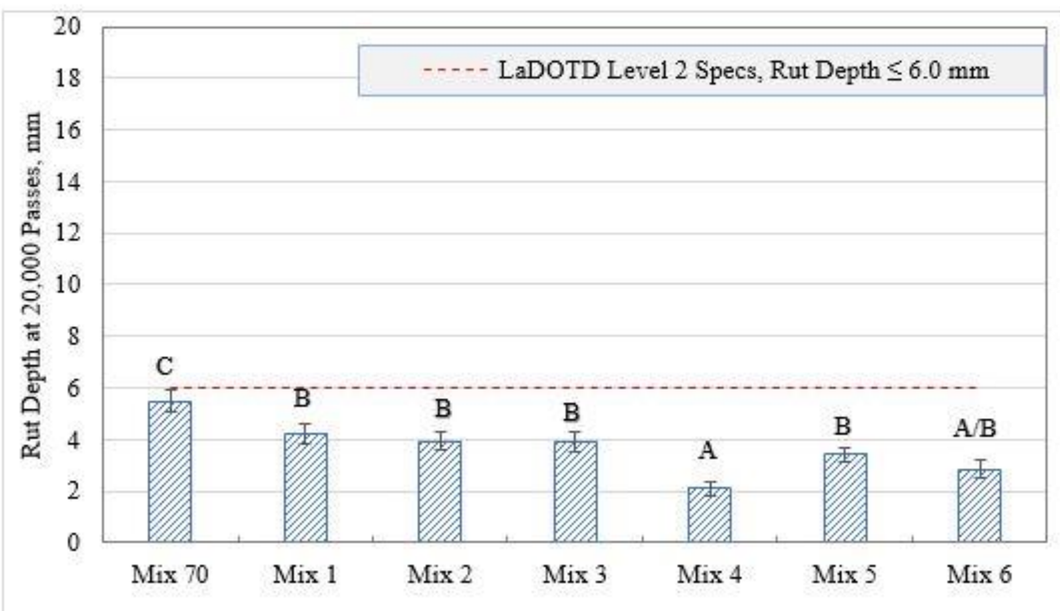
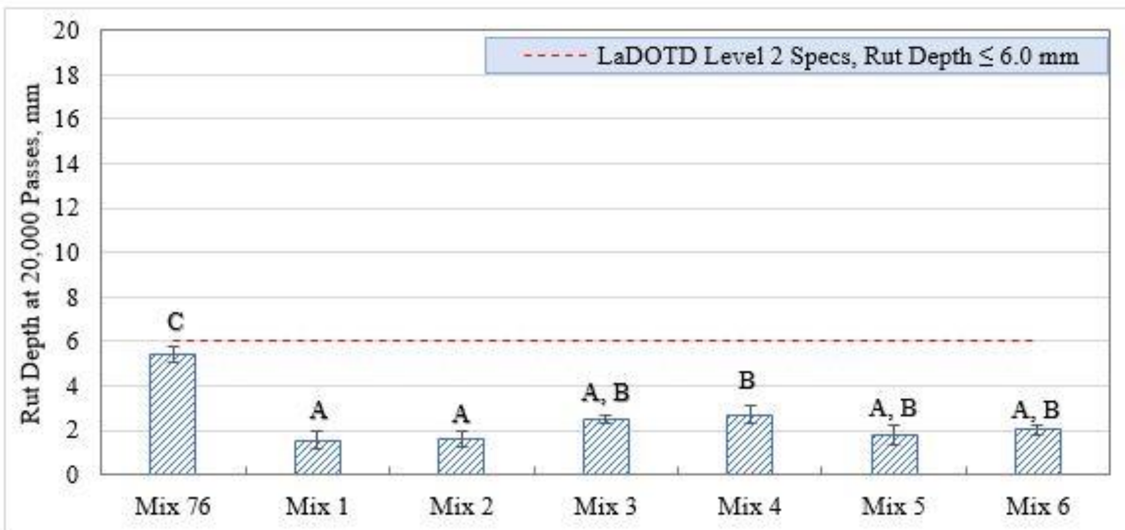


Figure 29 presents HWT rut depths at 20,000 passes for the asphalt mixtures evaluated. The coefficient of variation (CoV) of the rut depths varied between 7-13.5%, with an overall average of 10%. All mixtures evaluated met the maximum rut depth requirement specified by DOTD [25]. However, the control mixture Mix 76 showed higher rut depth at 20,000 passes compared to other mixtures evaluated, indicating that the addition of RAP materials could stiffen asphalt mixtures, even though a softer asphalt binder (PG 67-22) and RAs were used. It also implies that the use of RAs did not negatively impact the permanent deformation. It is noted that the studied asphalt mixtures exhibited a stripping inflection point (SIP) value beyond 20,000 passes, indicating that those mixtures were moisture-resistant.

Figure 29. HWT rutting depths for 50% RAP mixtures



Cracking Resistance

The critical strain energy release rate (J_c) values for the studied asphalt mixtures, obtained from the SCB J_c test, are shown in Figure 30. The averaged CoV for the strain energy (per-unit thickness) varied from 4-14%, with an overall average of 11%. Mix 1 and Mix 6 showed statistically similar SCB J_c values to Mix 70 and met the threshold of DOTD Level 2 mixture design [25]. Mixes 2 to 5 showed statistically lower SCB J_c values than Mix 70 and failed to meet the threshold of the DOTD Level 2 mixture design [25]. This implies that only asphalt mixtures containing 30% RAP content (RBR of 0.28) and RAs 1 and 6 complied with the DOTD specifications in terms of cracking resistance.

Figure 30. SCB J_c results of 30% RAP mixtures

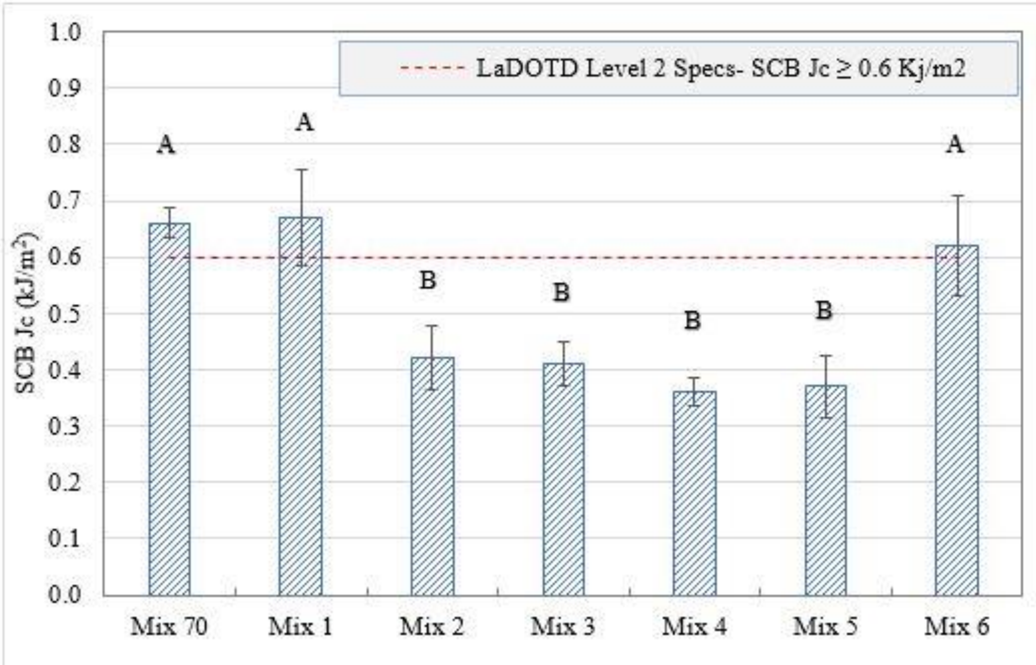
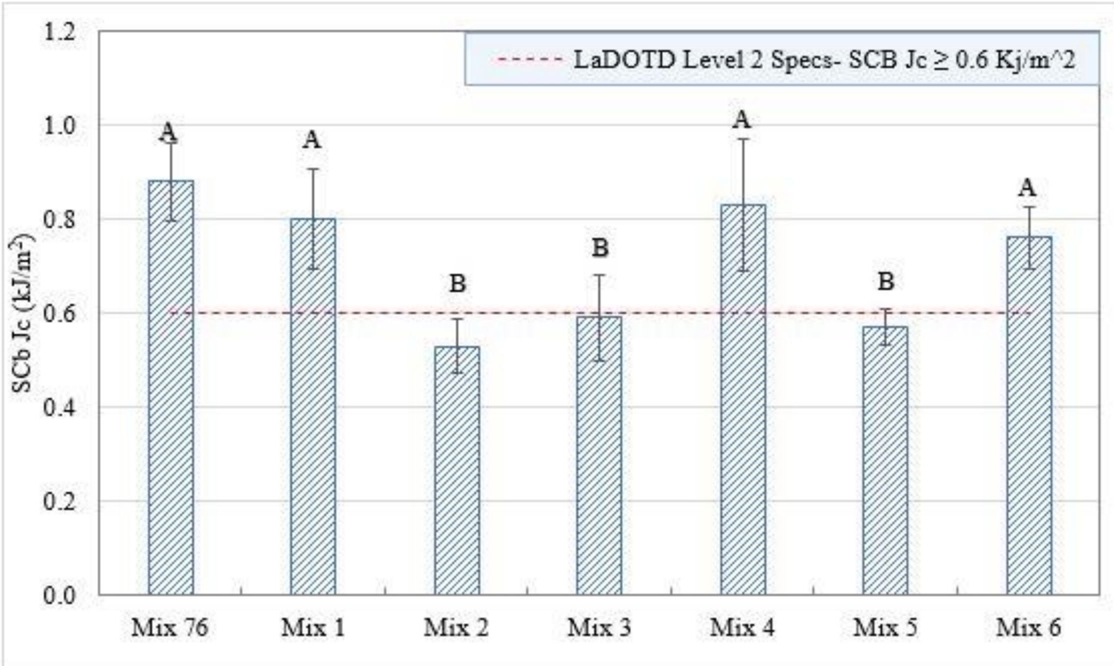


Figure 31 presents the SCB J_c values for the 50% RAP mixtures containing different RAs. The averaged CoV for the SCB J_c values ranged from 7-17%, with an overall average of 13% for mixtures evaluated. A higher J_c value is desired for intermediate-temperature crack resistance. DOTD specifies a "GO/NO-GO" minimum SCB J_c of 0.6 kJ/m^2 for Level 2 mixtures [25]. Level 2 mixtures are designed for traffic volumes greater than 3 million ESALs [25]. Mixes 1, 4, and 6 showed statistically similar SCB J_c values to Mix 76 and met the threshold for DOTD Level 2 mixture design. Mixes 2, 3, and 5 showed statistically lower SCB J_c values than Mix 76 and failed to meet the threshold for DOTD Level 2 mixture design. This implies that only asphalt mixtures containing high RAP content (RBR of 0.46) and utilizing RAs 1, 4, and 6 complied with DOTD specifications in terms of cracking resistance.

Figure 31. SCB J_c results for 50% RAP mixtures



Optimizing Dosages of RAs

RAs' dosages were initially optimized to yield asphalt binders equivalent to PG 70-22 and PG 76-22 asphalt binders when added to 30% and 50% RAP mixtures, respectively. However, the SCB J_c results showed inconsistency in meeting the cracking resistance criteria. In other words, two RAP-modified mixtures with the same asphalt binder grade could exhibit different cracking resistance. Consequently, asphalt mixtures containing various dosages of each RA were subjected to SCB testing to determine the optimum actual RAs' dosages. Table 11 and

Table 12 show the effect of changing RA dosages on cracking resistance (SCB J_c) for 30% and 50% RAP mixtures, respectively.

Table 11. Effect of RAs' dosage rate on cracking resistance in 30% RAP mixtures

RA	Dosage, %	SCB- J_c (kJ/m^2)	Continuous PG
RA1: Petroleum-derived aromatic oil using maltene blend (Reclamite Base)	0.0	0.37	PG 76.0-22.1
	6.0	0.90	PG 72.5-24.6
	12.0	0.67	PG 69.1-26.8
RA2: Modified soy-based oil	0.0	0.37	PG 76.0-22.1
	0.5	0.50	PG 75.2-23.7
	1.0	0.83	PG 74.4-24.1
	2.0	0.75	PG 73.0-25.2
	4.0	0.42	PG 70.5-26.7
RA3: Modified soy-based oil / Tall oil-derived phytosterol (by-product)	0.0	0.37	PG 76.0-22.1
	1.25/5.0	0.51	PG 72.0-24.6
	2.5/10.0	0.37	PG 69.3-25.9
RA4: Modified soy-based oil / Tall oil-derived phytosterol intermediate	0.0	0.37	PG 76.0-22.1
	1.5/3.75	0.90	PG 72.1-24.8
	3/7.5	0.41	PG 68.1-26.0
RA5: Modified soy-based oil / Tall oil-derived purified phytosterol (Sterols)	0.0	0.37	PG 76.0-22.1
	2.0/2.5	0.80	PG 72.4-24.7
	4.0/5.0	0.36	PG 69.0-26.3
RA6: Tall oil-derived fatty-acid-based oil	0.0	0.37	PG 76.0-22.1
	1.95	0.71	PG 72.4-24.8
	3.9	0.62	PG 70.6-26.9
RA7: Tall oil-derived phytosterol (by-product)	0.0	0.37	PG 76.0-22.1
	1.0	1.00	PG 75.7-23.3
	3.0	0.76	PG 74.8-24.3
	5.0	0.85	PG 73.8-24.4

Note: RBR: Recycled Binder Ratio; RA: Recycling agent; SCB: Semi-circular Bend test; PG: Performance grade of asphalt binder.

Table 12. Effect of RAs' dosage rate on cracking resistance in 50% RAP mixtures

RA	Dosage, %	SCB- J_c (kJ/m^2)	Asphalt mixture PG
RA1: Petroleum crude oil-derived aromatic oil using maltene blend (Reclamite Base)	0.0	0.29	PG 80.5-20.8
	6.0	0.45	PG 78.0-22.7
	12.0	0.80	PG 75.4-24.4
RA2: Modified soy-based oil	0.0	0.29	PG 80.5-20.8
	1.0	0.37	PG 79.3-22.4
	2.0	0.55	PG 78.3-23.1
	4.0	0.53	PG 76.3-24.5
	6.0	0.63	PG 74.3-26.8
	8.0	0.78	PG 74.3-28.1
RA3: Modified soy-based oil / Tall oil-derived phytosterol (by-product)	0.0	0.29	PG 80.5-20.8
	1.25/5.0	0.80	PG 77.5-22.9
	2.5/10.0	0.59	PG 75.4-24.4
RA4: Modified soy-based oil / Tall oil-derived phytosterol intermediate	0.0	0.29	PG 80.5-20.8
	1.5/3.75	1.00	PG 77.6-22.9
	3/7.5	0.83	PG 74.5-24.5
RA5: Modified soy-based oil / Tall oil-derived purified phytosterol (Sterols)	0.0	0.29	PG 80.5-20.8
	2.0/2.5	0.50	PG 77.8-23.0
	4.0/5.0	0.60	PG 75.2-24.5
RA6: Tall oil-derived fatty-acid-based oil	0.0	0.29	PG 80.5-20.8
	1.95	0.91	PG 78.5-22.8
	3.9	0.76	PG 76.4-24.6
RA7: Tall oil-derived phytosterol (by-product)	0.0	0.29	PG 80.5-20.8
	1.0	0.66	PG 81.5-21.7
	3.0	0.35	PG 80.8-22.6
	5.0	0.60	PG 80.0-23.2

Note: RBR: Recycled Binder Ratio; RA: Recycling agent; SCB: Semi-circular Bend test; PG: Performance grade of asphalt binder.

Based on the cracking resistance expressed in the SCB J_c results in Table 10 and Table 11, the RA dosages were selected per Table 12 and Figure 28.

Cracking Resistance at Optimized Dosages

Table 13, Figure 32, and Figure 33 present the SCB J_c values for the asphalt mixtures at the optimized dosage of their respective RAs.

Table 13. Optimized RAs' dosages based on cracking resistance

30% RAP mixtures			50% RAP mixtures		
RA	Dosage, %	SCB- J_c (kJ/m^2)	RA	Dosage, %	SCB- J_c (kJ/m^2)
RA1	6.0	0.90	RA1	12.0	0.80
RA2	1.0	0.83	RA2	8.0	0.75
RA3	1.25/5.0	0.51	RA3	1.25/5.0	0.80
RA4	1.5/3.75	0.90	RA4	1.5/3.75	1.00
RA5	2.0/2.5	0.80	RA5	2.0/2.5	0.60
RA6	1.95	0.71	RA6	1.95	0.91

Figure 32. SCB J_c results at optimized RAs' dosages for 30% RAP asphalt mixtures

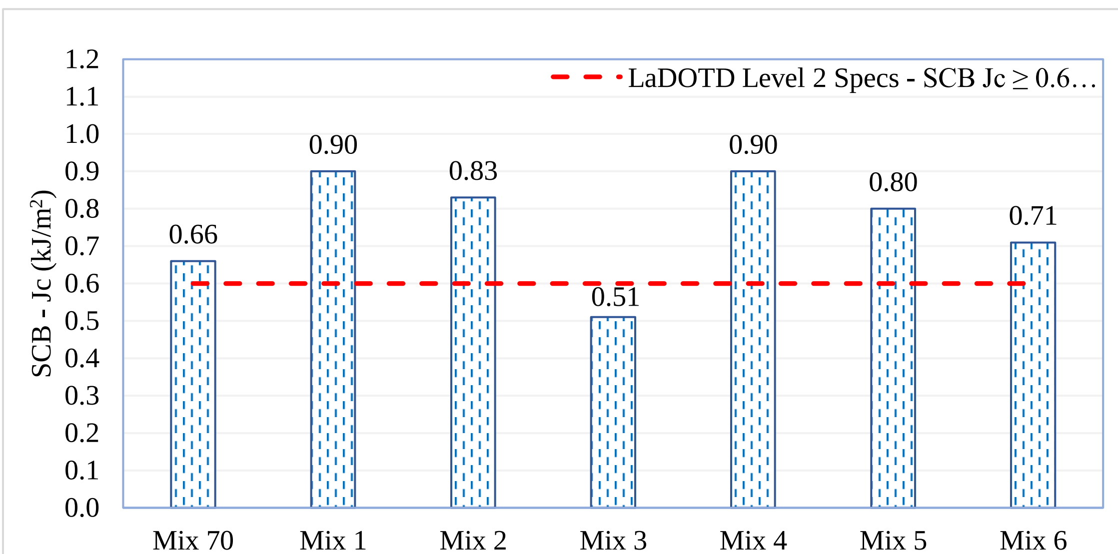
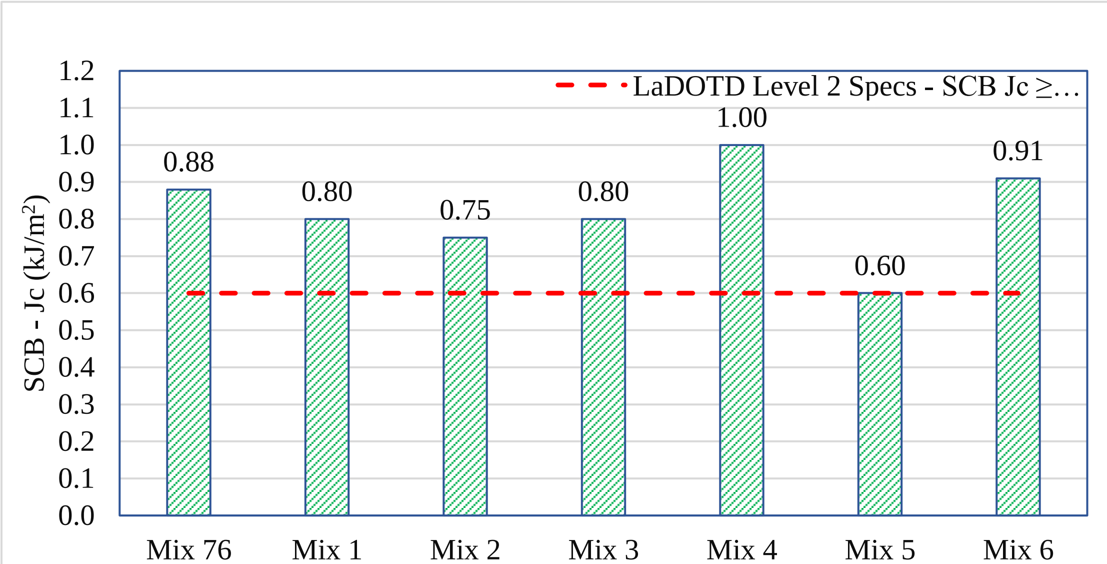


Figure 33. SCB J_c results at optimized RAs' dosages for 50% RAP asphalt mixtures



Permanent Deformation at Optimized Dosages

HWT tests were conducted for 30% RAP and 50% RAP mixtures at optimized dosages of their respective RAs. Figure 34 and Figure 35 present the HWT rut depth of RAP mixtures prepared at optimized dosages of their respective RAs, along with the conventional mixtures containing PG 70-22 and PG 76-22 asphalt binders. It is noted that all mixtures containing 50% RAP passed the rutting criteria specified by DOTD, except for Mix 5.

Figure 34. HWT test rut depths result at optimized RAs' dosages for 30% RAP asphalt mixtures

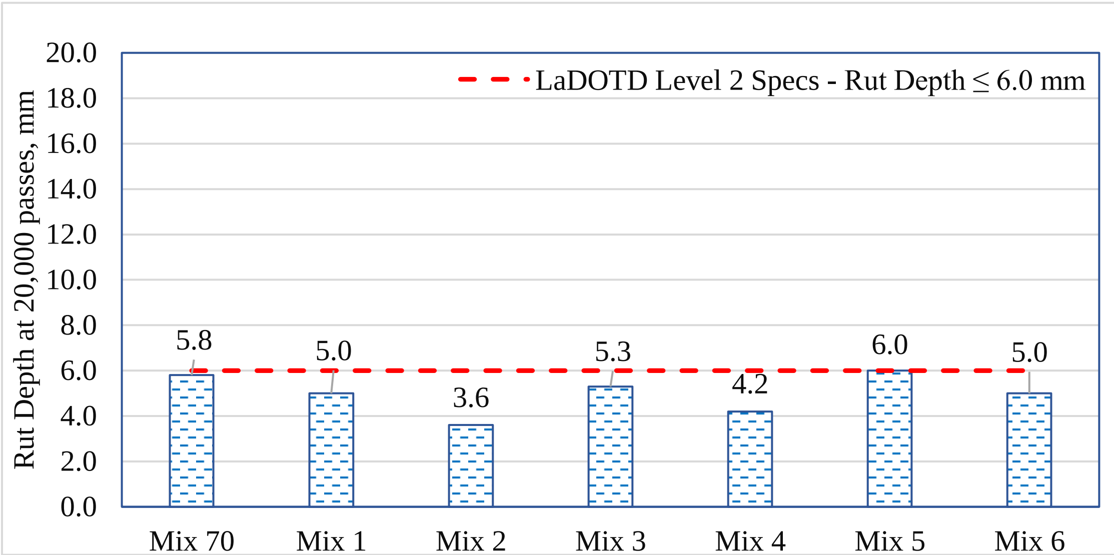
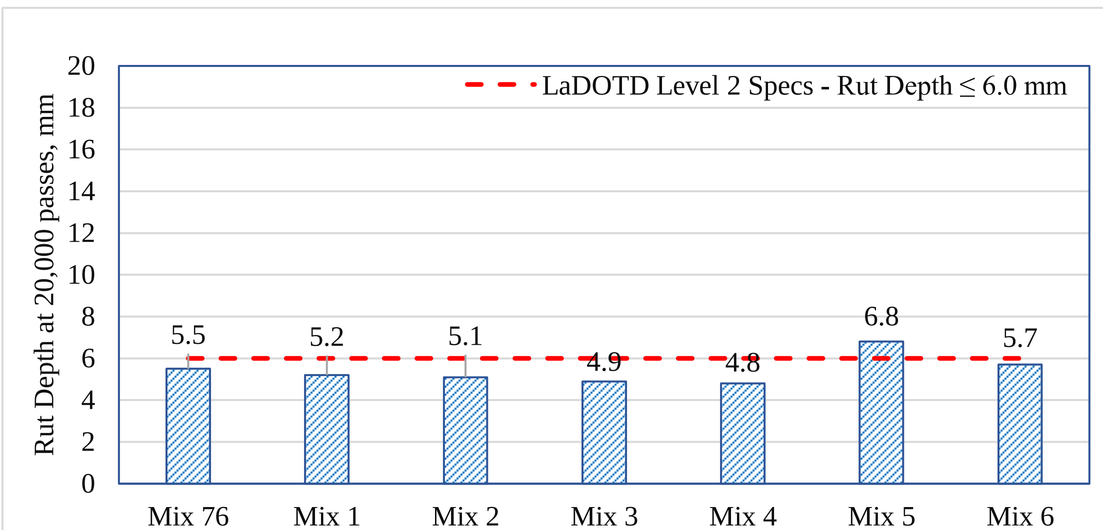


Figure 35. HWT test rut depths results at optimized RAs' dosages for 50% RAP asphalt mixtures

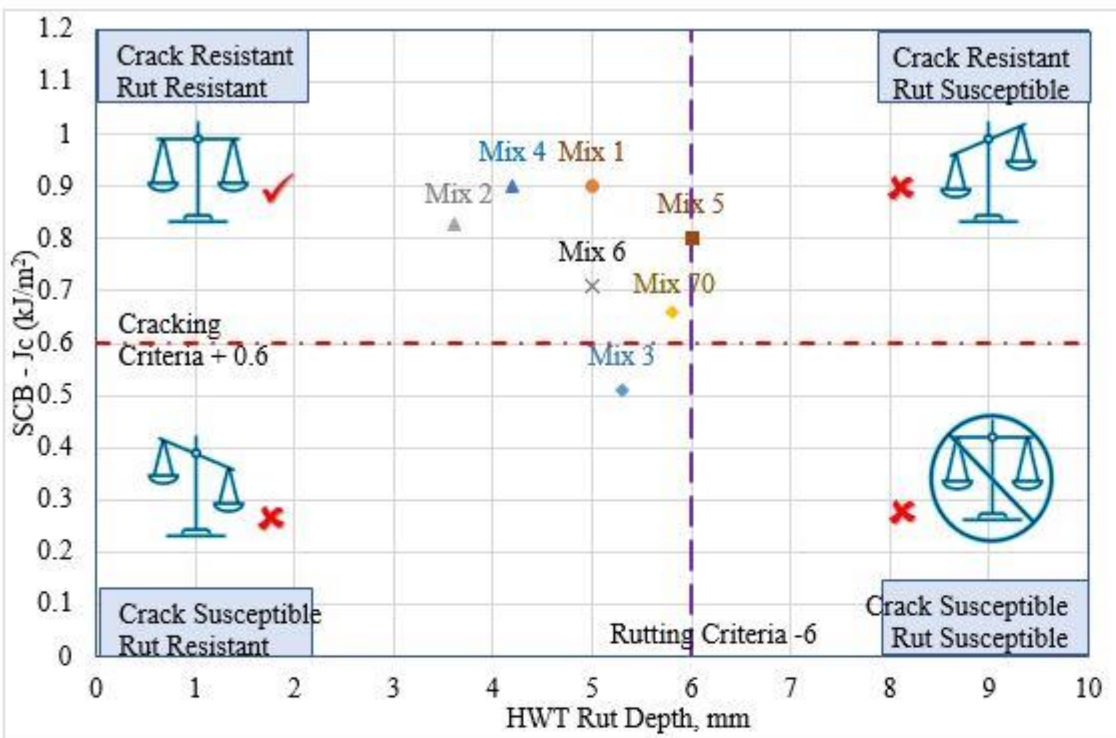


DOTD Balanced Mixture Design Criteria

Figure 36 and Figure 37 illustrate the DOTD balanced mixture design (BMD) framework for asphalt mixtures containing 30% and 50% RAP, respectively. In the DOTD's BMD approach, each produced asphalt mixture is subjected to stress tests to achieve a balance between cracking resistance (minimum SCB J_c value of 0.6 kJ/m^2 for Level 2 mixture design) and rutting resistance (maximum HWT rut depth of 6.0 mm for Level 2 mixture design) [25]. The horizontal dashed line in the figures represents the minimum cracking threshold, while the vertical dashed line represents the maximum rut-depth threshold. The BMD framework divides the performance space into four quadrants, as explained previously. Mixtures in the bottom-right quadrant fail to meet both rutting and cracking requirements. As expected, the control mixture Mix 70 met Louisiana's BMD criteria for Level 2, as shown in

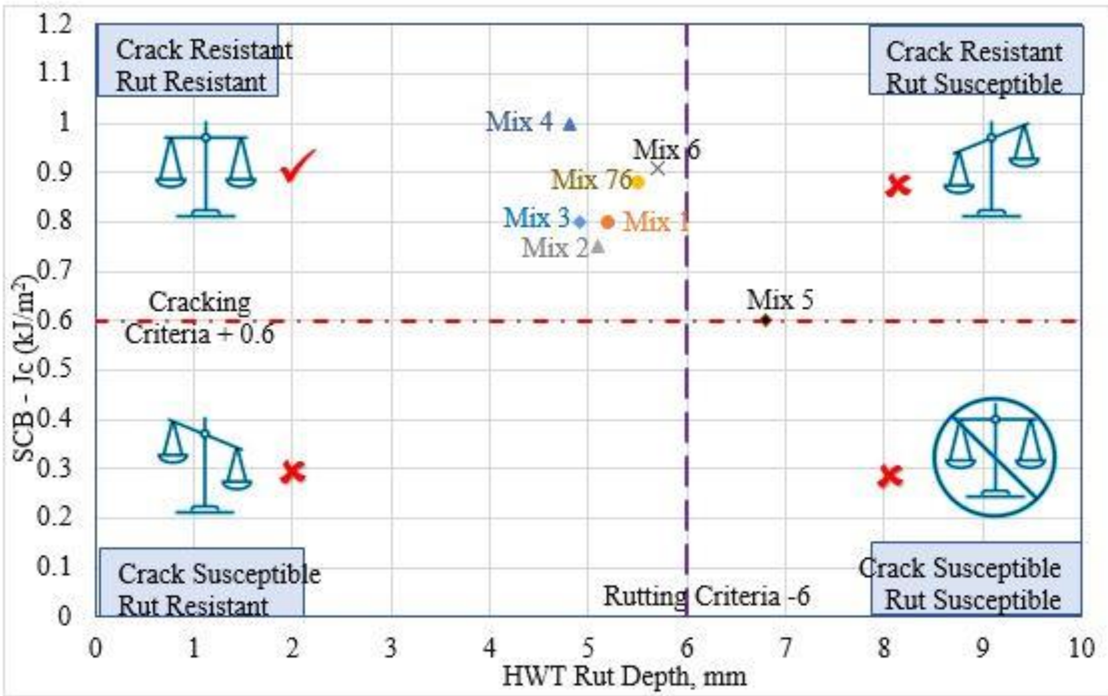
Figure 36. However, among the 30% RAP asphalt mixtures presented, Mix 3 did not meet the cracking criteria. This observation suggests that all rejuvenating agents (RAs) effectively enhanced the cracking resistance of the 30% RAP mixtures without negatively impacting rutting performance, except for RA 3. Mixture M3 contained an RA of soy oil blended with a tall oil-derived phytosterol containing industrial by-product.

Figure 36. Louisiana's balanced mix design framework for 30% RAP mixtures



The control mixture Mix 76 met Louisiana's BMD criteria for Level 2; see Figure 37. However, among the 50% RAP asphalt mixtures, Mix 5 failed to meet the rutting criteria. This observation indicates that all rejuvenating agents (RAs) were effective in enhancing the cracking resistance of the 50% RAP mixtures without compromising rutting performance, except for RA 5. Mixture M5 contained an RA of soy oil blended with tall oil-derived purified phytosterol, as presented in Figure 35.

Figure 37. Louisiana's balanced mix design framework for 50% RAP mixtures



Conclusion

The objective of this study was to assess the engineering performance of asphalt mixtures containing high RAP content (>25%). Three 12.5 mm NMAS asphalt mixtures with RAP contents of 0, 30, and 50% were evaluated. These RAP contents yielded RBRs of 0, 0.28, and 0.46, respectively. This study explored two approaches to address the challenges posed by hardened and oxidized RAP binders: 1) using a reagent catalyst (Lewis acid type—iron chloride, $FeCl_3$) to modify the chemical composition of the asphalt binder and disrupt the molecular associations formed in the aged RAP binder; and 2) using six types of Recycling Agents (RAs), (petroleum-derived aromatic oil, soy oil, and four types of tall-oil-derived phytosterol [industrial by-product, intermediate, purified, and fatty-acid-based]), to improve the cracking resistance of asphalt mixtures containing high RAP content.

The study included asphalt binder experiments such as chemical characterization using Fourier Transform Infrared Spectroscopy (FTIR) and saturates / aromatics / resins / asphaltenes (SARA) analysis, as well as rheological evaluation using Superpave performance grading. Additionally, the study included asphalt mixture experiments such as the dynamic modulus (DM) test to evaluate stiffness, Semi-circular Bend (SCB) test for fracture and fatigue resistance evaluation, Hamburg Wheel Tracking (HWT) test for assessing rutting resistance and moisture susceptibility, Thermal Stress-Restrained Specimen Tensile Strength (TSRST) test for evaluating low-temperature cracking resistance, and Cantabro abrasion test for assessing durability. The engineering performance acceptance criteria were evaluated under the balanced mix design (BMD) framework specified by Louisiana DOTD.

The results of the first approach, using $FeCl_3$, can be summarized as follows:

- **Stiffness.** The incorporation of $FeCl_3$ significantly decreased the stiffness of the mixture containing 30% RAP. Mixture M6730F showed similar stiffness to the conventional mixture M76.
- **Rutting and moisture damage resistance.** All studied mixtures met the requirements of the Louisiana specification. It is noted that the addition of $FeCl_3$ showed no adverse effect on rutting and moisture damage when comparing mixture M6730F with mixture M6730.
- **Intermediate temperature performance.** The incorporation of $FeCl_3$ improved the fracture and fatigue resistance of the asphalt mixture with 30% RAP. Mixture

M6730F showed comparable cracking resistance to the conventional mixture M76, which is widely used in Louisiana.

- **Durability-abrasion performance.** $FeCl_3$ enhanced durability by decreasing the abrasion loss percentage of M6730F (with $FeCl_3$) compared to the mixture M6730 (without $FeCl_3$).
- **Low-temperature performance.** The use of $FeCl_3$ effectively improved low-temperature cracking resistance. Notably, M6730F showed significantly better resistance to low-temperature cracking than mixture M6730 and similar performance to mixture M76. The addition of $FeCl_3$ to the mixture containing 50% RAP enhanced its low-temperature cracking performance to be similar to that of the mixture containing lower RAP content (M6730F).
- **Louisiana BMD framework.** Mixtures M76, M6730F, and M6750F complied with Louisiana's specifications. However, M6730 (30% RAP and no $FeCl_3$) did not meet the BMD criteria.

The results of the second approach, using RAs, can be summarized as follows:

- The evaluated mixtures complied with the Louisiana BMD criteria. The use of RAs did not negatively impact permanent deformation.
- As expected, mixtures containing RAP and RAs exhibited lower rut depth than the control mixtures due to the presence of the aged RAP binder.
- At optimized dosages, RAs were effective in mitigating cracking in asphalt mixtures containing 30% RAP and 50% RAP, as measured by the SCB J_c cracking test.

Acronyms, Abbreviations, and Symbols

Term	Description
AASHTO	American Association of State Highway and Transportation Officials
ANOVA	Analysis of Variance
ASTM	American Society of Testing and Materials
BBR	Bending Beam Rheometer
CoV	Coefficient of Variation
DOTD	Louisiana Department of Transportation and Development
DSR	Dynamic Shear Rheometer
FHWA	Federal Highway Administration
LA	Louisiana
LTRC	Louisiana Transportation Research Center
lb.	pound(s)
m	meter(s)
NMAS	Nominal Maximum Aggregate Size
PAV	Pressure Aging Vessel
PG	Performance Grade
RAP	Reclaimed asphalt pavement
RAS	Recycled asphalt pavement
RBR	Recycled Binder Ratio
RTFO	Rolling thin film oven
SARA	Saturates / Aromatics / Resins / Asphaltenes
SCB	Semi-circular Bend
TCE	Trichloroethylene
VECD	Viscoelastic continuum damage
VMA	Voids in the mineral aggregate

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