Connecting South Dakota and the Nation
South Dakota
Department of Transportation

U.S. Department of Transportation
Federal Highway Administration

SD2018-07-F

Evaluation of Asphalt Binder Rejuvenators for Use in Recycled Asphalt

SD2018-07
Final Report

Prepared by
National Center for Asphalt Technology at Auburn University
Office of Sponsored Programs
310 Samford Hall
Auburn, AL 36849-5131

August 2021

DISCLAIMER

The contents of this report, funded in part through grant(s) from the Federal Highway Administration, reflect the views of the authors who are responsible for the facts and accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of the South Dakota Department of Transportation, the State Transportation Commission, or the Federal Highway Administration. This report does not constitute a standard, specification, or regulation.

The South Dakota Department of Transportation provides services without regard to race, color, gender, religion, national origin, age or disability, according to the provisions contained in SDCL 20-13, Title VI of the Civil Rights Act of 1964, the Rehabilitation Act of 1973, as amended, the Americans With Disabilities Act of 1990 and Executive Order 12898, Federal Actions to Address Environmental Justice in Minority Populations and Low-Income Populations, 1994. Any person who has questions concerning this policy or who believes he or she has been discriminated against should contact the Department's Civil Rights Office at 605.773.3540.

ACKNOWLEDGEMENTS

The authors would like to acknowledge Ms. Pamela Turner from the National Center for Asphalt Technology for her assistance conducting the asphalt binder laboratory tests.

This work was performed under the supervision of the SD2018-07 Technical Panel:

Ken Swedeen ....DAPA
Daris Ormesher .... Research
Shea Lemmel ....Materials and Surfacing
Matt Stone.............................. Rapid City Region
Bob Longbons...................................... Research
Aaron Litka .... Research
Thad Bauer.. Research
Micah Howard..................................... Research
Phil Clements......................Project Development
Dave Huft .... Research
Bret Hestdalen. .... FHWA
Kevin Carlson..................................... Jebro, Inc.
Rick Rowen ....Materials and Surfacing

TECHNICAL REPORT STANDARD TITLE PAGE

<table>
<tr><td>1. Report No.
SD2018-07-F</td><td>2. Government Accession No.</td><td colspan="2">3. Recipient's Catalog No.</td></tr>
<tr><td colspan="2" rowspan="2">4. Title and Subtitle
Evaluation of Asphalt Binder Rejuvenators For Use in Recycled Asphalt</td><td colspan="2">5. Report Date
08/25/2021</td></tr>
<tr><td colspan="2">6. Performing Organization Code</td></tr>
<tr><td colspan="2">7. Author(s)
Carolina Rodezno, Raquel Moraes, Mawazo Fortunatus, Fan Yin</td><td colspan="2">8. Performing Organization Report No.</td></tr>
<tr><td colspan="2" rowspan="2">9. Performing Organization Name and Address
National Center for Asphalt Technology
at Auburn University
Office of Sponsored Programs
310 Samford Hall
Auburn, AL 36849-5131</td><td colspan="2">10. Work Unit No.</td></tr>
<tr><td colspan="2">11. Contract or Grant No.</td></tr>
<tr><td colspan="2" rowspan="2">12. Sponsoring Agency Name and Address
South Dakota Department of Transportation
Office of Research
700 East Broadway Avenue
Pierre, SD57501-2586</td><td colspan="2">13. Type of Report and Period Covered
Final Report
02/26/19-08/25/21</td></tr>
<tr><td colspan="2">14. Sponsoring Agency Code</td></tr>
<tr><td colspan="4">15. Supplementary Notes</td></tr>
<tr><td colspan="4">16. Abstract
This research evaluated the effectiveness of bio-based and petroleum-based rejuvenators in restoring the properties of asphalt binder blends and mixtures containing high content of reclaimed asphalt pavement (RAP). The rheological and chemical evaluation of binder blends included rotational viscosity, Superpave Performance Grading (PG), Multiple Stress Creep Recovery (MSCR), and Fourier Transform Infrared (FTIR) spectroscopy. The binder results indicated that the addition of RAP up to 50% of binder replacement to a PG 58-34 base binder resulted in increased cracking susceptibility after long-term aging, as indicated by the Glover-Rowe (*G-R*) parameter results. The addition of rejuvenators decreased the stiffness of the recycled asphalt binder blends and thus, improved the cracking susceptibility after long-term aging. This improvement was found as rejuvenator product dependent, and was usually influenced by the RAP content of each recycled asphalt binder blend. The petroleum-based rejuvenator (i.e., asphalt flux) was found as the product most susceptible to aging, indicating that this additive only acted as a softener and did not avoid further aging of the recycled asphaltic material. As expected, as the percentage of RAP increased (i.e., from 20% towards 50%) in the recycled asphalt binder blends, the growth of oxygen containing functionalities (i.e., carbonyl and sulfoxide groups) also increased. The reduction in oxygen-containing compounds due to the addition of rejuvenators was found dependent on the product type (i.e., composition), as well as recycled asphalt binder content.
The mixture performance evaluation included the Indirect Tensile Asphalt Cracking Test (IDEAL-CT) for intermediate-temperature cracking resistance, and Disc-Shaped Compact Tension Test DCT for low-temperature cracking resistance after being subjected to short-term oven aging (STOA) plus long-term oven aging (LTOA), and Asphalt Pavement Analyzer Test (APA) for rutting resistance after being subjected to STOA. Regarding mixture evaluation, APA and DCT results indicated that overall, the rejuvenated mixes had better rutting, moisture, and low-temperature cracking resistance than the control mixes. However, the rejuvenated mixes had reduced IDEAL-CT results than the control mixes, which indicated potentially increased susceptibility to intermediate-temperature cracking. Finally, the results of this research were used to develop a procedure to evaluate the effectiveness of rejuvenators in improving the performance of mixtures with high recycled asphalt material contents.</td></tr>
<tr><td colspan="2">17. Keywords</td><td colspan="2">18. Distribution Statement
No restrictions. This document is available to the public from the sponsoring agency.</td></tr>
<tr><td>19. Security Classification (of this report)
Unclassified</td><td>20. Security Classification (of this page)
Unclassified</td><td>21. No. of Pages
102</td><td>22. Price</td></tr>
</table>

TABLE OF CONTENTS

Table of Contents

LIST OF TABLES

LIST OF FIGURES

TABLE OF ACRONYMS

Acronym	Definition
AASHTO	American Association of State Highway and Transportation Officials
APA	Asphalt Pavement Analyzer
BBR	Bending Beam Rheometer
CT_{Index}	Cracking Tolerance Index
DCT	Disc-shaped Compact Tension Test
DOT	Department of Transportation
DSC	Differential Scanning Calorimetry
FHWA	Federal Highway Administration
FTIR-ATR	Fourier Transform Infrared Spectroscopy by Attenuated Reflectance
GC-MS	Gas Chromatography-Mass Spectrometry
G_f	Fracture Energy
GPC	Gel Permeation Chromatography
G_{sb}	Bulk specific gravity of the aggregate
G_{se}	Effective specific gravity of the aggregate
HMA	Hot Mix Asphalt
HWTT	Hamburg Wheel Tracking Test
IDEAL-CT	Indirect Tensile Asphalt Cracking Test
MSCR	Multiple-Stress Creep Recovery
N_{max}	Maximum number of gyrations at which the relative density is evaluated
NAPA	National Asphalt Pavement Association
NCAT	National Center for Asphalt Technology
NCHRP	National Cooperative Highway Research Program
NMAS	Nominal Maximum Aggregate Size
PAV	Pressure Aging Vessel
RBR	Recycled Binder Replacement
SBS	Styrene-Butadiene-Styrene
SDDOT	South Dakota Department of Transportation
RAM	Recycled Asphalt Materials
RAP	Reclaimed Asphalt Pavement
RAS	Recycled Asphalt Shingles
RAs	Recycling Agents
VFA	Voids Filled With Asphalt
VMA	Voids in Mineral Aggregate

1 EXECUTIVE SUMMARY

This research evaluated the effectiveness of rejuvenators in restoring the properties of asphalt blends and mixtures with high RAP contents. For this evaluation, rheological and chemical tests were utilized. These tests included: viscosity at 135°C, PG grading properties, Multiple Stress Creep Recovery (MSCR), and Fourier Transform Infrared Spectroscopy (FTIR). Asphalt blends with different RAP contents were tested with and without different rejuvenators. In addition to binder performance testing, this research also included mixture performance. Each asphalt mixtures was tested for rutting resistance (Asphalt Pavement Analyzer [APA]) after being subjected to short-term oven aging (STOA), and cracking resistance at intermediate temperature (Indirect Tensile Asphalt Cracking Test [IDEAL-CT]), and low temperature (Disc-Shaped Compact Tension Test [DCT]) after being subjected to STOA plus long-term oven aging (LTOA).

SUMMARY OF FINDINGS

Rotational Viscosity

- The viscosity of the asphalt base binder PG 58-34 increased with the addition of RAP binder, and this increase in viscosity was directly related to the RAP content.
- The addition of rejuvenators decreased the viscosity of the recycled binder blends. This decrease in viscosity was influenced by the rejuvenators' composition and dosage.
- The viscosity aging index results indicated that the addition of rejuvenators decreased the aging susceptibility of the recycled asphalt blends, with exception of the PG 58-34+50%RAP+2.0%RA_5 blend.

Performance Grading and ΔTc Parameter

- The addition of recycled asphalt binder to the PG 58-34 base binder increased the stiffness of the asphalt binders but reduced their fatigue resistance, thermal cracking resistance, and stress relaxation property after RTFO plus 60-hour of oxidative aging in the PAV.
- The incorporation of rejuvenators counterbalanced these negative effects with the exception of the blend PG 58-34 + 50% RAP + 2.0% RA_5.
- When considering 35% ABR, an improvement in the ΔTc parameter after RTFO plus 60-hour PAV was observed for all rejuvenated recycled binder blends. When considering 50% ABR, an improvement in the ΔTc parameter was observed for RA_1, RA_2, and RA_3. The addition of RA_5 resulted in a slightly more negative ΔTc.

MSCR

- J_{nr} and % Recovery MSCR testing parameters were found dependent on the constituents of the binder blends (i.e., base binder type, recycled binder percentage, and RA type and dosage).
- % Recovery parameter was found to be highly influenced by the creep compliance J_{nr} of the binders, regardless of the presence and dosage of rejuvenators.
- Rejuvenators increased the magnitude of J_{nr}. This increase varied depending on the chemical composition and dosage of the additives as well as the RAP content.

G-R Parameter and Black Space Diagram

- *G-R* parameter results highlighted the binder stiffening effect from RAP, since the recycled asphalt binder blends showed consistently higher *G-R* parameters than the control recycled binders.
- The addition of 50% RAP binder replacement to the PG 58-34 base binder resulted in failing the *G-R* parameter criterion of 600 kPa for the significant block cracking after RTFO plus 60-hour PAV aging.
- The addition of rejuvenators to the recycled binder blends decreased the stiffness of all blends, before and after aging. For the recycled binder blends after aging, this decrease in stiffness was accompanied by an increase in the phase angle (δ).
- After RTFO plus 60-hour PAV aging, regardless of the content of recycled binder (i.e., 35 or 50% RAP), the binder blends rejuvenated with RA_1, RA_3 and RA_4 were located below the G-R damage zone and thus, are not likely to experience premature block cracking in the field.
- G-R Aging Index calculated as the fraction of the G-R parameter of the RTFO plus 60-h PAV-aged sample over that of the unaged sample indicated that the bio-based rejuvenator RA_1 decreased the aging susceptibility of the recycled binder blends, regardless of the content of recycled binder (i.e., 35 or 50% RAP).
- Stiffness ratio results after long-term aging indicated that the all the rejuvenated recycled binder blends containing 35% RAP remained softer than the control recycled binder. When considering the rejuvenated recycled binder blends containing 50% RAP, rejuvenators RA_2 and RA_5 did not improve the stiffness of the control recycled binder.

FTIR

- As expected, as the percentage of RAP binder increased (i.e., from 20% towards 50%) in the recycled binder blends, the growth of oxygen containing functionalities (i.e., carbonyl and sulfoxide groups) also increased.
- When considering 35% ABR, the oxidation of the rejuvenated recycled binder blends containing RA_1, RA_2, and RA_4 remained below the oxidation of the PG 58-34 + 35% RAP control recycled binder. The opposite was observed for the rejuvenated recycled binder blends containing RA_3.
- When considering 50% ABR, rejuvenated recycled binder blends containing RA_1 and RA_5 presented a less degree of oxidation than the PG 58-34 + 35% RAP control recycled binder; while recycled binder blends containing RA_2 and RA_3 presented a higher degree of oxidation.
- The reduction in oxygen-containing compounds due to the addition of rejuvenators was found dependent on the RA type (i.e., composition) and dosage as well as the RAP binder content.

Mixture Performance Testing

APA

- All high RAP mixes with and without rejuvenators had less rut depth compared to the 20% ABR control mix, except for 35%ABR/RA_4 mix.
- Since none of the mixtures exceeded the 7 mm rut depth threshold required by South Dakota DOT, it is expected that all the mixtures would have adequate rutting performance.

IDEAL-CT

- CT_{index} results showed that the 20% ABR control mix had better intermediate temperature cracking resistance than the non-rejuvenated and rejuvenated 35% ABR and 50% ABR mixes. This can be partially attributed to the higher asphalt content of the control mix.
- The use of rejuvenators showed an improvement in the average CT_{Index} values for rejuvenated mixes when compared to non-rejuvenated mixes at the same ABR content. However, when the variability of the test was considered, the difference in CT_{Index} did not show a statistically significant improvement for the 35% ABR and 50% ABR mixes except for two cases: mix 35% ABR/No RA versus 35% ABR/RA_2 and mix 50% ABR/No RA vs 50% ABR/RA_3.
- Limited additional IDEAL-CT tests conducted with the 35%ABR mix and one rejuvenator suggested the following:
  - For this specific study, results have indicated that the rejuvenator dosages recommended by the suppliers may not be sufficient to improve the intermediate temperature cracking resistance of high RAP mixes. This could be a consequence of the approach followed by the suppliers to determine the rejuvenator dosage being too conservative. Selecting the rejuvenator dosage by targeting the low-temperature PG grade (-34°C) of the virgin binder (after RFTO plus 60-hour PAV aging) could be more appropriate but warrant further investigation.
  - Tests conducted on samples after STOA and LTOA suggested that the effectiveness of rejuvenators may diminish with aging.
  - When a 75% binder availability Factor (BAF) was used, RA_1 was effective on improving the cracking performance of the 35% ABR mix, at intermediate temperature. This could be an indication that the use of rejuvenators in combination with higher asphalt content may produce mixes with improved intermediate temperature cracking performance.

DCT

- Fracture energy results showed that for the 35%ABR mixes, all the mixes except the non-rejuvenated mix and the mix with RA_3 had higher mean values compared to the 20%ABR mix. The non-rejuvenated mix had the lowest mean fracture energy. For the 50% ABR mixes, all the mixes had lower mean fracture energy values compared to the 20%ABR mix. In addition, except for 50% ABR/RA_3 mix, the mean fracture energy of all rejuvenated mixes were higher compared to the fracture energy of the corresponding non-rejuvenated mix.
- Statistical analysis showed that for the 35% ABR mixes, significant differences only exist between fracture energy of the non-rejuvenated mix versus mixes with RA2 and RA4.On the other hand, statistical analysis indicated that all the 50% ABR mixes with and without rejuvenators had similar fracture energy results and thus, are expected to have equivalent resistance against thermal cracking.

The results of this research were used to develop a procedure to evaluate rejuvenators in improving the performance of mixtures with high recycled asphalt material contents. It is important to note that this procedure was developed based on binder and mixture test results corresponding to a limited set of raw materials (i.e., one virgin binder, one aggregate source, and one RAP source). Therefore, it is highly recommended that this procedure be re-verified using tests results conducted with additional materials used in South Dakota.

2 PROBLEM DESCRIPTION

The South Dakota Department of Transportation (SDDOT) routinely uses reclaimed asphalt pavement (RAP) in hot mix asphalt (HMA) because of the significant economic and environmental benefits, including cost savings, conservation of natural resources, reduction in energy consumption and emissions, and recycling of construction materials that otherwise would be disposed in landfills.

There is a general agreement that while the stiffness and rutting resistance of asphalt mixtures increase with increasing RAP percentage, the mixtures tend to have reduced cracking resistance and durability due to the heavily aged RAP asphalt binders that are stiffer and more brittle than virgin binders. Because of this limitation, SDDOT currently allows up to 20 % RAP by weight of aggregates to be used in mainline mix designs.

A wide range of products known as asphalt binder rejuvenators exist on the market that claim to decrease the viscosity of aged binders and restore their rheological properties, allowing a higher percentage of RAP to be used. Since the components and formulation of rejuvenators vary from one product to another, research is needed to evaluate the effectiveness of rejuvenators with South Dakota conditions and materials. The study should determine if rejuvenators could increase the flexibility of recycled binders in HMA and what effect they would have on the long-term performance of asphalt mixtures. Additionally, the study should develop a laboratory testing protocol for SDDOT to evaluate rejuvenators other than those included in this study.

2.1 Research Objectives

The three research objectives of this project were:

1. Compile and evaluate data on the state of practice for use of recycled asphalt binder rejuvenators.
2. Develop a testing protocol or procedure that can be used to evaluate recycled asphalt binder rejuvenators.
3. Evaluate a minimum of five binder rejuvenation products that could routinely be used in South Dakota.

2.2 Tasks Description

This research project included the following tasks.

Task 1: Meet with the project's technical panel to review the project scope and work plan.

The project was initiated with a meeting of the co-principal investigator, Dr. Fan Yin, with the technical panel to review and refine the proposed work plan. This meeting took place on May 29, 2019.

Task 2: Review and summarize literature regarding use of asphalt binder rejuvenators.

The research team conducted a comprehensive literature review to identify and synthesize existing studies on the use of asphalt binder rejuvenators. Review topics included:

- Classification of asphalt binder rejuvenators;
- Determination of the optimum dosage of rejuvenators;
- Rheological characterization of recycled asphalt binders with rejuvenators;
- Chemical characterization of recycled asphalt binders with rejuvenators;
- Laboratory characterization of recycled asphalt mixtures with rejuvenators; and
- Field performance of recycled asphalt mixtures with rejuvenators.

Task 3: Based on the results of Task 2, develop a survey instrument to be sent to selected U.S. state and Canadian provincial transportation departments to determine how they evaluate and use asphalt binder rejuvenators.

The research team developed a survey of U.S. state and Canadian provincial highway agencies to determine how they evaluate and use asphalt binder rejuvenators.

Task 4: Submit a technical memorandum to the technical panel detailing the results of Task 2 and 3.

Upon completion of Task 2 and 3, the research team submitted a technical memorandum with the literature review and survey results to the technical panel for review. This memorandum was submitted on July 1, 2019.

Task 5: Upon approval of the survey instrument, conduct the survey and analyze results.

Once the survey was approved by the technical panel, it was sent to U.S. state and Canadian provincial agencies to gather information about practices to evaluate the use of rejuvenators. Upon completion of the survey, the research team analyzed the responses.

Task 6: Based on literature search and survey results, propose testing procedures for both asphalt binders and asphalt concrete mixes to evaluate the effectiveness of asphalt binder rejuvenators, including methods to predict their long-term effect on pavement performance.

As part of Task 6, the research team developed a testing plan for both asphalt binders and asphalt mixtures to evaluate the effectiveness of rejuvenators in restoring the rheological properties of recycled asphalt binder.

Task 7: Submit a technical memorandum to the technical panel detailing the results of Task 5 & 6.

At the conclusion of Task 6, the research team submitted a technical memorandum to the panel detailing the results of the survey and the complete testing plan. This memorandum was submitted on December 16, 2019. The research team received comments from the technical panel on May 17, 2020. A revised memorandum addressing comment from the technical panel was submitted on May 29, 2020.

Task 8: Conduct testing using the procedure approved in Task 6.

Once the proposed testing plan was approved, the research team executed the work plan.

Task 9: Submit a technical memorandum to the technical panel detailing the results of Task 8.

The research team submitted a technical memorandum for this task on May 28, 2021.

PHASE IV

Task 10: Develop recommendations for rejuvenator evaluation and use, including any new specifications and test methods.

The research team developed a procedure for SDDOT to select appropriate rejuvenators to be used in conjunction with recycled asphalt materials.

Task 11: In conformance with Guidelines for Performing Research for the South Dakota Department of Transportation, prepare a final report summarizing the research methodology, findings, conclusions, and recommendations.

This final report contains an executive summary, problem statement, project objectives, literature review, work plan, results of the executed work plan, findings, conclusions, and recommendations. The final report follows the *Guidelines for Performing Research for the South Dakota Department of Transportation.*

Task 12: Make an executive presentation to the South Dakota Department of Transportation Research Review Board at the conclusion of the project.

The research team delivered a closeout executive presentation at the September 1, 2021 Research Review Board meeting.

3 LITERATURE REVIEW ON THE USE OF REJUVENATORS WITH RECYCLED ASPHALT

3.1 Background

The use of recycled asphalt materials, including reclaimed asphalt pavements (RAP) and recycled asphalt shingles (RAS), has significant economic and environmental benefits, including cost savings, conservation of natural resources, and reduction in energy consumption and emissions. According to the most recent National Asphalt Pavement Association (NAPA) survey, the total estimated tons of RAP and RAS used in asphalt mixes in 2017 were 76.2 million tons and 944,000 tons, respectively (Williams et al., 2017). Furthermore, the reported average RAP and RAS contents in state project mixes were 20 percent and 0.3 percent, respectively. There is a general agreement among the asphalt researchers and practitioners that the stiffness and rutting resistance of asphalt mixes containing RAP and RAS increase with increasing recycled materials content, but meanwhile, the mixes tend to be more susceptible to cracking and less durable due to heavily aged binders in RAP and RAS that are stiffer and more brittle than virgin binders. Over the years, the performance of asphalt mixtures mixes containing RAP and RAS has been found dependent on the properties of their constitutive components as well as the degree of blending between recycled and virgin binders.

3.2 Classification of Asphalt Binder Rejuvenators

Over the last decade, asphalt researchers and practitioners have explored the incorporation of petroleum and bio-based rejuvenators to help mitigate the stiffening effect of RAP and RAS materials through uniform dispersion within the mix and diffusion into heavily aged recycled binders. Rejuvenators, also known as recycling agents (RAs), have been defined as organic materials with chemical and physical characteristics selected to restore the properties of aged asphalt in order to target specification limits (Asphalt Institute, 1996). For optimal restoration of the aged asphalt binder properties, consideration should be given not only to the viscosity-reducing capacity of the rejuvenator, but also to its chemical composition. Furthermore, the degree of diffusion of the rejuvenator into the aged binder is of the utmost importance, since it will allow changes in the intermolecular agglomeration and self-assembly of the asphalt polar micelles, affecting the overall performance properties of the recycled asphalt mixes.

Although different classifications may be employed for rejuvenators based on the material source or manufacturing process, it is also important to differentiate among different products based on the asphalt chemical fraction with the most affinity with the rejuvenator.

Asphalt binder is a complex mixture of high molecular weight hydrocarbon molecules, naturally occurring heteroatoms (nitrogen, oxygen, and sulfur), and trace metals (e.g., vanadium and nickel) that contribute to the polarity within the asphalt molecules. Therefore, asphalt binders are a continuum of molecules with a gradual transition in polarity, molecular weight, and functionality. The composition of asphalt binder is usually defined in terms of the relative quantity of its so-called SARA fractions: saturates (S), aromatics (A), resins (R), and asphaltenes (A), which have increasing molecular polarity as listed (saturates have the lowest and asphaltenes the highest) (Corbett, 1969). Asphalt binder is often described as a colloid that consists of dispersion of asphaltenes in an oily matrix constituted by saturates, aromatics, and resins. It has been established that asphaltenes are stabilized in crude oils by natural resins that are surfactant-like agents.

Rejuvenators that are most compatible with the aromatics (i.e., low-polarity naphthenic aromatic fraction) of the asphalt binder will reduce the viscosity and modulus of the binder through lowering the viscosity of the continuous solvent phase. These products have little effect on the intermolecular agglomeration and self-assembly of the asphalt polar micelles. Rejuvenators that show affinity for multiple fractions of the asphalt binder and are produced through careful engineering of the source material, whether petroleum or bio-based, will reduce the viscosity of the binder through restoration of the original binder asphaltenes-to-maltenes ratio (i.e., the asphalt chemical fractions). Rejuvenators that exhibit low compatibility with the aromatics, asphaltenes, and resins fractions of the asphalt binder, especially at low temperatures, have in their composition paraffinic and saturated material with high crystalline fractions. The dispersion of such lower viscosity additives in the asphalt will reduce the modulus of the binder. However, the effectiveness of these products was found to diminish with aging since these additives can lead to colloidal instability resulting in the precipitation of the asphaltenes fraction (Johnson and Hesp, 2014).

Table 1 presents a partial list of commercially available rejuvenator products in the United States.

Table 1. Partial List of Commercially Available Rejuvenator Products in the United States.

Category	RAs Commercially Available	Description
Aromatic Oils	Hydrolene® ValAro 130A®	Prepared from aromatic crude oil. Aromatic hydrocarbons are cyclic and derivatives of benzene (rings are characterized by alternating double bonds).
Naphthenic Oils	Cyclogen L® HyPrene BO150® Reclamite®	Prepared from naphthenic crude oil. Naphthene hydrocarbons are ringed molecules and are also called cycloparaffins.
Paraffinic Oils	Valero VP 165® Waste Engine Oil (WEO) Waste Engine Oil Bottoms (WEOB)	Prepared by solvent separation techniques from paraffinic crude oil. Paraffin hydrocarbons are characterized by open or straight chains joined by single bonds.
Naphthenic or Paraffinic Oils	Re-refined Engine Oil Bottoms (REOB)/Vacuum Tower Asphalt Extender (VTAE)	Residual distillation product from a vacuum tower in a re-refinery of used lubricating oil. Prepared from either naphthenic or paraffinic crude oil.
Tall Oils	Sylvaroad™	By-product of the Kraft process of wood pulp manufacture when pulping mainly coniferous trees.
Fatty Acids	Anova® Delta S® Modified Vegetable Oils Recycled Vegetable Oils	Derived from bio-based sources.

3.3 Determination of Optimum Dosage of Asphalt Rejuvenators

In addition to quality, the dosage of rejuvenators also plays a significant role on the performance of the recycled asphalt binders and mixes. An excessive rejuvenator dosage may produce mixes with increased susceptibility to rutting whereas inadequate dosage may soften the mix but not necessarily improve its cracking resistance (Arámbula-Mercado et al., 2018). The optimum dosage of rejuvenators in a recycled asphalt mix has been found dependent on the source and grade of the virgin binder as well as by the aging level and proportion of the recycled materials. The most common methods for determining the dosage of rejuvenators rely on either the manufacturer recommendation, field experience, or using blending charts. Almost all rejuvenator products on the market have a specified range of dosage specified by the manufacturer, whereas determining dosage by field experience comes with the knowledge an engineer/technician has acquired while working with a particular product. On the other hand, determining the rejuvenator dosage using blending charts involves determining physical properties, such as viscosity, penetration, and the performance grade (PG), of the recycled binder blends.

Zaumanis et al. (2015) used a penetration test to determine the dosage of rejuvenators required to restore a reclaimed binder with a penetration of 19 mm x 0.1 mm to the target level of virgin binder having a penetration of 78 mm x 0.1 mm. As shown in Figure 1, an exponential relationship was observed between the penetration index and the rejuvenator dosage. Organic based products such as waste vegetable (WV) grease, WV oil, organic oil, and distilled tall oil reached the target penetration at 8-11% dosage, while petroleum-based products including WEO, aromatic extract, and virgin binder achieved the target penetration at 14-21% dosage. As a conclusion of this study, the authors suggested 12% as the optimum dosage for all rejuvenators to restore the penetration of the 100% reclaimed asphalt binder.

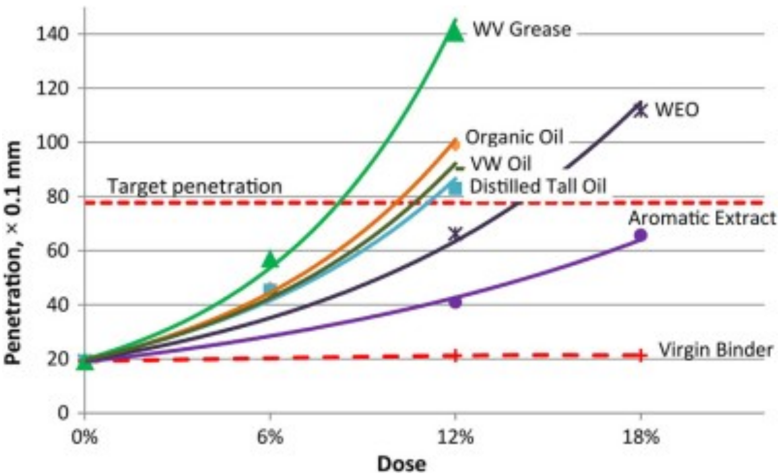


Figure 1. Penetration of RAP Binder versus Rejuvenator Dosage (Zaumanis et al., 2015).

With the development in mix design procedures and innovative technologies in the asphalt industry, determining the optimum dosage of asphalt rejuvenators in mixtures with high recycled materials content based on a PG blending chart seems reasonable and trustworthy. Several studies conducted by researchers such as Tran et al. (2012), Mogawer et al. (2013), Zaumanis et al. (2015), and Arámbula-Mercado et al. (2018) have successfully applied the concept to accomplish their research objectives.

Arámbula-Mercado et al. (2018) suggested a practical three-step protocol for selection of rejuvenator dosage. The first step involves preparation of recycled blends which requires first extracting and recovering the reclaimed asphalt binder from the RAP/RAS materials, and then formulating the recycled binder blend using the reclaimed binders, the substitute (base or virgin) binder, and a rejuvenator. In the protocol, three recycled blends are recommended: the control blend without a rejuvenator, a binder blend with a rejuvenator dosage between 2-5% by total binder, and another blend with a rejuvenator dosage between 8- 10% by total binder. The second step consists of measuring the high-temperature and low-temperature PG of the recycled blends in accordance with AASHTO M320. Lastly, a linear blending chart containing the true performance grades of the blended binder on the y-axis versus the rejuvenator dosage on the x-axis is developed.

After developing the PG blending chart, Arámbula-Mercado et al. (2018) presented three rejuvenator dosage selection methods. The objective of these methods was to restore the rheology of the recycled binder blend to that of the target binder PG needed to satisfy the traffic and climate requirements for guaranteed long-term pavement performance (Kaseer et al., 2018). The first method involved determining the rejuvenator dosage that met the low-temperature PG of the target binder then verifying whether the high-temperature PG was met as well. The second method consisted of achieving an ΔTc of 5°C or less as recommended by Anderson et al. (2011). However, Arámbula-Mercado et al. (2018) aged the recycled binder for 20 hours in a pressure aging vessel (PAV) prior to bending beam rheometer (BBR) testing instead of the 40 hours recommended by Anderson et al. (2011). The 20-hrs approach was followed by the authors to avoid the necessity of increasing the rejuvenator dosage that could cause poor rutting resistance. The last method consisted of restoring the high-temperature PG by adding enough rejuvenator to match that of the target binder without sacrificing the cracking and rutting resistance of the blended binder. When these methods were compared, method 2 called for the highest rejuvenator dosage, followed by method 3 and then method 1. While these methods evaluated the dosage of rejuvenator required to restore the rheological proprieties of the recycled binder, they were not assessed at the mixture level to evaluate the long-term durability and cracking potential of the final rejuvenated asphalt mixes.

Zaumanis et al. (2014) proposed a similar approach that required the construction of a PG blending chart using recycled binder blends at two rejuvenator dosages: zero (no rejuvenator) and a predicted optimum dosage. The authors found that there was a strong linear relationship between the rejuvenator dosage and the PG of the recycled binder blend, and that a deviation in the measured versus predicted rejuvenator dosage did not exceed 1.1%, which was not considered practically significant.

3.4 Rheological Characterization of Recycled Asphalt Binders with Rejuvenators

Recycled asphalt materials are useful alternatives to virgin materials because they reduce the use of virgin aggregates and the amount of virgin binder required in the production of asphalt mixes. However, the hard, oxidized nature of reclaimed binder is a major concern when incorporating recycled materials into asphalt mixes since the stiff binder in the RAM can cause premature fatigue and low temperature cracking failures in asphalt pavements (McDaniel and Anderson, 2001). Therefore, the performance of asphalt mixes containing RAP and/or RAS is dependent on the properties of its constitutive components, and the level of blending between the aged and unaged binder is influenced by the chemical composition of the individual binders.

Over the years, researchers have investigated the effect of various rejuvenators on the rheological performance of asphalt binder blends with high RAP and/or RAS contents. By using the Multiple-Stress Creep Recovery (MSCR) test, Mogawer et al. (2013) reported higher J_{nr} values for an asphalt binder containing a rejuvenator in comparison to a virgin binder, indicating that the selected rejuvenator acted by softening the virgin binder. Furthermore, the authors concluded that different rejuvenators can have different binder softening effects. Further, several studies applied the DSR frequency sweep test showing that inclusion of recycled materials usually resulted in an increase of $|G^*|$ and a reduction of δ (Yu et al., 2014; Haghshenas et al., 2016; Grilli et al., 2017; Yin et al., 2017). Conversely, in the same studies, the inclusion of asphalt rejuvenators was reflected as a reduction of $|G^*|$ and an increase of δ. In general, the proportion of any induced strain in asphalt binder that is attributable to non-recoverable, viscous flow increases with both loading time and temperature. Non-load related cracking of asphalt pavements (i.e. transverse and block cracks) is related to oxidation and hardening of the asphalt binder, which is the main concern when incorporating RAP and/or RAS material in the production of HMA.

To estimate the performance implications of RAP and/or RAS, mix designers have been using blending charts to interpolate the effects of recycled materials on blended binder properties. These charts have been proven effective for RAP materials and using standard Superpave PG test methods during the NCHRP 09-12 study. However, research performed by Bonaquist (2011a) showed that the blending charts may not accurately predict blended binder properties for RAS binders, particularly at low temperature. Therefore, researchers have made significant efforts to classify cracking resistance of asphalt binders using an index parameter. Table 2 presents rheological tests that have been used to identify the cracking potential of asphalt binders. The description of each test is included as follows.

Table 2. Rheological Tests Used to Identify the Cracking Potential of Asphalt Binders.

Test Type	Standard	Research Parameter
Dynamic Shear Rheometer (DSR) Mastercurve	AASHTO T315	Glover-Rowe (G-R)
Linear Amplitude Sweep (LAS)	AASHTO TP101	Cycles to Failure (N_f)
Bending Beam Rheometer (BBR)	AASHTO T313	Stiffness, m-value, and ΔT_c
	AASHTO TP122	Physical hardening behaviour

Kandhal et al. (1977) evaluated many pavements in Pennsylvania and noted that the decrease in low-temperature ductility was an important factor as asphalt binder aged (i.e., age-induced surface damage is related to a tensile failure strain in the brittle region). Following this work and by using a mechanical-empirical relationship with observed cracking, Glover et al. (2005) suggested a parameter [$G'/(\eta'/G')$], relating storage modulus (G') and dynamic viscosity (η') to ductility at 15°C and 0.005 rad/s, which served as a surrogate for tensile strain at failure.

The AASHTO TP 101 test method, *Estimating Damage Tolerance of Asphalt Binders Using the Linear Amplitude Sweep*, was proposed and used in various studies to estimate the fatigue cracking resistance of asphalt binders (Hintz et al., 2011). The LAS test considers pavement structure (i.e., strain) and traffic (i.e., number of cycles to failure). In this test, the use of viscoelastic continuum damage mechanics allows for prediction of fatigue life at any strain amplitude from a single 30-minute test. The test consists of two steps: a frequency sweep and an amplitude sweep. In the first part of the LAS procedure, an initial 100 cycles are applied at small strain (0.1%) to determine undamaged linear viscoelastic properties. The second part of the procedure consists of ramping strain amplitude, starting at 0.1% and ending at 30% applied strain, over 3100 cycles of loading at 10 Hz. Once the strain sweep is applied to the sample, damage accumulation can be then determined through Viscoelastic Continuum Damage (VECD) analysis, resulting in the fatigue power law damage model (Equation 1), and the corresponding coefficients, *A* and *B*.

$$N_f = A\,(\gamma_{max})^{-B} \qquad \text{(Equation 1)}$$

N_f is the traffic volume failure criteria and is defined as the number of cycles to fatigue failure at a user-defined damage level. γ_{max} is the maximum tensile strain expected in the binder phase under traffic loading, which will be a function of pavement structure. Coefficient *A* is the LAS power-law parameter representing the intercept at 1% strain. Coefficient *B* is the LAS power-law parameter representing the slope of the N_f-strain curve. The logarithmic slope of the storage modulus [G'(ω)] as a function of angular frequency is used to calculate the damage accumulation and the coefficient *B*. As shown in Figure 2, the LAS fatigue test procedure was validated through comparison with performance of Long-Term Pavement Performance (LTPP) test sections showing good correlation with field measurements (Hintz et al., 2011).

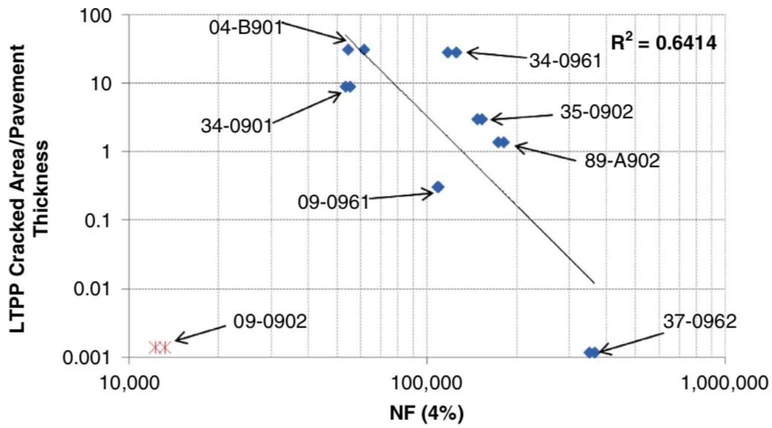


Figure 2. LTPP Measurements vs. LAS Number of Cycles to Failure (N_f) (Hintz et al., 2011).

Anderson et al. (2011) investigated the relationship between ductility and binder properties to non-load associated cracking potential for airport pavements. The findings of the study identified the Glover parameter [G′/(η′/G′)], the fatigue parameter *B*, and ΔT_c as parameters to indicate changes in binder cracking susceptibility with aging. ΔT_c is the difference between the continuous low temperature binder grade measured via the BBR creep stiffness (related to thermal stresses in an asphalt pavement due to shrinking) and m-value (related to the ability of an asphalt pavement to relieve these stresses). The authors suggested that asphalt binders with low (more negative) ΔT_c have less ductility and relaxation properties than asphalt binders with higher (less negative or positive) ΔT_c. A maximum ΔT_c threshold of -5°C after 40 hours of PAV aging was suggested to minimize the risk of aged-related cracking. Supporting this finding, Reinke (2017) showed that binders with a highly negative ΔT_c have been implicated in projects with high rates of cracking. Regarding the importance of the ΔT_c parameter for RAP binders modified with polymers and recycling agents, a Pooled Fund study sponsored by Colorado, Idaho, Kansas, and Wisconsin DOTs showed that ΔT_c is an applicable parameter to differentiate between stiffness and relaxation properties of different modification technologies and base asphalts (Figure 3) (Bahia et al., 2018).

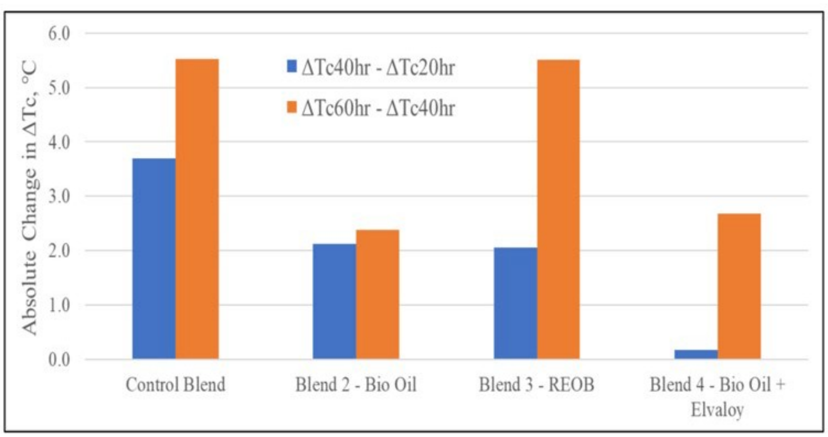


Figure 3. Absolute Change in ΔT_c for RAP Binders Modified with Polymers and Rejuvenators (Bahia et al., 2018).

Rowe (2011) proposed modifications of the Glover parameter by introducing the Glover-Rowe (G-R) parameter (Equation 2), which focused attention on the complex modulus (|G*|) and phase angle (δ) of asphalt binders at 15°C and a frequency of 0.005 rad/s. On his proposal, Rowe ignored the frequency term and expressed the G-R parameter purely in terms of |G*| and δ, allowing users to plot the ductility-based failure planes in a Black Space diagram. It is known that asphalt binders with higher values of G-R experienced a higher level of oxidative aging than those with lower values of G-R parameter (Yin et al., 2017; Zhou et al., 2015). Figure 4 presents an example of a Black Space diagram of recycled and rejuvenated asphalt binder blends.

$$G-R\ Parameter = \left\{ \frac{|G^*| \left[\cos(\delta)^2\right]}{\sin(\delta)} \right\}_{T=15^\circ C,\, f=0.005 rad/s} \quad \text{(Equation 2)}$$

Asphalt binders that are excessively aged, due to susceptibility to oxidation and/or the presence of higher percentages of recycled asphalt materials in a mix, are more prone to low temperature cracking. Time-dependent hardening was observed near the glass transition (T_g) temperature of asphalt binders during the Strategic Highway Research Program (SHRP) contract A002-A and was referred to as physical hardening (Anderson et al., 1994; Bahia, 1991). Physical hardening causes time-dependent isothermal changes in the rheological behavior and specific volume of asphalt binders (Anderson and Marasteanu, 1999). The process is reversible: when the asphalt binder is heated to room temperature or above, the effect of physical hardening is completely removed. Physical hardening for asphaltic materials is generally observed both above and below T_g. The study performed during the SHRP program resulted in a requirement in the AASTHO M320 specification of testing in the BBR after 1 and 24 hours of conditioning at low temperature. Although this requirement was not implemented, recent work by Hesp and Subramani (2009) observed better correlations between BBR results and the low temperature field when physical hardening was considered. Tabatabaee et al. (2012) showed that the rate of physical hardening peaked at the glass transition temperature (T_g) and became relatively insignificant beyond the limits of the glass transition region. Anderson and Marasteanu (1999) showed that asphalt binders with higher wax content had stronger physical hardening effects both above and below their T_g.

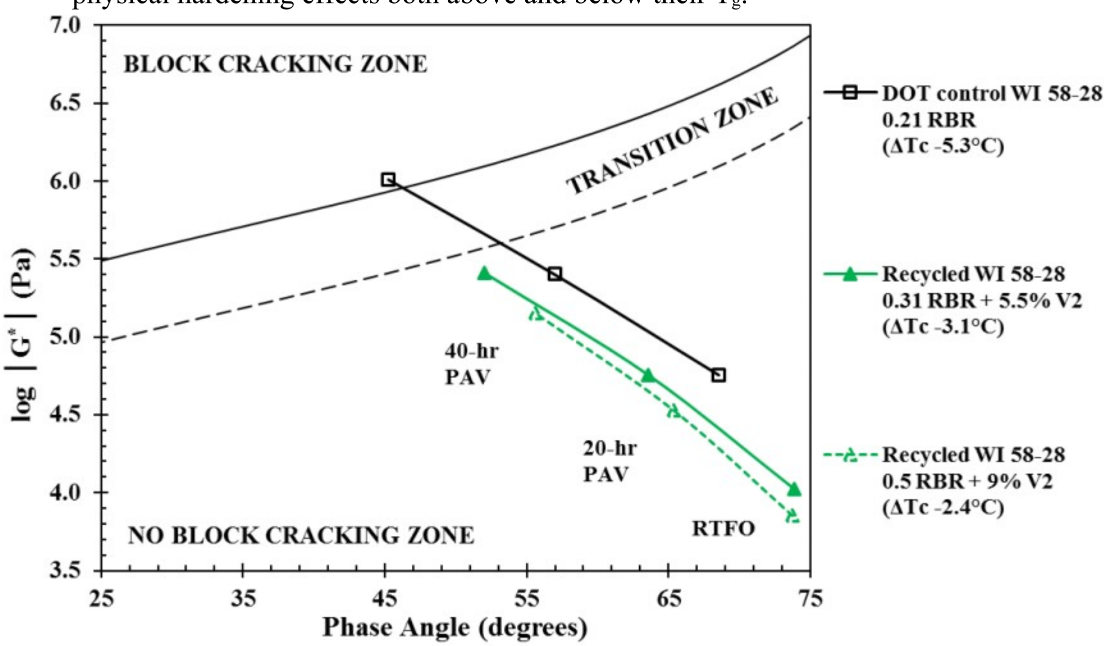


Figure 4. |G*| and δ in Black Space for Recycled and Rejuvenated Binder Blends with a Target PG 58-28 Climate (Kaseer et al., 2018).

3.5 Chemical Characterization of Recycled Asphalt Binders with Rejuvenators

The SHRP goal of developing a better understanding of how asphalt chemistry influences performance resulted in a microstructure model that related asphalt chemistry to physical properties. The model predicts a variety of intermolecular associations as largely responsible for the physical properties of asphalt. The principal cause of asphalt aging and embrittlement in service is the atmospheric oxidation of molecules with the formation of highly polar and strongly interacting functional groups containing oxygen (Petersen, 2009). Therefore, binder oxidation has a significant impact on age-related pavement failure, since through oxidation the binder becomes stiffer and more brittle, reducing the pavement service life (Petersen et al., 1993). As asphalts age, they harden; this results in a progressive increase in the stiffness modulus of the asphalt, together with a reduction in its stress relaxation capability (Read and Whiteoak, 2003). Since the aging behavior of asphalt mixes containing recycled asphalt materials is influenced by the chemical composition of the individual binders, chemical analyses are important to investigate the impact of RAP and/or RAS on the molecular distribution, thermal response, and chemical composition of the resulting blends.

3.5.1 Gel Permeation Chromatography

The Gel Permeation Chromatography (GPC) technique is used to determine the molecular size distribution (MSD) of asphalt binders (aged, unaged, modified and unmodified), providing a distinct and reproducible molecular-size distribution curve (chromatogram) of the asphalt sample in solution (Jennings et al., 1980; Churchill et al., 1995). In this method, the asphalt binder is dissolved in a solvent and then injected into the GPC system. The injected sample travels through a series of columns that separate the sample based on molecular size (Figure 5). The larger molecular size particles exit the columns first and are detected by the system's detectors. The smaller molecular size particles travel into the pores of the columns, and therefore, have longer retention times. As a result, the chromatogram of molecular size distribution (which can be thought of as analogous to a type of sieve analysis of the sample) is obtained.

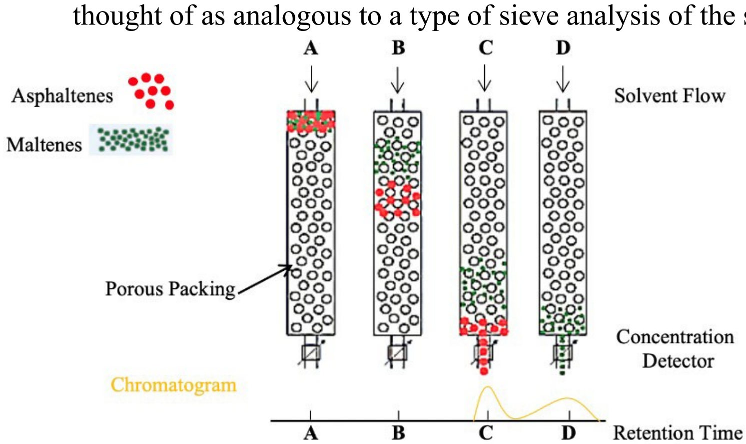
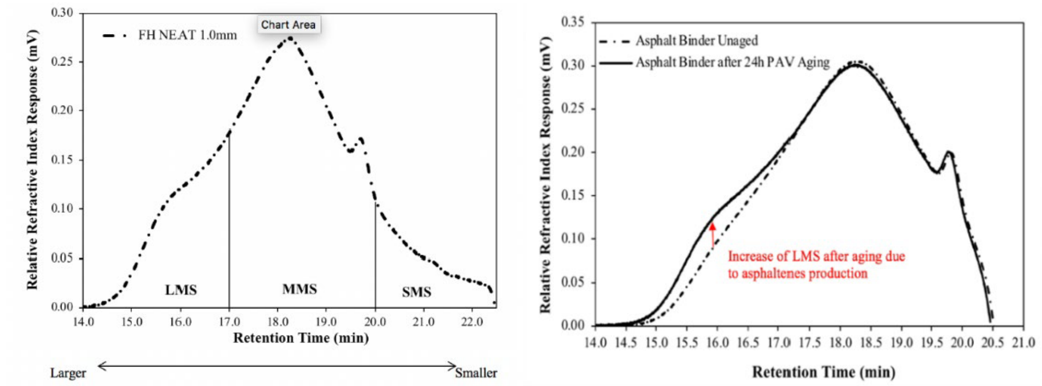


Figure 5. Schematic of Gel Permeation Chromatography (Moraes and Bahia, 2015a).

The chromatogram allows the classification of the chemical composition of asphalt binders into three groups based on their molecular size. These three groups are: large molecular size molecules (LMS), medium molecular size molecules (MMS), and small molecular size (SMS) molecules. Thus, the GPC chromatograms can provide insights about what fractions of the asphalt binder are affected after oxidative aging. It has been reported that a strong correlation exists between LMS and asphaltenes content (Moraes and Bahia, 2015a) (Figure 6).



(a) (b)

Figure 6. (a) GPC Chromatogram of an Asphalt Binder; (b) GPC Chromatogram of an Asphalt Binder before and After 24 Hours of PAV Aging (Moraes and Bahia, 2015a).

One of the great advantages of GPC is its ability to separate by molecular size rather than by solubility or adsorptivity. The GPC is a simple separation technique available that responds to molecular weight alone and not to chemical structure. This feature makes GPC especially suited for fractionating complex mixtures, like asphalt binders. Table 3 presents an overview of the literature involving the application of GPC to asphalt binders.

Table 3. Examples of Application of GPC to Asphalt Binders (Moraes, 2014).

Gel Permeation Chromatography Application	Reference
Determine asphalt molecular weight distributions.	Snyder, 1969; Ying et al., 2013.
Use of GPC to characterize asphalt properties and the relationship of GPC parameters to pavement performance.	Jennings et al., 1980; Jennings et al., 1993; Yapp et al., 1991.
Evaluate the effects of oxidative aging on asphalt binders and mixes using the gel permeation chromatography procedure.	Kim and Burati, 1993; Churchill et al., 1995; Siddiqui and Ali, 1999; Lu and Isacsson, 2002; Doh et al., 2008; Lee et al., 2009.
Estimate absolute viscosity of aged binder in Reclaimed Asphalt Pavement (RAP) by using gel permeation chromatograph technique.	Kim et al., 2006.
Characterize blends of laboratory-aged crumb rubber modified binders (CRM) and rejuvenating agents by using GPC.	Shen et al., 2007.
Demonstrate that GPC can be used as a simple screening test to identify when asphalt binder has been modified with a polymer.	McCann et al., 2011.
Evaluate Reclaimed Asphalt Pavement (RAP) blending efficiency by using gel permeation chromatograph technique.	Bowers, 2013.
Investigate the oxidative aging levels of polymer-modified asphalt produced with Warm Mix Asphalt (WMA) technologies.	Kim et al., 2013.
Correlated an increase in large molecular sizes to the complex modulus (|G*|) of asphalt binder.	Zhao et al., 2013.
Evaluate changes caused by oxidative aging in the colloidal structure of the asphalt due to changes in the degree of association of the different asphalt fractions (i.e. asphaltenes and maltenes).	Moraes and Bahia, 2015a.
Evaluate the effects of asphalt binders' rejuvenators during aging.	Li et al., 2016.

3.5.2 Glass Transition Temperature

The glass-transition temperature (T_g) has been considered as a characterization parameter that helps to determine the process and aging level of asphalt binders (Moraes and Bahia, 2015b). The T_g depends on the asphalt source and the degree of aging, since complex arrangements of molecules are formed (Turner et al., 1997). Conducting glass-transition measurements on asphalts with different amount of asphaltenes, Wada (1960) showed that the glass-transition temperature increased with an increase of the asphaltenes content. The transition to glassy behavior is known to increase the brittleness of the binder extensively, reducing the potential for stress relaxation, increasing stiffness, and therefore resulting in higher cracking susceptibility. There are speculations in the asphalt community that the glass-transition temperature of asphalt is responsible for low-temperature cracking of the mix (Marasteanu et al., 2007).

By using a dilatometric system to measure the glass transition temperature of asphalt binder, Moraes and Bahia (2015b) showed that oxidative aging and increase in asphaltenes content shifted the T_g towards higher temperatures, thus increasing the susceptibility of the binder to cracking and durability issues due to the ductile-to-brittle transition behavior. The behavior presented in Figure 7 is explained by the authors as the result of the effect of the aging process on the asphaltenes and resins asphalt fractions. At the beginning of the oxidative aging process, the glass-transition behavior of the evaluated neat binder was dominated by the increase in the lower T_g resins fraction, which lead to an overall decrease in the glass-transition temperature (i.e., becomes more negative). After six hours of PAV aging, the asphaltenes content started to increase which resulted in an increase of the T_g.

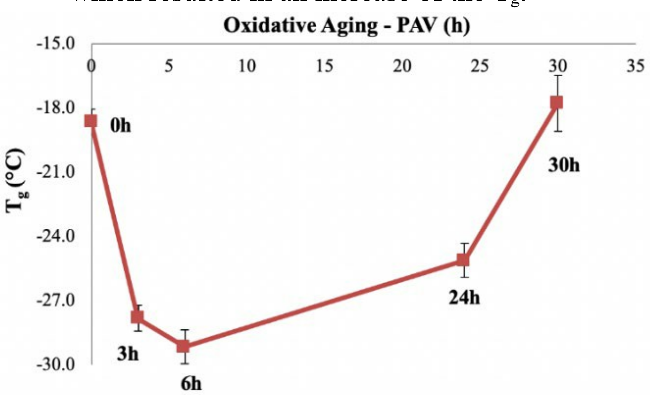


Figure 7. T_g of Neat Asphalt Binder after Different Aging Conditioning in PAV (Moraes and Bahia, 2015b).

The T_g of asphalt binders can also be determined by using Differential Scanning Calorimetry (DSC). In this technique, the difference in the amount of heat required to increase the temperature of a sample and a reference material are measured as a function of temperature.

3.5.3 Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy by Attenuated Total Reflectance (FTIR-ATR) exploits the attenuation of light reflected internally in a non-absorbing prism, due to energy absorption of an analyte in contact with the reflecting surface (Figure 8). It has been applied to asphalt binder for characterization of its chemical composition and aging, for detection of impurities, and for studying polymer modification. Two of the major advantages of FTIR-ATR applied to asphalts, compared to transmittance FTIR, are: (a) the spectrum is obtained without solvent, and (b) the chemical influence of solvents can be avoided (Kelli-Anne et al., 2014).

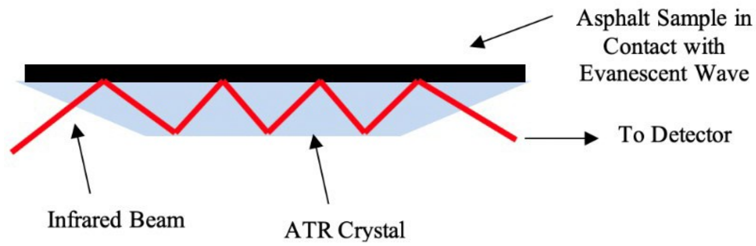


Figure 8. A Multiple Reflection FTIR-ATR System.

Existing literature shows that the change in chemical structure of asphalt binders can be obtained with the calculation of functional and structural indices of some groups from FTIR-ATR spectra, since with oxidative aging the absorbance bands representing oxygen-containing functionalities of asphalt increase (Jennings et al., 1980). Thus, to quantify oxidation-related changes collected by means of infrared absorption, band areas values can be used to calculate chemical changes in carbonyl (C=O) and sulfoxide (S=O) groups. As can be seen in Figure 9 the content of carbonyl compounds increases during aging, and the degree of the changes is dependent on the asphalt binder source.

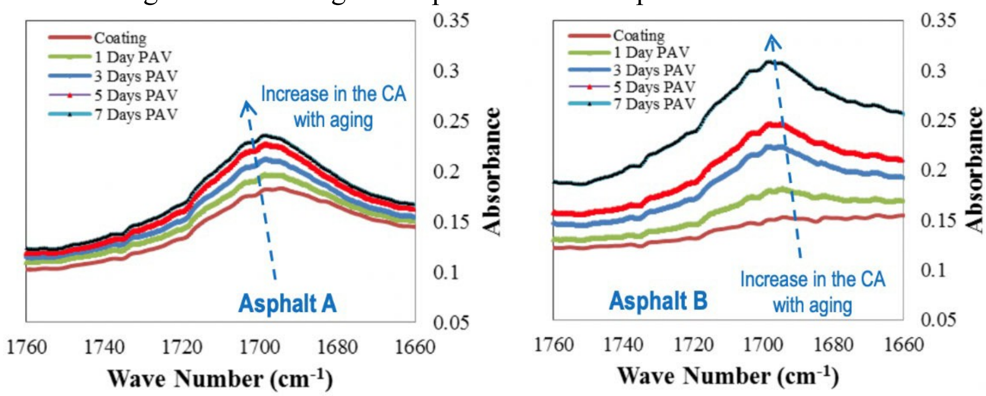


Figure 9. Increase in C=O Area with PAV Aging Time for Asphalt Binders from Different Sources.

The absorption spectrum of carbonyl functions (such as ketones, dicarboxylic anhydrides, and carboxylic acids) is calculated by integrating the area of the spectrum between the wavelengths of 1660 and 1753 cm^{-1} and using the magnitude of the absorption at 1753 cm^{-1} as the baseline. For asphaltic materials, because of overlapping between the peaks at ~1700 cm^{-1} (carbonyl functions) and at ~1600 cm^{-1} (aromatic function), it is preferred to consider the surface area between these two limits (RILEM, 2012). Sulfoxide area is calculated by integrating the area of the spectrum between the wavelengths of 995 and 1047 cm^{-1} and using the magnitude of the absorption at 1047 cm^{-1} as the baseline. Table 4 presents studies involving the application of FTIR to asphalt binders.

Table 4. Examples of Application of FTIR to Asphalt Binders.

FTIR Application	Literature
Investigate the diffusion of rejuvenators within the asphalt binder.	Karlsson and Isacsson, 2003.
Investigate the effect of antioxidants in the aging susceptibility of SBS polymer modified asphalt binder.	Ouyang et al., 2006.
Evaluate changes in C=O and S=O groups with the addition of rejuvenators to asphalt binder.	Abbas et al., 2013.
Validate rheological results which indicated that aging susceptibility of asphalt binders modified with polymers and rejuvenators is dependent on modification chemistry.	Li et al., 2016.

3.5.4 Gas Chromatograph/Mass Spectrometry

Gas chromatography-mass spectrometry (GC-MS) is a method that combines the features of gas-liquid chromatography and mass spectrometry to identify different substances within a test sample. The GC/MS technique is comprised of a gas chromatograph (GC) coupled to a mass spectrometer (MS), by which complex mixtures of chemicals may be separated, identified, and quantified. The GC utilizes a capillary column that depends on the column's dimensions (length, diameter, film thickness) as well as the phase properties of the sample being analyzed. The difference in the chemical properties between different molecules in a mixture will separate the molecules as the sample travels the length of the column. Since the molecules take different amounts of time (i.e., retention time) to travel through the GC, the MS downstream can capture, ionize, accelerate, deflect, and detect the ionized molecules separately. This MS performs this process by breaking each molecule into ionized fragments and detecting these fragments using their mass to charge ratio.

The GC/MS technique can be used to investigate the chemical composition (i.e., fatty acid content) of some anti-aging additives (i.e., bio-oils) in order to correlate the ratio of the components to the effectiveness of each additive. Since the effectiveness of bio-rejuvenators in changing asphalt binder properties could be related to its composition, the fatty acid and non-fat acid content of oils can be a useful parameter when choosing among different bio-rejuvenators.

A fatty acid is a carboxylic acid with a long aliphatic chain, which is either saturated (no carbon-carbon double bond) or unsaturated (with carbon-carbon double bond). If saturated, the chain of carbon atoms holds as many hydrogen atoms as possible. If unsaturated, the fatty acid can be further classified as monounsaturated (with one carbon-carbon double bond) or polyunsaturated (with >1 carbon-carbon double bond). It is important to mention that the stability of these fatty acids is related to the degree of unsaturation. An example of the GC/MS results for characterization of different bio-based asphalt rejuvenators is presented in Figure 10.

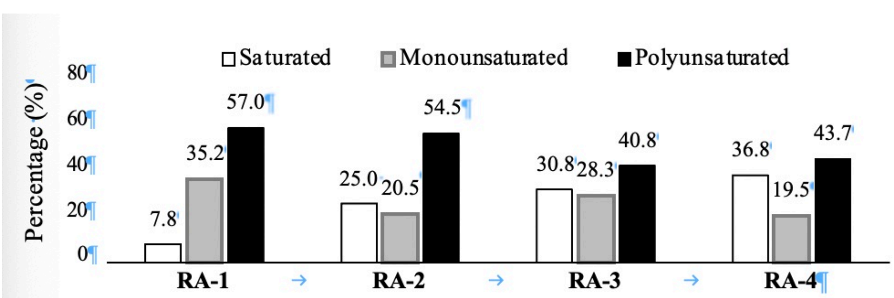


Figure 10. Fatty Acid Content of Bio-based Recycling Agents.

By using GC/MS, Zhou et al. (2018) evaluated the chemical composition of eight bio-based rejuvenators (Figure 11). The rheological performance of RAP binders modified with the rejuvenators was also investigated in the study. The authors suggested that the total fatty acid content measured by GC/MS is a good performance indicator for bio-based rejuvenators due to two factors: (1) the low temperature PG grade of recycled asphalt binders is controlled primarily by its relaxation property (or m-value); and (2) the total fatty acid content has higher correlation with the m-based low temperature PG in comparison with dynamic viscosity.

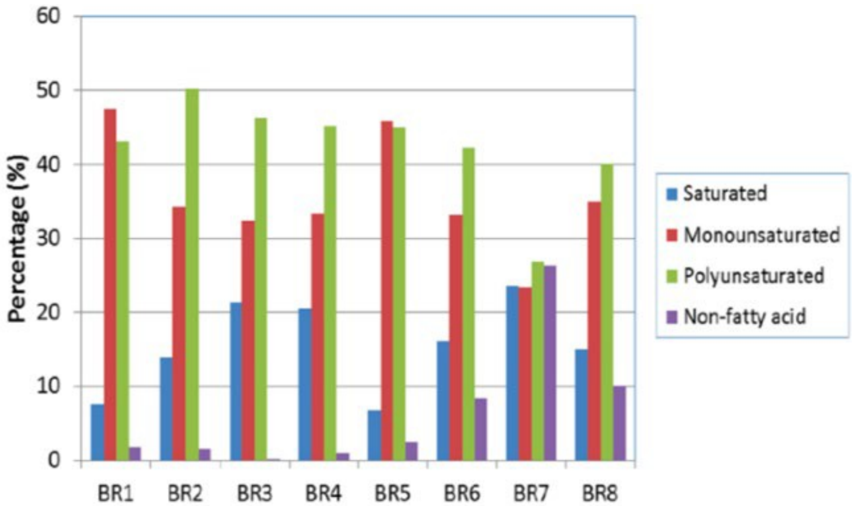
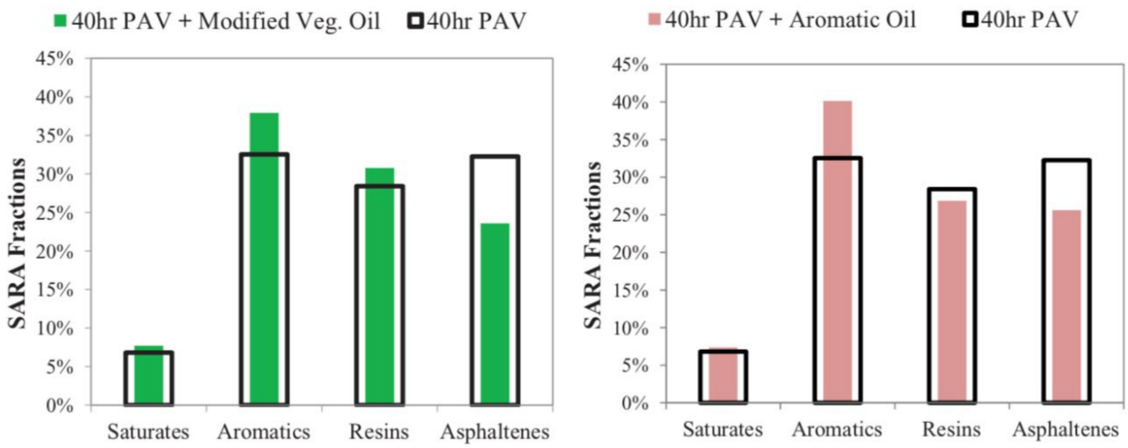


Figure 11. Fatty Acid and Non-fatty Acid Contents (Zhou et al., 2018).

3.5.5 Saturates, Aromatics, Resins and Asphaltenes Fractionation

Researchers have investigated the effects of rejuvenators on the asphalt binder SARA fractions. Yu et al. (2014) observed that the addition of an aromatic extract to an aged binder introduced more saturates and aromatics fractions, while lowering the fractions of resins and asphaltenes. The authors observed that the large amount of saturates contained in the aromatic extract led to a significant increase in the saturate fraction in the rejuvenated asphalt samples. Tabatabaee and Kurt (2017) observed that the addition of rejuvenators increased the maltene phase of an oxidized asphalt binder, and as a result reduced the asphaltenes to maltene ratio. As it can be seen in Figure 12(a), the addition of an aromatic oil-based rejuvenator had the highest contribution to the aromatic fraction of the aged binder, while the other maltene fractions remained relatively unchanged. On the other hand, the addition of a modified vegetable oil-based rejuvenator [Figure 12(b)] increased both the aromatics and the resins content of the maltene phase.



(a) (b)

Figure 12. Effect of Rejuvenation on SARA Fractions of 40-h PAV-aged Asphalt Binder using: (a) an Aromatic Oil-based Rejuvenator and (b) a Modified Vegetable Oil-based Rejuvenator (Tabatabaee and Kurt, 2017).

3.5.6 Atomic Force Microscopy

Loeber et al. (1996) were among the first researchers to investigate asphalt binders with atomic force microscopy (AFM). The authors applied force-mode (contact) AFM imaging and revealed structures present in greater number in gel-type asphalts (i.e., asphalt binders with higher asphaltenes content). The authors created the term “bumble bees” to describe the structures, which resembled the yellow and black stripes of a bumble bee. Pauli et al. (2001) used AFM to study the SHRP asphalts and found that both lateral-scanning friction-force AFM and tapping-mode AFM revealed the “bumble bee” structures identical to the type reported by Loeber et al. (1996). Over the years, the colloidal instability and the relative balance of asphalt fractions have been related to the increase in the “bee” structures formed as part of the sample surface topography measured using AFM. Tabatabaee and Kurt (2017) investigated the effect of a modified vegetable oil-based rejuvenator on the bee structures and the surface topography of two highly aged asphalt binders. One binder was solvent extracted from a South Carolina RAP source, while the other binder labelled as “3xPAV” was the SHRP core binder AAA-1, highly laboratory aged through 60 hours of PAV aging. Each binder was modified with 15% by wt. of a modified vegetable oil-based rejuvenator. The AFM results presented in Figure 13 show that the addition of the modified vegetable oil-based RA resulted in a significant reduction in the bee structuring measured on the surface of the highly aged asphalt binder. The authors reported this finding as an indicator of the effect of the specific type of rejuvenator in the restoration of the colloidal stability and phase compatibility affected through aging.

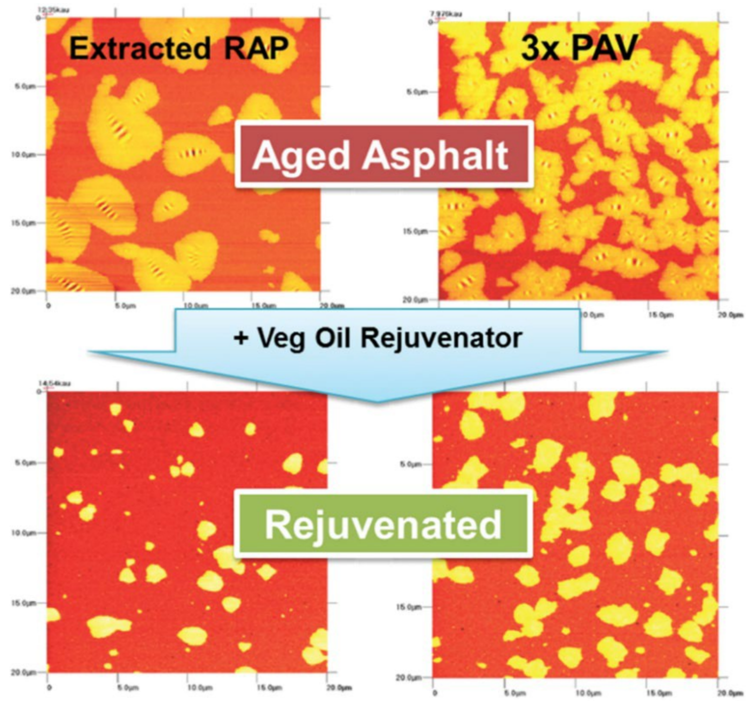


Figure 13. AFM Phase Image Scans of Solvent Spin-cast Films of Asphalt Binder; Results Showing the Effect of the Modified Vegetable Oil-based Rejuvenator in Decreasing the "bee" Structuring in Highly Aged Asphalt Binder (Tabatabaee and Kurt, 2017).

Menapace et al. (2018) observed that changes in rejuvenated binders' flow properties measured in DSR were also detected in AFM in terms of reduction in surface roughness, smoother borders of dispersed domains, increased matrix area, and better dispersion of domains. However, the dispersed domains observed with AFM were in the order of microns, and they did not correspond to the nanoaggregates of asphaltenes found in crude oils. The authors concluded that asphalt rejuvenators may initially disperse the chemical species that form bee-like structures, which appear to re-associate again during PAV aging. However, this does not always occur.

3.6 Performance Testing of Recycled Asphalt Mixes with Rejuvenators

3.6.1 State of Practice on Mixture Performance Tests

In 2018, NCAT conducted the NCHRP project 20-07/Task 406 to develop a framework for balanced mix design and investigate the implementation status of mixture performance tests (West et al. 2018). A survey conducted as part of this project identified 26 state highway agencies that required at least one mixture performance test in their mix design specifications. Among these agencies, most focused on the evaluation of rutting resistance while only a few assessed cracking resistance. However, for mixes with recycled asphalt materials, cracking is a more critical mode of distress than rutting due to reduced flexibility and relaxation properties. Based on different mechanism in crack initiation and propagation, cracking can be further categorized into bottom-up fatigue, top-down fatigue, reflection, and thermal cracking. The primary distresses of asphalt pavements in South Dakota have been found to be rutting, thermal cracking, reflection cracking, fatigue cracking, and moisture damage. In the survey, state highway agencies were asked to select the most "potential" performance tests based on their experience and knowledge. Table 5 summarizes the top two selections for each mode of distress.

Table 5. Selection of Mixture Performance Tests by State Highway Agencies (West et al., 2018).

Pavement Distress	Top Two Selections
Rutting	Hamburg Wheel Tracking Test, Asphalt Pavement Analyzer
Thermal Cracking	Disc-shaped Compact Tension Test, Semi-circular Bend Test
Reflection Cracking	Overlay Test, Illinois Flexibility Index Test
Bottom-up Fatigue Cracking	Illinois Flexibility Index Test, Bending Beam Fatigue Test
Top-Down Fatigue Cracking	Illinois Flexibility Index Test, Direct Tension Cyclic Fatigue Test
Moisture Damage	Hamburg Wheel Tracking Test, Tensile Strength Ratio

The NCHRP project also identified the following nine critical steps for implementation of a test method into routine practice. A comprehensive literature review was conducted to determine the implementation status of each mixture performance test available.

1. Develop draft test method and prototype equipment;
2. Evaluate sensitivity to materials and relationship to other lab properties;
3. Establish preliminary field performance relationship;
4. Conduct ruggedness experiment to refine its critical aspects;
5. Develop commercial equipment specification and pooled fund purchasing;
6. Conduct round-robin testing to establish precision and bias information;
7. Conduct robust validation of the test to set criteria for specifications;
8. Conduct training and certification;
9. Implement into engineering practice.

As part of this research project, the research team assessed mixture performance in terms of resistance to common distresses as follows: APA for rutting evaluation, IDEAL-CT test for intermediate-temperature cracking evaluation, and DCT for thermal cracking evaluation.

3.6.2 Effect of Rejuvenators on Asphalt Binders and Mixes with High Recycled Materials

Concerns exist with respect to the addition of rejuvenators to binders as it relates to both high and low temperature performance. For example, the softening effect of these additives can detrimentally affect the high temperature rutting resistance of the resultant asphalt binders and mixes. Furthermore, the effectiveness of rejuvenators in improving mixture cracking resistance was found to diminish with extended aging conditioning (Yin et al., 2017; Bahia et al., 2018; Epps Martin et al., 2018). This raises the question whether these agents can improve the long-term durability and cracking performance of asphalt mixes with RAP and RAS. Thus, it is crucial to take into consideration the effect of oxidative aging while investigating how rejuvenators affect the aged asphalt binders and what performance characteristics these recycled materials exhibit. Furthermore, it is necessary to develop a method to evaluate these parameters, so they can be controlled to produce asphalt mixes containing RAP/RAS with satisfactory performance.

Seidel et al. (2012) investigated the effect of soy fatty acids on the rheological properties of the asphalt binder and concluded that soy fatty acids had potential as a fluxing agent for binders that were stiff and hard to mix. Villanueva et al. (2008) investigated the effect of lubricating oil on low-temperature performance of binders. The authors found that the low-temperature grade of the modified asphalt was not significantly improved with the selected RA, due to a lack of effects on m-value results in the BBR. Used-tire-derived pyrolytic oil was used Yousefi et al. (2000), and the authors found that asphalt binder modified with 10% pyrolytic oil has better low temperature properties. Rubab et al., (Rubab, Burke, Wright, and Hesp, 2011) investigated the chemical aging behavior of asphalt binder modified with WEO. The authors reported that although engine oil residue economically increased the Superpave grade span, it also led to a significant increase in the rate of oxidation of the asphaltic material.

Over the years, researchers have investigated the effect of various rejuvenators on the performance of asphalt mixes with high RAP and/or RAS contents. Key findings from these studies are summarized in Table 6.

Table 6. Effect of Rejuvenators on the Performance of Asphalt Mixes with High RAP and/or RAS Content.

Research Finding		References
Pros	Rejuvenators can soften the aged asphalt binders and reduce the stiffness of asphalt mixes containing RAP/RAS.	Mallick et al., 2010; O'Sullivan, 2011; Hajj et al., 2013; Im and Zhou, 2014; Haghshenas et al. (2016); Bonicelli et al., 2017; Kaseer et al. (2017); Epps Martin et al., 2018.
	Rejuvenators may increase the intermediate-temperature cracking resistance of asphalt mixes containing RAP/RAS.	Mallick et al., 2010; Tran et al., 2012; Hajj et al., 2013; Im and Zhou, 2014; Zhou et al., 2015; Yin et al., 2017; Epps Martin et al., 2018; Espinoza-Luque et al., 2018.
	Rejuvenators may increase the thermal cracking resistance of asphalt mixes containing RAP/RAS.	Shen et al., 2004; Tran et al., 2012; Mogawer et al., 2013; Zaumanis et al., 2013; Yan et al., 2014; Epps Martin et al., 2018.
	Rejuvenators may decrease the moisture susceptibility of asphalt mixes containing RAP/RAS.	Hajj et al., 2013; Im and Zhou, 2014.
Cons	Rejuvenators may decrease the stiffness and rutting resistance of asphalt mixes containing RAP/RAS.	Shen et al., 2007; Tran et al., 2012; Mogawer et al., 2013; Zhou et al., 2015; Arámbula-Mercado et al., 2018; Epps Martin et al., 2018; Espinoza-Luque et al., 2018; Kaseer et al., 2018.
	Rejuvenators can increase the aging susceptibility of asphalt mixes.	Mogawer et al., 2015; Yin et al., 2017; Epps Martin et al., 2018.

3.6.3 Effect of Rejuvenators on Degree of Blending Between Virgin and Recycled Binders

The degree of blending between recycled and virgin binders has a significant effect on the volumetric and performance properties of asphalt mixes (Copeland, 2011). If the degree of blending is overestimated, the mix will not have enough asphalt binder (or is too "dry") and thus becomes more susceptible to cracking and durability related distresses. On the other hand, underestimating the degree of blending will yield a mix with excessive asphalt binder and increased susceptibility to deformation and bleeding issues (Copeland, 2011; Coffey et al., 2012). Therefore, establishing a good understanding of the degree of blending between recycled and virgin binders is important to ensure the satisfactory performance of asphalt mixes containing RAP and RAS.

Yu et al. (2017) adopted the "testing of coarse-aggregate, fine-RAP mix" method to determine the degree of blending between RAP and virgin binders. The method, developed by Huang et al. (2005), requires the preparation of a RAP mix using fine RAP materials (i.e., passing a No. 4 sieve) and coarse virgin aggregates (i.e., retained on a 9.5mm sieve). After mixing, the loose mix is separated into two fractions using a No. 4

sieve. As shown in Figure 14, the finer fraction corresponds to a blend of fine RAP materials and virgin binder, and the coarser fraction is a blend of virgin aggregate, virgin binder, and a portion of RAP binder. The asphalt binder of each fraction is then extracted, recovered, and tested to determine their properties. This method assumes that if a full blending between RAP and virgin binders occurs, the properties of the recovered binders from the two fractions would be the same; otherwise, the recovered binder from the finer RAP fraction would be stiffer and more brittle than that recovered from the coarser aggregate fraction due to the presence of a higher percentage of RAP binder.

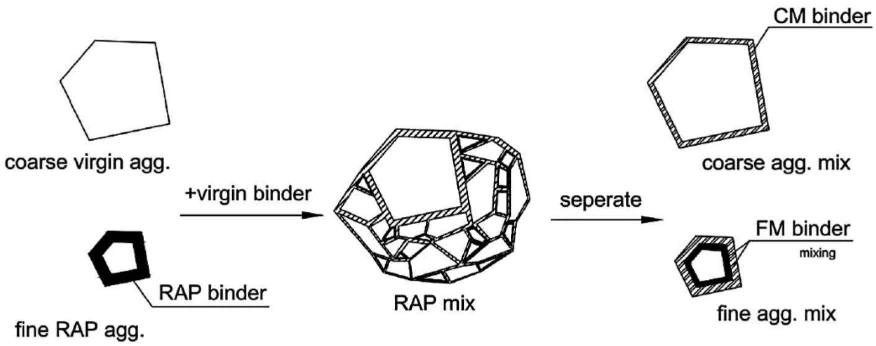


Figure 14. Illustration of the Testing of Gap-graded RAP Mix (Yu et al., 2017).

Yu et al. (2017) selected two DSR rutting parameters [i.e., DSR G*/sinδ and non-recoverable creep compliance (J_{nr}) from the Multiple Stress Creep Recover (MSCR) test] and two DSR fatigue parameters (i.e., G*sinδ and fracture energy measured from a monotonic fatigue test) to quantify the difference in the properties of the two extracted binders. Three RAP mixes containing a PG 64-22 virgin binder and 20%, 40%, and 60% RAP were tested. As shown in Table 7, the degree of blending of these mixes was found to vary from 20% to 85% and the results were not sensitive to the specific rutting or fatigue parameters used. Additionally, the addition of a recycling agent was found to improve the degree of blending of the 60% RAP mix by approximately 10%.

Table 7. Degree of Blending Results (Yu et al., 2017).

Mix Type	Degree of Blending				
	G*/sinδ	MSCR J_{nr}	G*sinδ	Fracture Energy	Average
20% RAP	36.2%	32.9%	21.2%	20.9%	27.8%
40% RAP	83.0%	84.0%	81.6%	84.8%	83.4%
60% RAP	73.5%	73.7%	63.6%	71.4%	70.6%
60% RAP + Recycling Agent	84.8%	91.4%	68.0%	74.9%	79.8%

As part of the NCHRP project 09-58, Kaseer et al. (2019) developed a method that requires the preparation and testing of two gap-graded mixes to determine the degree of blending between RAP and virgin binders. As illustrated in Figure 15, a gap-graded virgin mix is prepared by mixing virgin binder and virgin aggregate blends consisting of 3/8”, #4, #8, and #30 fractions. Additionally, a gap-graded RAP mix is prepared in the same manner as the virgin mix, except for replacing the #4 fraction of virgin aggregates with the same size of RAP materials. The total binder content of the two mixes are kept the same. After mixing and short-term conditioning for two hours at 135°C, both mixes are manually separated into three fractions using 3/8” and #4 sieves. The binder content of each fraction is then determined using the ignition oven. Finally, the difference in the binder content of the #4 fractions between the virgin and RAP mixes is used to determine the amount of “active” RAP binder contributing to the total mix and calculate the RAP binder availability factor (BAF).

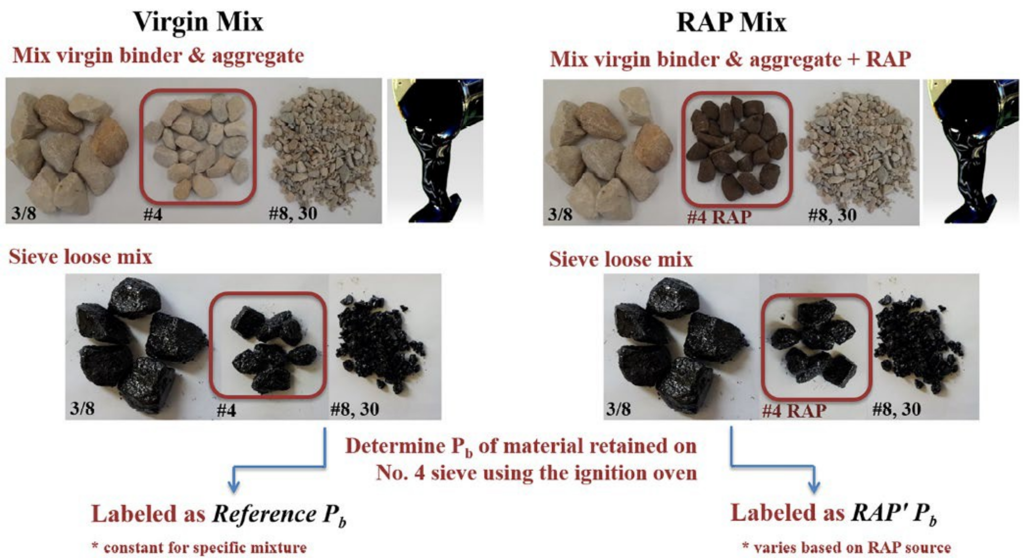
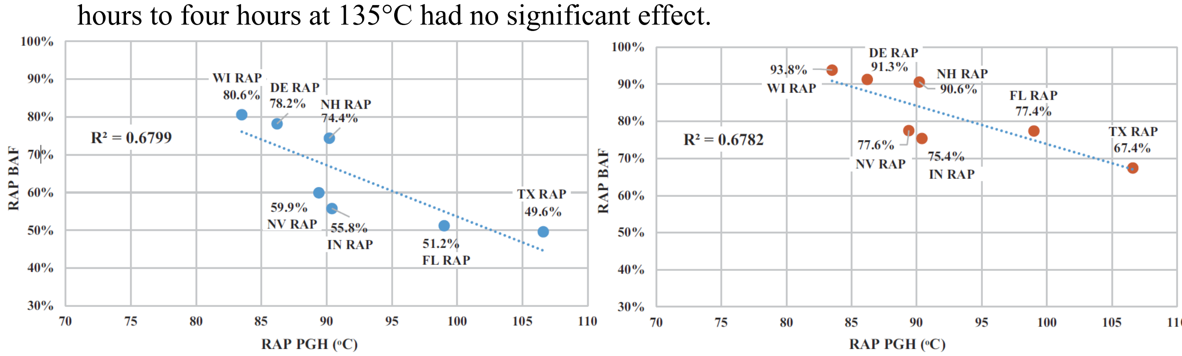


Figure 15. Illustration of Testing of Gap-graded Virgin and RAP Mixes (Kaseer et al., 2019).

Kaseer et al. (2019) used this method to evaluate the effects of mixing temperature, short-term conditioning time, RAP source and binder grade, and recycling agents on the RAP BAF. Test results showed that the RAP binder availability increased as the stiffness of the extracted RAP binder decreased and as the mixing temperature increased. As shown in Figure 16, a reasonable linear relationship was found between the RAP BAF and the high-temperature performance grade of the extracted RAP binder. These results indicated that stiffer and more heavily aged RAP materials are likely to have a reduced amount of "active" binders available contributing to the total binder content of the mix. Further, the addition of recycling agents was found to improve the RAP binder availability while extending the short-term conditioning time from two hours to four hours at 135°C had no significant effect.



(a) (b)

Figure 16. Linear Relationships between RAP BAF and High-temperature PG of Extracted RAP Binder; (a) Mixing Temperature at 140°C, and (b) Mixing Temperature of 150°C (Kaseer et al., 2019).

Castorena et al. (2016) introduced the use of energy dispersive X-ray spectroscopy (EDS) scanning electron microscopy (SEM) to qualitatively evaluate the degree of blending between RAP and virgin binders. In this method, titanium dioxide power is pre-blended into the virgin binder prior to the preparation of asphalt mixes, which allows the delineation of RAP and virgin binders in the EDS mapping. During testing, the locations of RAP binder and virgin binder are determined by comparing the carbon EDS maps and titanium EDS maps. Because titanium dioxide is present only in virgin binder, areas with carbon but no titanium correspond to RAP binder while areas with carbon and titanium indicate the presence of virgin binder, possibly blended with RAP binder, as shown in Figure 17.

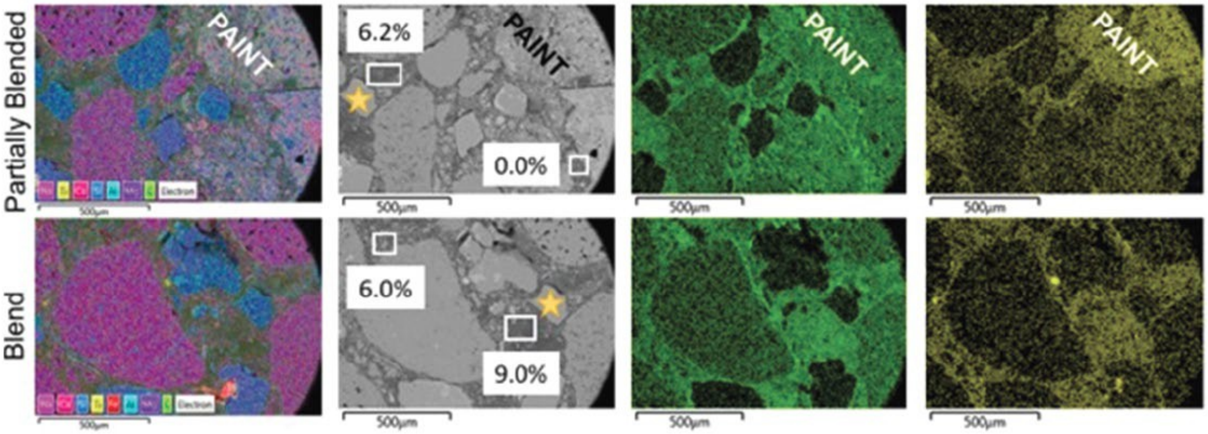


Figure 17. Examples of EDS SEM Images for Quantifying the Degree of Blending between RAP and Virgin Binders (Castorena et al., 2016).

Jiang et al. (2018) adopted the EDS SEM method described above and proposed the use of element mass ratio of titanium over sulfur (Ti:S) to quantify the degree of blending between RAP and virgin binders. Four asphalt mixes with 0%, 15%, 30%, and 50% RAP were tested. The effect of mix aging and the use of recycling agents on the degree of blending between RAP and virgin binders was evaluated. As shown in Figure 18, the degree of blending varied from 40 to 100% among the RAP mixes tested. In general, the degree of blending decreased as the RAP content in the mix increased. In addition, loose mix aging for 12 hours at 135°C prior to compaction and the use of recycling agents greatly improved the degree of blending between RAP and virgin binders.

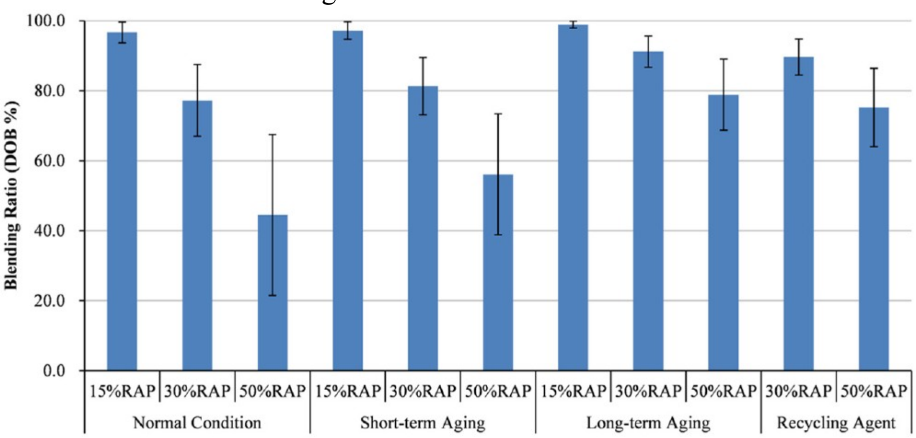


Figure 18. Summary of Degree of Blending Results from EDS SEM Analysis (Jiang et al., 2018).

3.7 Field Performance of Recycled Asphalt Mixes with Rejuvenators

Extensive laboratory studies have been carried out on the usage of rejuvenators in recycled mixes (Tran et al., 2012; Mogawer et al., 2013; Zaumanis et al., 2014; Zaumanis et al., 2015; Cooper Jr et al., 2015). In these studies, rheological evaluation of the recycled binder blends for PG grading was performed. Furthermore, mixture tests for moisture damage, permanent deformation, mixture stiffness, low temperature, and top-down cracking were performed on the recycled mixes. Despite some studies showing improvement in the cracking resistance when rejuvenators were incorporated in recycled mixes with high RAP contents, some studies showed that the rejuvenators were not effective in the mixture rutting and moisture susceptibility (Mogawer et al., 2013; Lee et al., 2018).

In the most recent NAPA survey, the percentage of RAP mixes incorporating asphalt rejuvenators has fluctuated year to year with 4 percent in 2017, 7 percent in 2016, and 3 percent in 2015 (Williams et al., 2018). Nevertheless, only few documented field projects of high RAP/RAS mixes with rejuvenators are available in the United States. In 2017, NCAT conducted a study for the Alabama Department of Transportation (ALDOT) to evaluate the performance of two rejuvenators in 12.5mm NMAS recycled mixes containing a PG 67-22 virgin binder (Xie et al., 2017). The control mix contained 20% RAP and was produced as hot mix asphalt (HMA). The mixes with the two rejuvenators were produced as warm mix asphalt (WMA) and contained 25% RAP and 5% RAS. The control mix contained a liquid anti-stripping additive, which was added at the rate of 0.80% by weight of the virgin binder. The rejuvenators used in this study were composed of fatty acids derivates (Product I), and fast pyrolysis of pine trees (Product II). The dosage of Product I was 7.8% and Product II was 13.8% as recommended by the manufacturer. For the mix with Product I, an anti-stripping additive was included at a rate of 0.52% by weight of the virgin binder. For the mix with Product II, a different anti-stripping additive was added at a rate of 0.43% by weight of the virgin binder. After being subjected to traffic loading for two years, the average International Roughness Index (IRI) of the control, Product I, and Product II test sections were 54.5 in./mi., 59 in./mi., and 45 in./mi, respectively. Furthermore, the rut depth of the control, Product I, and Product II test sections were 0.18 in., 0.19 in., and 0.25 in., respectively. All test sections showed a good ride quality and rutting performance, with IRI and rut depth less than the FHWA's recommended thresholds of 95 in./mi. and 0.5 in., respectively. Moreover, low-severity level longitudinal cracking was observed on the northbound outside lane of the control mix test section, while no cracking was observed on the inside lane of the southbound. The test sections with rejuvenators showed higher level of cracking severity. The test section with Product I showed mainly alligator cracking, while the section with Product II showed mostly longitudinal and transverse cracking. Based on these cracking differences, the authors concluded that the rejuvenators used were not effective for recycled mixes with 25% RAP and 5% RAS.

Kaseer et al. (2018) evaluated the field performance of asphalt mixes containing rejuvenators in Texas, Nevada, Indiana, Wisconsin, and Delaware. The Texas test sections comprised of a control section and a rejuvenated section, both with 0.28 recycled binder replacement (RBR) (0.1 RAP + 0.18 RAS). While the control mix included a WMA additive to aid with compactability during construction, the rejuvenated mix included 2.7% tall oil rejuvenator to soften the aged binder. For the Nevada project, the control mix contained 0.15 RBR (all from RAP), while two rejuvenated mixes contained 0.3 RBR with 2% tall oil and 2% aromatic extract as rejuvenators, respectively. For the Indiana project, the control mix contained 0.32 RBR (0.25 RAP + 0.07 RAS) while the rejuvenated mix contained 0.42 RBR (0.14 RAP + 0.28 RAS) and 3.5% tall oil rejuvenator. For the Wisconsin project, the control and rejuvenated test sections contained 0.22 and 0.31 RBR (all from RAP), respectively. The rejuvenated mix contained 1.2% modified vegetable oil.

Finally, in the Delaware project, the control mix and the rejuvenated mix contained 0.34 RBR (0.17 RAP + 0.17 RAS) and 0.41 RBR (0.24 RAP + 0.17 RAS), respectively. The rejuvenated mix was dosed with 0.8% tall oil. The Texas test sections were designed to assess the effectiveness of rejuvenators on improving the performance of asphalt mixes with the same RBR content, while the Nevada, Indiana, Wisconsin, and Delaware test sections were designed to evaluate the performance of rejuvenated mixes with higher RBR content as compared to those currently used by the DOTs.

All test sections were opened to traffic for a duration of time ranging from one year (Wisconsin and Delaware test sections) to three years (Texas test sections). In Texas, visual field distress survey indicated moderate cracking for the rejuvenated section as compared to low-severity cracking observed on the control section. After two years of service, the control mix in Indiana exhibited minimum visible cracking whereas the rejuvenated mix showed a significant amount of low-severity transverse and longitudinal cracking and some alligator cracking. The Delaware and Nevada projects showed no or minimal visible cracking on both the control and rejuvenated mixes after one and two years of service, respectively. Low-severity cracking was observed on both the control and rejuvenated mixes for the Wisconsin test sections. However, the underlaying pavement layer on the Wisconsin test section was a Portland cement concrete layer with existing transverse cracking, which likely facilitated the reflective cracking of the surface of the asphalt mixture.

For all test sections, field cores were obtained soon after construction and tested for resilient modulus and cracking susceptibility (I-FIT test) as per ASTM D7369 and AASHTO TP124. Statistical analysis of the control and rejuvenated mixes of their resilient moduli using Tukey's HSD analysis showed that the mixes placed in Texas, Wisconsin, and Delaware test sections had statistically equivalent stiffness. Due to the higher RBR, the rejuvenated mixes in Nevada and Indiana exhibited higher resilient modulus as compared to the control mixes. All mixes with rejuvenators were found to be more susceptible to cracking as compared to the control mixes, since they exhibited statistically low flexibility index in the I-FIT test. Based on the visual distress survey and laboratory testing of the field cores, the authors concluded that the addition of asphalt rejuvenators did not necessarily improve the cracking resistance of high RBR mixes as compared to the control mixes.

While the aforementioned studies have showed that rejuvenators were not effective in improving the cracking resistance of asphalt mixes, there is a relative lack of understanding on the full effect and implications of rejuvenator usage. Due to the numerous choices and alternatives of products in the market, a comprehensive study of the effect of these new additives on short- and long-term performance is necessary to the asphalt industry. Therefore, research studies dedicated to performance and compositional evaluation of this class of additives is in need.

3.8 Literature Review Summary

Key findings of the literature review are summarized as follows:

Classification of Asphalt Binder Rejuvenators

- Asphalt rejuvenators refer to organic materials with chemical and physical characteristics selected to restore the properties of aged asphalt in order to target specification limits.
- Rejuvenators can be generally categorized as aromatic oils, naphthenic oils, paraffinic oils, tall oils, and fatty acids.

- Rejuvenators that are most compatible with the aromatics of the asphalt binder can reduce the viscosity and modulus of the binder but have little effect on the intermolecular agglomeration and self-assembly of the asphalt polar micelles.
- Rejuvenators showing affinity for multiple fractions of the asphalt binder reduce the viscosity of the binder through restoration of the original binder asphaltenes-to-maltenes ratio.
- Rejuvenators exhibiting low compatibility with the aromatics, asphaltenes, and resins fractions of the asphalt binder typically have paraffinic and saturated material with high crystalline fractions. Although these products can reduce the binder modulus, their effectiveness was found to diminish with aging and is not sufficient to ensure satisfactory long-term pavement performance.

<u>Determination of Optimum Dosage of Asphalt Rejuvenators</u>

The most common methods for determining the dosage of recycling agents rely on either the manufacturer recommendation, field experience, or using blending charts. Almost all rejuvenator products on the market have a specified range of dosage specified by the manufacturer, whereas determining dosage by field experience comes with the knowledge an engineer/technician has acquired while working with a particular product. Determination of rejuvenator dosage using blending charts requires measuring the physical and/or rheological properties such as viscosity, penetration, and the performance grade of the recycled binder blends with and without rejuvenators.

<u>Rheological and Chemical Characterization of Recycled Asphalt Binders with Rejuvenators</u>

- The performance of asphalt binders and mixes containing RAP/RAS is dependent on the properties of its constitutive components, and the level of blending between the aged and unaged binder is also influenced by the chemical composition of the individual binders. Therefore, the addition of rejuvenators will affect the performance of the resultant asphalt binders and mixes.
- The effectiveness of rejuvenators in improving asphalt binder and mixture performance vary significantly from product to product and is dependent on the material source and manufacturing process.
- Non-load related cracking of asphalt pavements (i.e. transverse and block cracks) are related to oxidation and hardening of the asphalt binder, which is the main concern when incorporating RAP/RAS materials in the production of HMA. Therefore, significant research efforts have been spent to evaluate the cracking resistance of recycled asphalt binders using an index parameter, such as:
  - G-R parameter from DSR master curves;
  - N_f (number of cycles to fatigue failure) from the LAS test; and
  - Creep stiffness (S), m-value, ΔT_c, and physical hardening from BBR measurements.
- Since the asphalt binder oxidation has a significant impact on age-related pavement failure, chemical testing was found important to characterize asphalt aging and understand how rejuvenators affect the chemistry and aging of asphalt binders. Six chemical techniques have been used to investigate the aging characteristics of asphalt binders in the presence of rejuvenators:
  - GPC, for evaluation of the molecular size distribution (MSD);
  - DSC, for determination of the glass transition temperature (T_g);
  - FTIR-ATR, for calculation of both carbonyl and sulfoxide groups;
  - GC-MS, for evaluation of the fatty acid content of recycling agents;
  - SARA Fractionation, for evaluation of Saturates, Aromatics, Resins and Asphaltenes; and

- AFM, for evaluation of the morphology of binders.

Performance Testing of Recycled Asphalt Mixes with Rejuvenators

- Many state highway agencies have begun or are in the process of implementing mixture performance testing in their mix design specifications, especially for asphalt mixes containing high RAP/RAS contents.
- The primary distresses of asphalt pavements in South Dakota are rutting, thermal cracking, reflection cracking, fatigue cracking, and moisture damage.
- The selection of the “best” (most promising) mixture performance tests by state highway agencies are:
  - HWTT and APA for rutting evaluation;
  - DCT and low-temperature SCB for thermal cracking evaluation;
  - OT and I-FIT for reflection cracking evaluation;
  - I-FIT and BBF for bottom-up fatigue cracking evaluation;
  - I-FIT and AMPT cyclic fatigue for top-down cracking evaluation; and
  - HWTT and TSR for moisture damage evaluation.
- Laboratory testing on recycled asphalt mixes showed that the inclusion of rejuvenators usually improved mixture overall flexibility, durability, and cracking resistance, while reducing mixture stiffness and rutting resistance. Further, certain rejuvenator products were found to increase the aging susceptibility of recycled asphalt binders and mixes.
- A few studies found that the use of rejuvenators improved the degree of blending between virgin and recycled RAP binders.

Field Performance of Recycled Asphalt Mixes with Rejuvenators

While a few field studies showed that the use of rejuvenators did not necessarily improve the long-term cracking resistance of recycled asphalt mixes, there is a relative lack of understanding the full effect and implications of rejuvenator usage. Due to the numerous choices and alternatives of products on the market, a comprehensive study of the effect of these new additives on the short- and long-term pavement performance is necessary for the asphalt industry.

4 SURVEY OF STATE DOTS

4.1 Introduction

Qualtrics online survey software was used to assess the current state of practice on the evaluation and use of asphalt binder rejuvenators in asphalt mixtures. The questionnaire presented in Appendix A was sent to state DOTs after review and approval from the SDDOT technical panel. The survey questions were intended to gather information about the current use of recycled asphalt materials (i.e., RAP and RAS), maximum contents allowed, performance tests if required, and pavement distresses. The survey also included questions related to the current use of rejuvenators in asphalt mixtures with recycled materials and how asphalt rejuvenators are evaluated and approved by state DOTs.

The survey was distributed to 50 state DOTs on August 8, 2019. A total of 41 responses were received with a response rate of 82 percent. Figure 19 shows states that responded to the survey; their responses are discussed in the following sections.

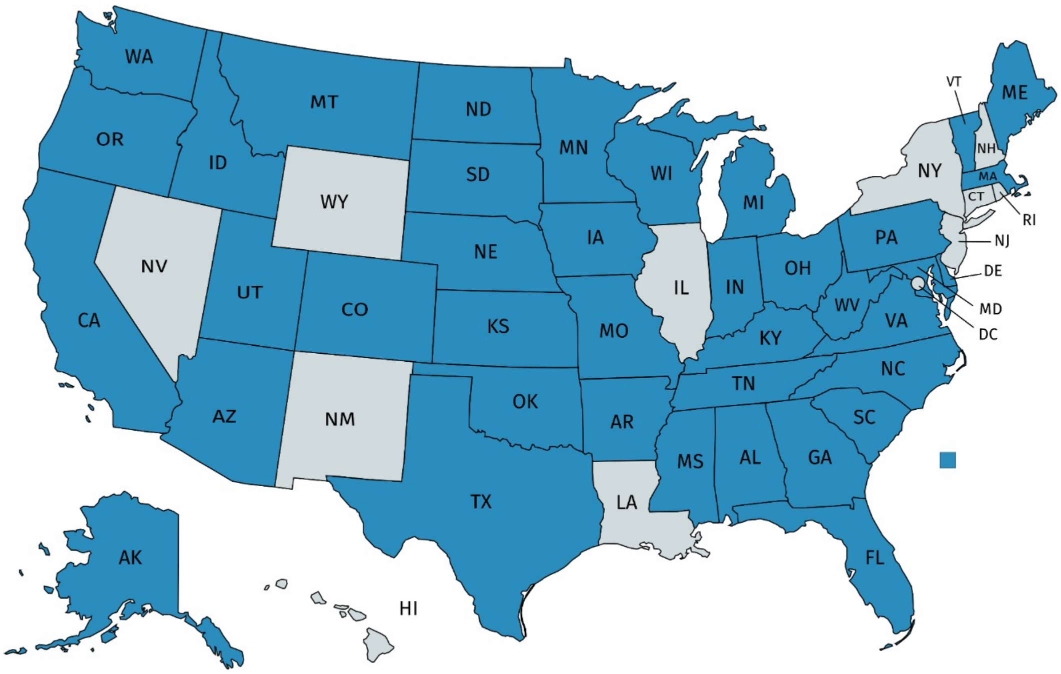


Figure 19. US Map of State DOTs Survey Responses.

4.2 RAP and RAS Current Practices

All of the 41 state DOT respondents shown in Figure 19 indicated that they currently allow the use of RAP in their asphalt mixtures. Utah indicated that although it's allowed in some surface mixtures, RAP is not allowed in specialty surface mixtures such as SMA (stone mastic asphalt), OGSC (open graded surface course), and BWC (bounded wearing course). Alaska allows the use of RAP in Marshall mixtures, but not in Superpave mixtures. With respect to RAS usage, only 27 agencies allow its use, as shown in Figure 20.

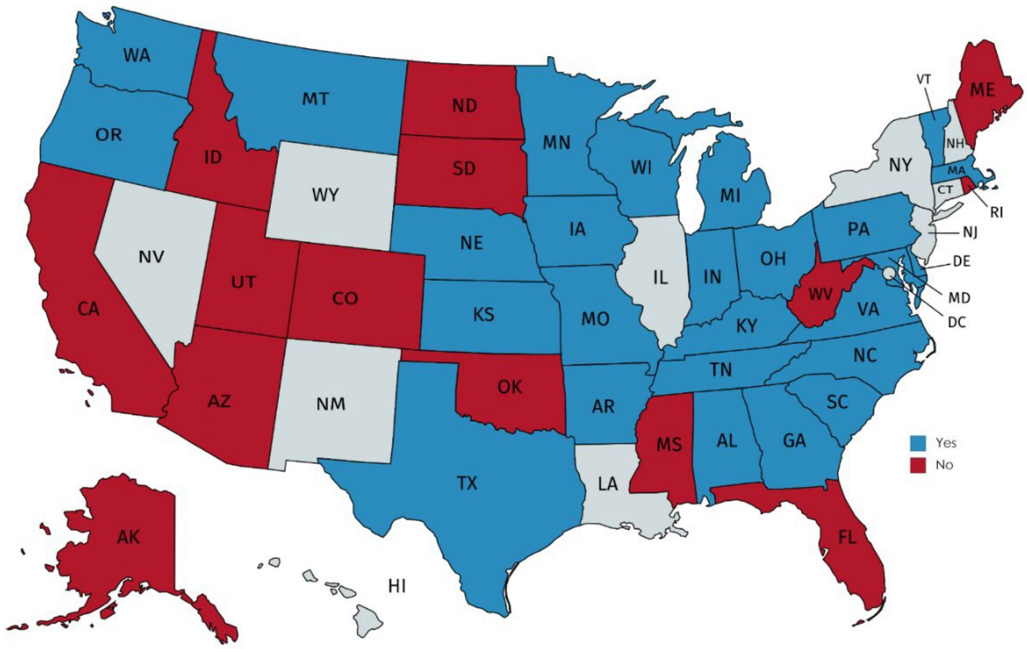


Figure 20. US Map on State DOTs that Allow Use of RAS in Asphalt Mixtures.

The survey identified three approaches used by state DOTs to specify the RAP and/or RAS percentages: (1) by asphalt binder replacement, (2) by weight of the total mix, and (3) by weight of aggregate. As shown in Figure 21 and Figure 22, most agencies specified the percentages of recycled materials by weight of the total mix or by asphalt binder replacement, but some agencies specified recycled materials percentages by weight of the aggregate.

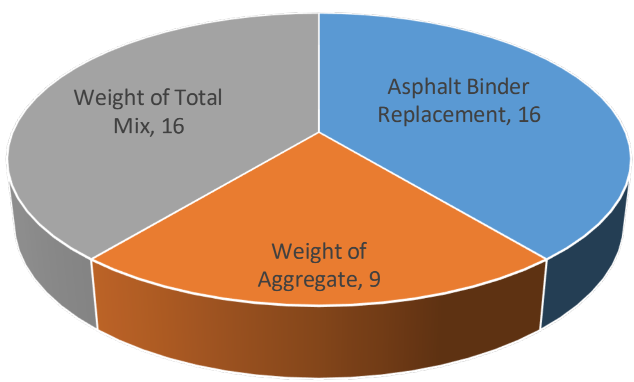


Figure 21. State DOTs Practice to Specify RAP Content.

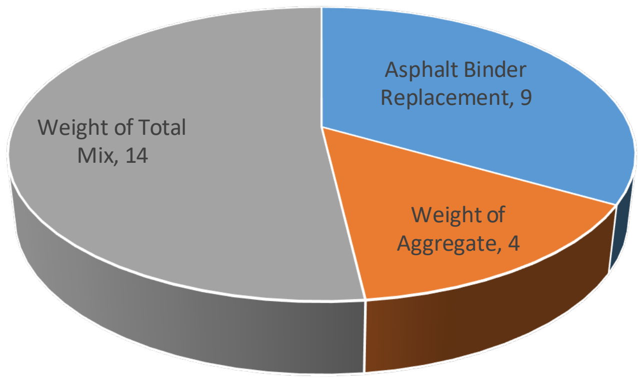


Figure 22. State DOTs Practice to Specify RAS Content.

The survey asked the respondents about their maximum allowable RAP percentages in their surface, intermediate, and binder layers. Figure 23, Figure 24, and Figure 25 show the responses specified by asphalt binder replacement, by weight of the total mix, and by weight of the aggregate. Although the maximum allowable RAP is different depending on how RAP content is reported, most responses indicated that the range is between 21 to 30 percent (or 21-30 binder replacement) for surface layers, and more than 30 percent for intermediate and binder layers. A few respondents indicated that they do not specify a maximum allowable RAP percentage. For these cases, agencies typically require additional testing to ensure that the composite binder complies with the project's binder PG requirement. As an example, this evaluation may require the final grade of the blended binder to meet AASHTO M 323 requirements through blending chart analysis.

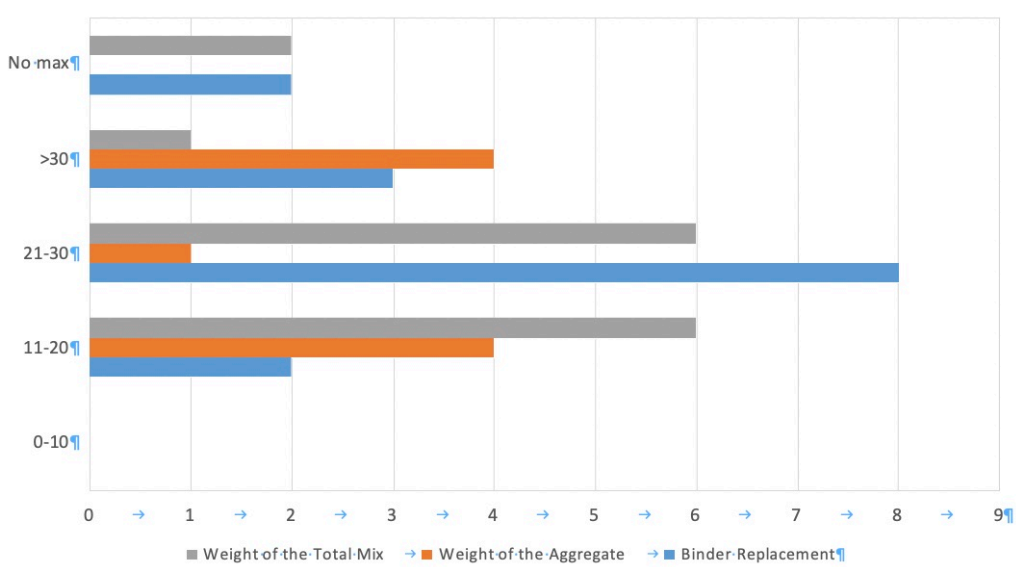


Figure 23. Maximum Allowable Percentages of RAP in Surface Layers.

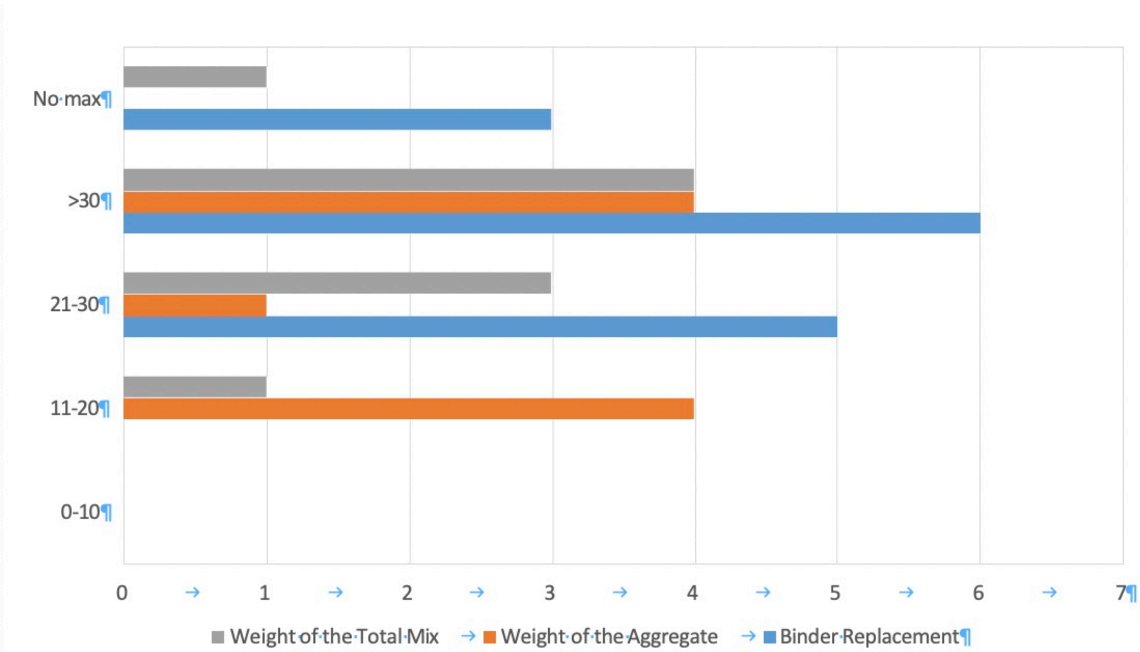


Figure 24. Maximum Allowable Percentages of RAP in Intermediate Layers.

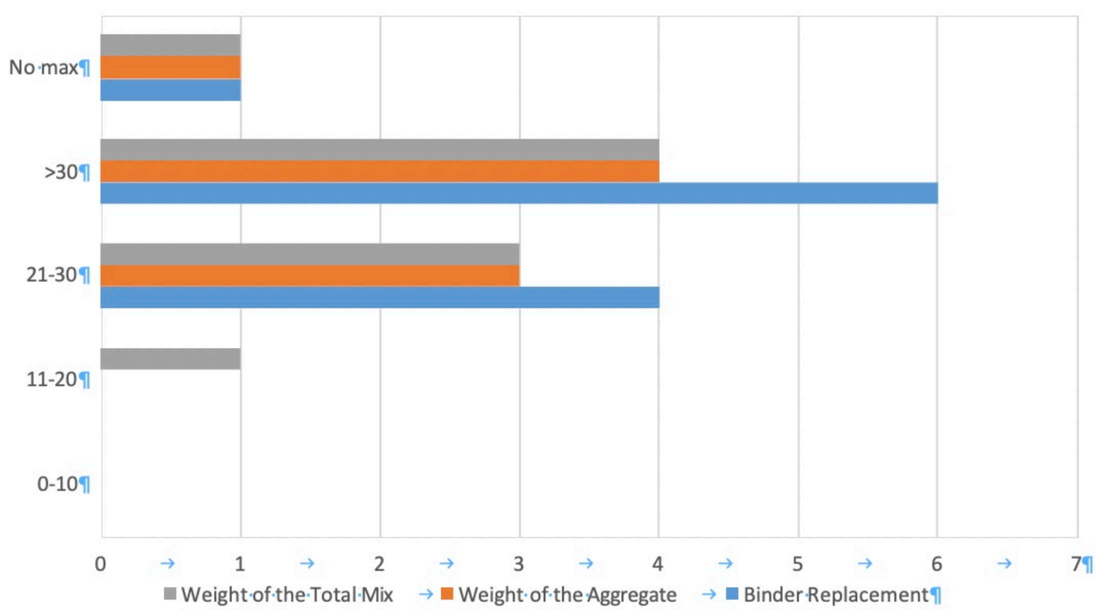


Figure 25. Maximum Allowable Percentages of RAP in Binder Layers.

With respect to the use of RAS, the survey asked the respondents about their maximum allowable RAS percentages in their surface, intermediate, and binder layers. Figure 26, Figure 27, and Figure 28 show the responses specified by weight of the total mix, and by weight of aggregate. For agencies that specified RAS by weight of the aggregate or by weight of the mix, the majority allow the use of more than 3 percent, but not exceeding 5 percent. When RAS is specified by binder replacement, most respondents indicated that the range is between 0 to 20 percent regardless of the type of layer. Two agencies allow more than 20 percent RAS binder replacement, but not exceeding 25 percent.

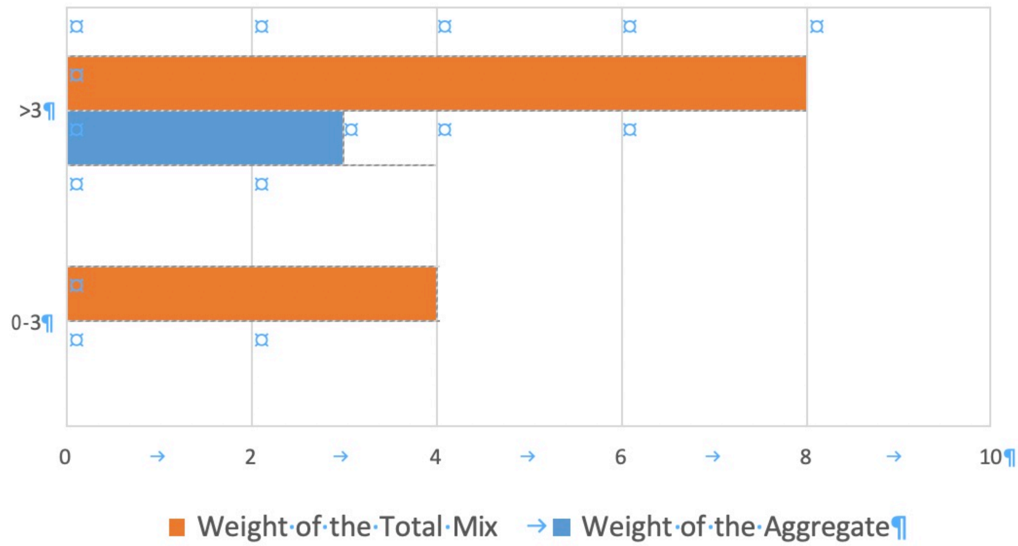


Figure 26. Maximum Allowable Percentages of RAS in Surface Mixtures.

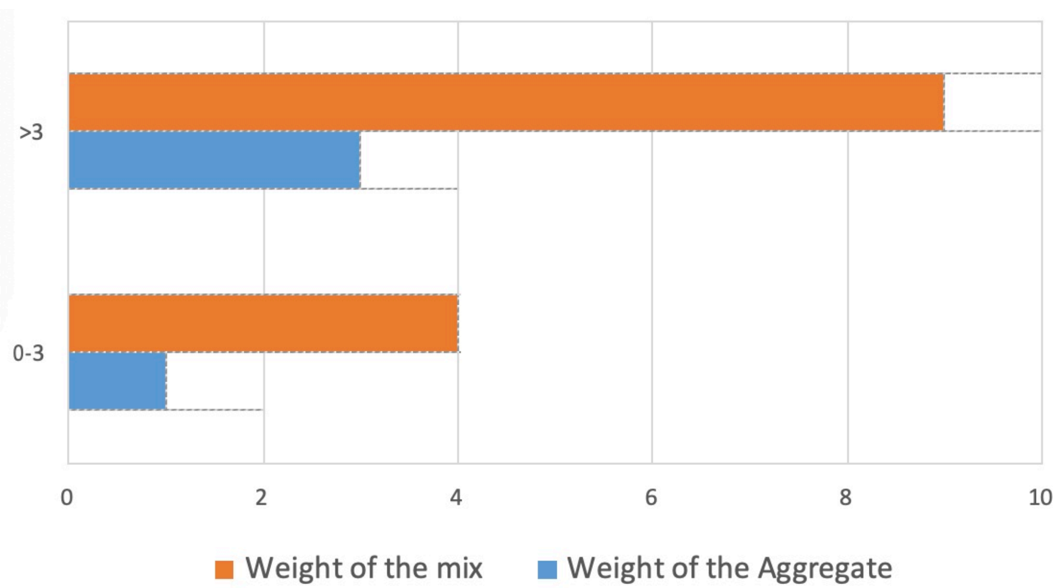


Figure 27. Maximum Allowable Percentages of RAS in Intermediate Layers.

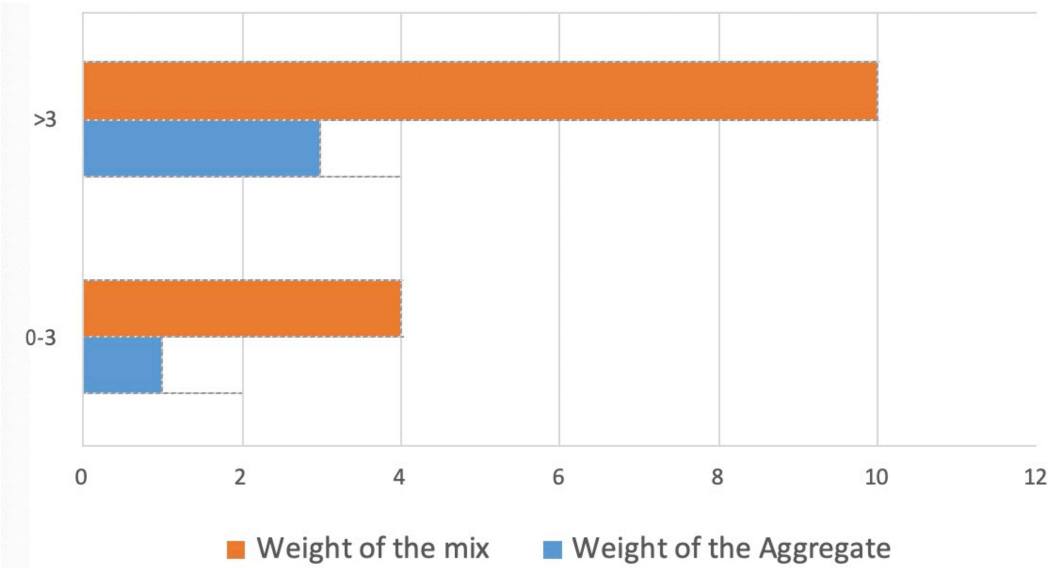


Figure 28. Maximum Allowable Percentages of RAS in Binder Layers.

State DOTs were also asked about their current practices for approving RAP and/or RAS materials for use in asphalt mixtures. Their responses are presented in Figure 29. Determination of gradation and asphalt content are the two most common requirements followed by measuring aggregate specific gravity, fractionation of RAP, and grading the extracted binder. Three agencies indicated that they do not have specific requirements for the approval of RAP and RAS.

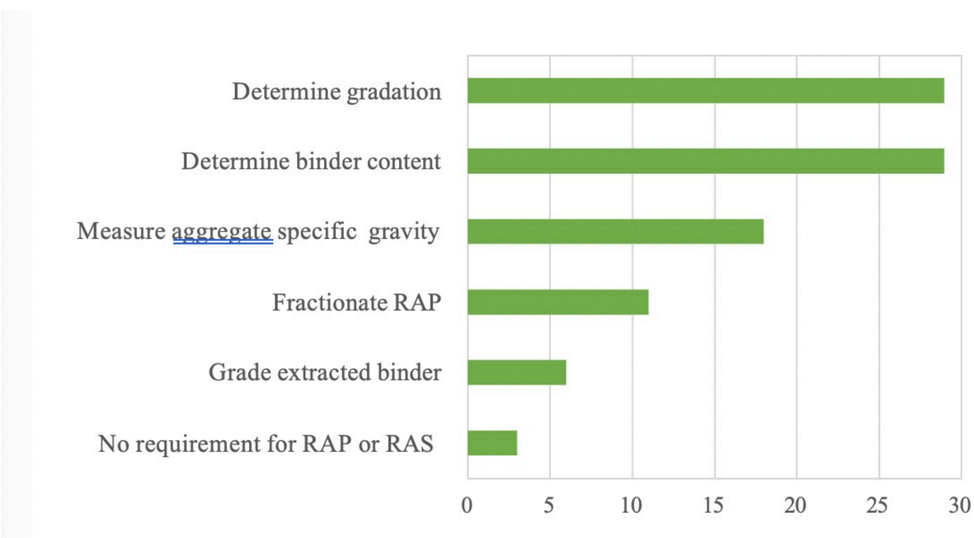


Figure 29. Current Practices for Approving RAP and/or RAS materials.

Additional specific requirements listed by agencies included:

- Ohio: RAS is given 12% AC no matter how much asphalt is in it.
- Kansas: They typically specify millings from project as RAP source and assign Gsb of RAP. The Gsb value is based on historical data (Gsb of the mix being milled). Otherwise Gse of RAP is calculated and

used as Gsb of RAP. If Gse is used then RAP is typically limited to 15% max (instead of 25% max). RAS - Gse of RAS is calculated and used as Gsb of RAS. The RAS source must meet the approval of the Engineer.

- Missouri: For RAP the Gse of the material is determined and then a correction factor of 0.98 is used to obtain the Gsb of the material.
- Idaho: RAP must come from public agency roadway or be tested and meet DOT requirements.
- Iowa: RAS must be certified by an Approved Supplier, per Iowa DOT Materials IM 506.

4.3 Pavement Distresses and Performance Tests for Mixtures with RAP and/or RAS

Respondents were asked if they had observed any distresses in surface mixtures with RAP and/or RAS. As illustrated in Figure 30, the top three most common distresses are reflection cracking, fatigue cracking, and thermal cracking. In addition, two agencies identified top-down cracking and two identified raveling as additional distresses that have been observed. One agency also reported centerline joint deterioration for surface mixtures with RAP and/or RAS. Finally, four agencies indicated that no specific distresses have been identified that can be attributed to the use of recycled materials.

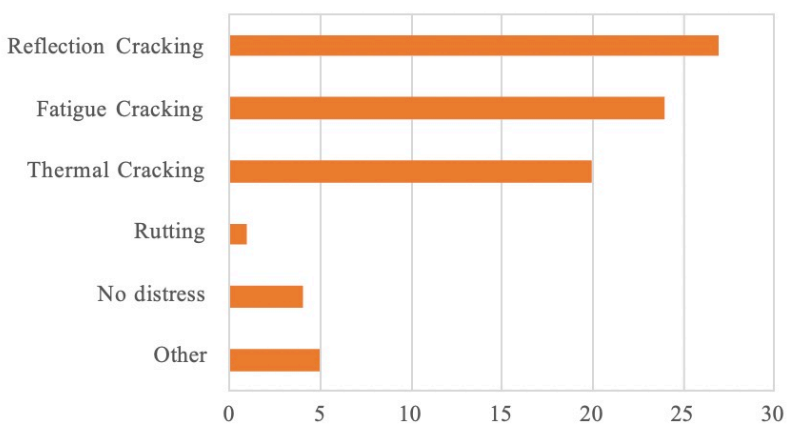


Figure 30. Pavement Distresses Observed on Mixtures with RAP and/or RAS.

With respect to performance tests required for asphalt mixtures with RAP and/or RAS, a total of 16 agencies require a rutting test; 11 of those require an AASHTO procedure that is either T 324, T 340, or T 245 as presented in Table 8, and five agencies require a state DOT procedure that is typically a modification of AASHTO T 324. A thermal cracking test is required by three agencies: AASHTO T 321 for New Jersey, TP 124 for Vermont, and ASTM D 7313 for Iowa. A fatigue cracking (either top-down or bottom up) test is required by three agencies as well; New Jersey and Iowa follow AASHTO T 321 and Vermont follows AASHTO TP 124. Finally, two agencies require a reflection cracking test: Vermont follows AASHTO TP 124 and New Jersey follows a state test procedure (New Jersey, NJ B-10).

Table 8. Performance Tests by Distress Type Required by Agencies.

Distress Evaluated	Test Procedure*	Agency
Rutting	AASHTO T 324	UT, MA, VT, CA, GA, IA
	AASHTO T 340	SD, NJ, OR, ID, NC

	AASHTO T 245	TN
	Supplement 1057	OH
	Tex-242-F	TX
	MT-334	MT
	AR 480	AR
Thermal Cracking	AASHTO T 321	NJ
	ASTM D 7313	IA
	AASHTO TP 124	VT
Reflection Cracking	AASHTO TP 124	VT
	NJ B-10	NJ
Bottom-up and Top-down Fatigue Cracking	AASHTO T 321	NJ, IA
	AASHTO TP 124	VT

*AASHTO TP 124, Determining the Fracture Potential of Asphalt Mixtures Using Semicircular Bend Geometry (SCB) at Intermediate Temperature. AASHTO T 324, Hamburg Wheel-Tracking Testing of Compacted Asphalt Mixtures. T 340, Determining Rutting Susceptibility of HMA Using the Asphalt Pavement Analyzer (APA). AASHTO T 245, Standard Method of Test Resistance to Plastic Flow of Asphalt Mixtures using Marshall Apparatus, Supplement 1057-Loaded Wheel Tester Asphalt Mix Rut Testing Method, Tex-242-F- Hamburg Wheel-Tracking Test, MT-334-Method of Test for Hamburg Wheel-Track Testing of Compacted Bituminous Mixtures, AR 480-Determining Rutting Susceptibility Using a Loaded Wheel Tester (LWT) ASTM D7313, Determining Fracture Energy of Asphalt-Aggregate Mixtures Using the Disk-Shaped Compact Tension Geometry.

4.4 Current Practice for Use of Rejuvenators

Regarding the use of rejuvenators, agencies were asked if they require or allow the use of rejuvenators in asphalt mixtures with RAP and/or RAS materials. As presented in Figure 31, the majority of the responses indicate that rejuvenators are currently not required or allowed. Only six agencies allow the use of rejuvenators; they are: Idaho, Missouri, Montana, New Jersey, Oregon, and Wisconsin.

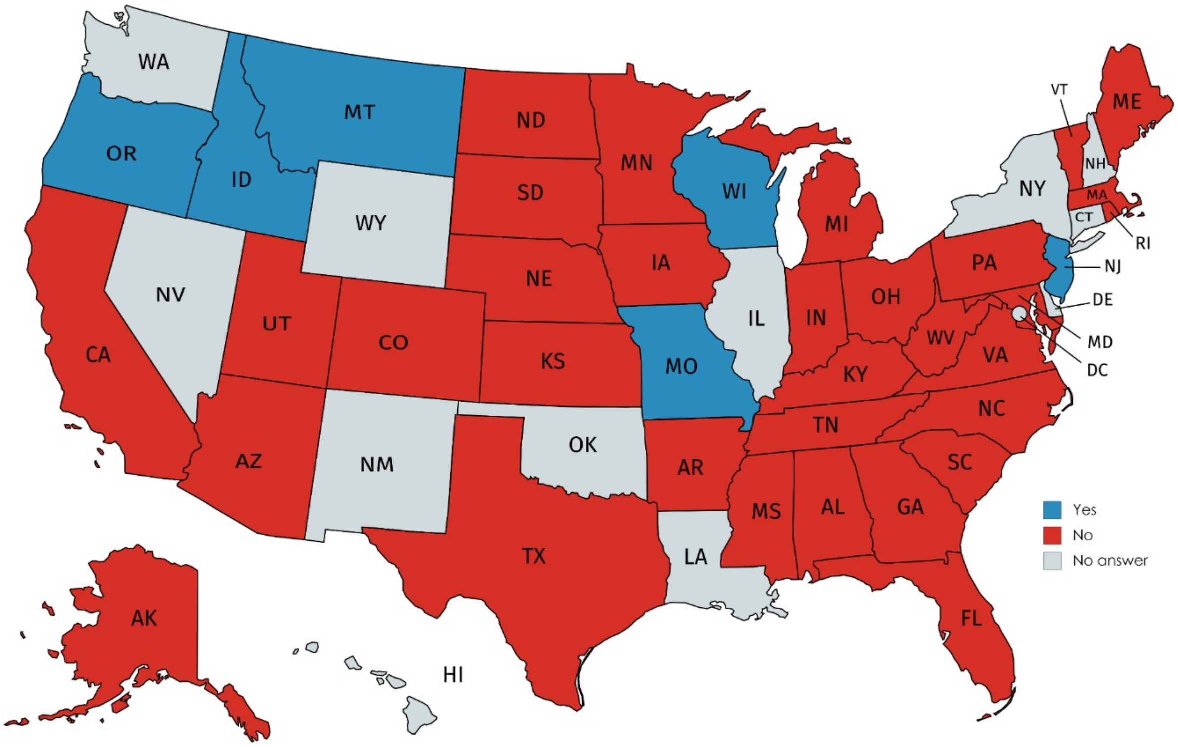


Figure 31. Current Rejuvenators Practice in Asphalt Mixtures

For the six agencies that allow the use of rejuvenators, they were asked if they have criteria to decide when rejuvenators should be used in their asphalt mixtures. Five agencies indicated that they do not have such a criterion. New Jersey indicated that when high RAP mixtures are used, the contractor needs to work with the rejuvenator supplier to determine a dosage rate to achieve the level of performance required. When the agencies were asked how they evaluate and approve rejuvenators, similarly to the previous questions, respondents indicated that it is not specified, or the rejuvenators are not evaluated separately. One agency reported that “they are silent on rejuvenator, they are neither required nor disallowed”.

Among the six agencies that allow the use of rejuvenators, only one indicated that they have an approved products list. Rejuvenator products identified from this list include Evoflex CA, Evoflex CA-7 and Evoflex 8182 from Ingevity, and JIVE from POET, as well as Hydrogreen and BITUTECH RAP from Green Asphalt Technologies (both listed as past products). When agencies were asked how they determine the dosage of rejuvenators to be used, New Jersey indicated that they verify the rejuvenator dosage rate by conducting a rutting and a cracking test on the resultant mixtures while other agencies do not specify the how the dosage rate of rejuvenators is determined. Agencies that allow the use of rejuvenators were also asked if they would accept testing from the AASHTO National Transportation Product Evaluation Program (NTPEP) in lieu of conducting in-house testing for evaluation and approval of asphalt rejuvenators. All of them had a positive response.

The last question of the survey asked if the agencies had constructed field sections using RAP and/or RAS asphalt mixtures with rejuvenators. Only two agencies had a positive response: Wisconsin and Missouri. Wisconsin indicated that their test section was included as part of NCHRP Project 9-58 “The Effects of Recycling Agents on Asphalt Mixtures with High RAS and RAP Binder Ratios”, which was completed in 2018. Missouri did not provide detailed information about their field section.

4.5 Survey Results Summary

A survey was conducted as part of project SD 2018-07 to obtain information from state DOTs regarding practices for the use of recycled materials and rejuvenators. A total of 41 agencies submitted responses. The survey shows that all of the 41 agencies that submitted responses allow the use of RAP, but only 27 allow the use of RAS. Although many states allow the use of RAP and/or RAS, some have restrictions in the type of mixtures that recycled materials can be used (e.g., not allowed in some specialty mixtures or surface mixtures).

Agencies specify RAP and/or RAS percentages either by asphalt binder replacement, by weight of the total mix, or by weight of aggregate. Therefore, it is important to specify how the percentage of recycled materials is specified to avoid confusions. Typical maximum RAP content allowed by agencies is 21 to 30 percent by asphalt binder replacement (or binder replacement ratio). When RAS is allowed, typical content is 0 to 20 percent when specified by asphalt binder replacement, or 3 to 5 percent when specified by weight of the aggregate or the mixture.

The top three common distresses observed in surface mixtures with RAP and/or RAS are reflection cracking, fatigue cracking, and thermal cracking. From the responses received, only three agencies require the use of cracking tests for design of RAP and/or RAS asphalt mixtures: New Jersey, Vermont, and Iowa.

The survey responses indicated that only six agencies currently allow the use of rejuvenators: Idaho, Missouri, Montana, New Jersey, Oregon, and Wisconsin. However, none of the agencies has an established procedure or criteria for evaluation and approval of rejuvenators.

5 EXPERIMENTAL PLAN

The experimental plan developed for this project is presented in Figure 32. It included the evaluation of virgin and RAP binders, recycled binder blends, and mixtures with and without rejuvenators. In addition, rheological and chemical evaluation of recycled binder blends and mixture performance tests to evaluate rutting, intermediate temperature cracking, and low temperature cracking resistance were conducted. The testing plan was divided into two subtasks as presented in the following sections.

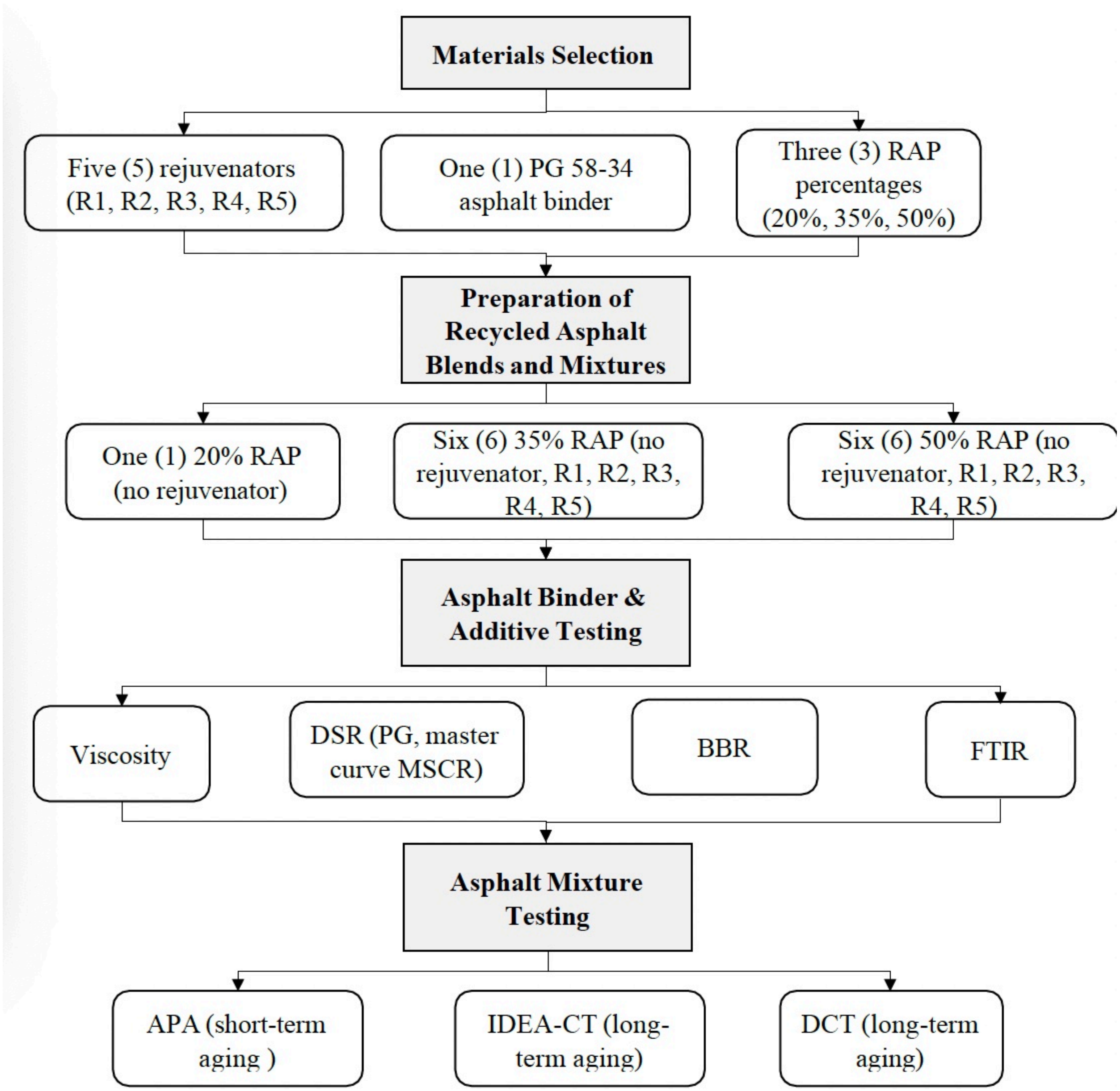


Figure 32. Experimental Plan

5.1 Evaluation of the Effectiveness of Rejuvenator Additives on Restoring Binder Properties

The objective of this subtask was to investigate the effectiveness of rejuvenators on restoring the properties of the asphalt binder after exposure to oxidative aging. In order to develop applicable results for SDDOT, an asphalt binder PG 58-34 supplied by SDDOT was used. This binder was blended with RAP binder from a RAP material provided by SDDOT at different RAP proportions, as indicated in Table 3. Three recycled asphalt blends consisting of 20%, 35%, and 50% extracted RAP binders were utilized. The 20% RAP blend represents the current SDDOT's practice on the use of RAP in mainline mix designs and was considered the control blend for performance evaluation. In addition, five different rejuvenator additives (RAs) selected by the SDDOT were blended with the higher RAP dosages (i.e., 35% and 50%) for investigation of their capability of decreasing the detrimental effects of aging on the rheological properties of the resultant binder. As presented in Table 9, thirteen blends with and without rejuvenators were evaluated.

Table 9. Testing Matrix for Asphalt Binder Testing.

Material/Blending Ratios	Factor	Description
Virgin Binder	1	Asphalt Binder (PG 58-34)
RAP Binder	1	One RAP source
Blending Ratio Neat/RAP and Neat/RAP/RA	13	Neat + 20% RAP Neat + 35% RAP Neat + 50% RAP Neat + 35% RAP + RA (i.e., RA_1, RA_2, RA_3, RA_4, RA_5) Neat + 50% RAP + RA (i.e., RA_1, RA_2, RA_3, RA_4, RA_5)

The binder blended binders along with the virgin binder were evaluated in accordance with the tests described in Table 10. The virgin and blended binders will be subjected to aging simulated in the Rolling Thin-Film Oven (RTFO, AASHTO T 240) and the PAV. Since the hardening of asphalt binder during the service period of the pavement (long-term aging) is mainly due to oxidation, the effects of an extended cycle of PAV aging (i.e., 60 hours) was investigated.

Table 10. Binder Testing for Materials Characterization.

Property	Test Type	Standard	Testing		Research Parameter
			Conditions	Aging Level	
Flow Properties	Rotational Viscosity	AASHTO T 316	@ 135°C	Unaged and RTFO+PAV	η
PG Grading	Dynamic Shear Rheometer (DSR)	AASHTO M 320	@ High and Intermediate Temp.	Unaged and RTFO	$\|G^*\|/\sin\delta$, $\|G^*\|$, δ, η^*
Intermediate Temperature Cracking Resistance	DSR Mastercurve	AASHTO T 315	Frequency Sweep (range of 0.1 to 10 rad/s), and Temp. over the range of 10-70°C	Unaged and RTFO+PAV	Glover-Rowe parameter $[G^*(\cos\delta)^2/\sin\delta]$
Low Temperature Cracking Resistance	Bending Beam Rheometer (BBR)	AASHTO T 313	@ Low PG Temp.	RTFO+PAV	Stiffness, m-value, ΔT_c

Oxidative Aging Products	FTIR-ATR	Pre-normative FTIR method	Scans acquired at region of 4000 – 650 cm^{-1} with a resolution of 4 cm^{-1}	Unaged and RTFO+PAV	Carbonyl and Sulfoxide Functions

High Temperature: At high temperature, viscosity results were used to evaluate the effects of rejuvenators on restoring the overall binder resistance to flow. Furthermore, complex shear modulus (|G*|) and phase angle (δ) were utilized to evaluate the effect of rejuvenators on the total response [i.e., elastic (recoverable) and viscous (non-recoverable)] of the asphalt binders to load.

Intermediate Temperature: To investigate the cracking potential of the binders containing rejuvenators, in the DSR a 1% strain amplitude was applied using a frequency sweep over the range of 0.1 to 10 rad/s and seven testing temperatures (i.e., 10, 20, 30, 40, 50, 60 and 70°C). The data was used to produce a master curve using the principle of time-temperature superposition and a fit to the Christensen-Anderson model (Christensen and Anderson, 1992) at reference temperature of 15°C. The master curves were then utilized to calculate the Glover-Rowe (*G-R*) parameter. The *G-R* parameter considers both binder stiffness and embrittlement and offers an indication of the cracking potential of asphalt binders at intermediate temperature. It is known that asphalt binders with higher value of *G-R* experienced a higher level of oxidative aging than those with lower values of *G-R* parameter.

Low Temperature: For low-temperature investigation of the effectiveness of rejuvenators on binder rheological properties, Stiffness, m-value and ΔT_c were used. Creep stiffness (S) is related to thermal stresses that occurs in the asphalt pavement due to shrinkage; and the m-value is related to the ability of the asphalt pavement to relax these stresses. The ΔT_c parameter was used as a means of indexing the non-load associated cracking potential of asphalt binders containing rejuvenators.

Oxidative Aging Products: The principal cause of asphalt aging and embrittlement in service is the atmospheric oxidation of molecules with the formation of highly polar and strongly interacting functional groups containing oxygen (Petersen, 2009). Therefore, binder oxidation has a significant impact on age-related pavement failure, since through oxidation the binder becomes stiffer and more brittle reducing the performance of the pavement (Petersen et al., 1993). As asphalts age, they harden; this results in a progressive increase in the stiffness of the asphalt, together with a reduction in its stress relaxation capability.

Since the level of interaction between rejuvenators and asphalt binders is influenced by the chemical composition of the individual binders, Fourier Transform Infrared Spectroscopy by Attenuated Total Reflectance (FTIR-ATR) analysis was performed to investigate the impact of rejuvenators on the chemical composition of the resulting blends. According to the literature, the change in chemical structure of asphalt binders can be obtained with the calculation of functional and structural indices of some groups from FTIR-ATR spectra, since with oxidative aging the absorbance bands representing oxygen-containing functionalities (i.e., ketones, sulfoxides, dicarboxylic anhydrides, and carboxylic acids) of asphalt increase (Petersen, 2009). Thus, to quantify oxidation-related changes collected by means of infrared absorption, band areas values can be used to calculate chemical changes in carbonyl and sulfoxide groups.

5.2 Evaluation of the Effectiveness of Rejuvenator Additives in Improving Performance Characteristics of Asphalt Mixtures

Materials and Mix Designs

The objective of this subtask was to characterize the performance of recycled asphalt mixtures containing high RAP contents and rejuvenators. Virgin aggregates for this project were supplied by a contractor from South Dakota whereas RAP was supplied by the SDDOT. Along with the raw material, a preliminary mix design (aggregate blend only with no RAP) was provided as a preliminary baseline mix. A total of eleven mixes were included in this study as presented in Table 11. Mixtures were designed to attain asphalt binder replacements (ABR) of 20% ABR, 35%ABR, and 50%ABR. The 20% ABR mix was used as the "control mix" for performance comparison in this study since 20% ABR is currently the ABR content currently allowed in South Dakota asphalt mixtures. The 35% ABR and 50% ABR mixes were prepared with and without rejuvenators. The base binder was a PG 58-34 modified with Styrene-Butadiene-Styrene (SBS).

Four bio-based and one petroleum-based (softener/flux) rejuvenators were used. They are referenced as

RA_1, RA_2, RA_3, RA_4, and RA_5 in this report. RA_1 and RA_2 are obtained from modified vegetable oils; whereas RA_3 is obtained from derivates of fatty acids and RA_4 comes from modified paper by-products. Table 12 presents the particle size distribution of the aggregates along with their physical properties (Table 13) as reported by the contractor.

Table 11. Asphalt Mixture Composition.

Factors	Description
Asphalt Binder Replacement, with and without rejuvenators	PG 58-34 + RAP @ 20% ABR PG 58-34 + RAP @ 35% ABR + No RA PG 58-34 + RAP @ 35% ABR + RA_1, RA_2, RA_3, RA_4 PG 58-34 + RAP @ 50% ABR + No RA PG 58-34 + RAP @ 50% ABR + RA_1, RA_2, RA_3, RA_5

Table 12. Particle Size Distribution of Aggregates.

Sieve size	3/4"X5/8"	5/8"X3/8"	3/8" minus	#4X#20	Man. Sand	RAP
3/4" (19.0 mm)	100.0	100.0	100.0	100.0	100.0	100.0
5/8" (16.0 mm)	91.0	100.0	100.0	100.0	100.0	99.0
1/2" (12.5 mm)	42.0	95.0	100.0	100.0	100.0	96.0
3/8" (9.5 mm)	9.0	70.0	100.0	100.0	100.0	90.0
#4 (4.75 mm)	4.2	10.0	75.0	99.0	100.0	73.0
#8 (2.36 mm)	4.0	7.8	54.0	28.0	97.0	58.0
#16 (1.18 mm)	3.9	7.3	39.0	4.0	73.0	45.0
#40 (0.420 mm)	3.8	6.9	27.0	1.4	45.0	31.0
#200 (0.075 mm)	1.8	3.0	8.8	0.3	5.0	9.0

Table 13. Properties of Aggregates.

Property	3/4"X5/8"	5/8"X3/8"	3/8" minus	#4X#20	Man Sand
Flat and elongation (3:1)	31	42	27	-	-
Flat and elongation (5:1)	5	10	8	-	-
Sand Equivalency	-	-	76	100	86
Fine aggregate angularity	-	-	48	48	48

All mixes for this project were designed at NCAT following Superpave procedures (AASHTO R 35) and the 2015 South Dakota DOT Standard Specifications for Roads and Bridges Manual. The mixes were designed to meet class Q2 traffic requirements per South Dakota DOT as shown in Table 14. Based on SDDOT specification, class Q2 traffic level refers to truck traffic that does not exceed 249 trucks per day.

Table 14. SDDOT Specifications for a 12.5 mm-NMAS mix for Class Q2 Traffic.

Criteria	SDDOT Specs.
N_{design} gyration	50
Air voids content (V_a), %	4.0
Voids in mineral aggregates (VMA), %	>14.5
Voids filled with asphalt (VFA), %	65-80

Dust to binder (D/B) ratio	0.6-1.4

Table 15 shows the cold feed percentages for the JMFs. The rejuvenator dosage rates by weight of total binder are presented in Table 16. As requested by the SDDOT, the dosage rates of the bio-based rejuvenators were selected based on the recommendation of each supplier to target the low temperature PG grade of the 20% ABR mix. Each rejuvenator supplier was provided with virgin binder and extracted RAP binder PG characterization results conducted at NCAT. The supplier of RA_1 requested a sample of recovered RAP binder to conduct his own testing. The supplier of RA_5 indicated that there was no need to add a rejuvenator to the 35%ABR mix since the final blend would be close to a PG 64-28. The final dosage rates recommended by the suppliers ranged from 1.8 to 4.0%, with higher dosages for 50 % ABR mixes. The flux dosage rate of the 35% ABR mix was optimized at NCAT to match the low PG grade of 20%ABR mix after 60 hours of conditioning in the PAV. Although this dosage rate is not practically feasible, it was included in the evaluation to assess its performance. Since the dosage rate required for the 35% ABR mix was extremely high and closer to the maximum limit allowable, only the 35% ABR mix was evaluated with the asphalt flux.

Table 15. Material Proportions of the Aggregate Blends

Aggregate Blend	3/4"X5/8"	5/8"X3/8"	3/8" minus	#4X#20	Man. Sand	RAP
20 ABR	10.0%	15.0%	11.0%	20.0%	24.0%	20.0%
35 ABR	9.0%	15.0%	-	11.0%	31.0%	34.0%
50 ABR	9.0%	11.0%	-	11.0%	27.0%	42.0%

Table 16. Recycling Agent Dosage Rates

Binder Blend	Recycling Agent	Dosage Rate (%)
35 ABR	RA_1	1.8
	RA_2	2.8
	RA_3	2.1
	RA_4	45
50 ABR	RA_1	3.1
	RA_2	4.0
	RA_3	3.8
	RA_5	2.0

Before mixing, the asphalt binder was preheated in an oven for three to four hours at 300 ±5°F. RAP was preheated at 275±5°F for at least an hour and a half but not more than three hours. The virgin aggregates were preheated overnight at 350±10°F. Except for the control mix (20 ABR), a low-speed shear mixer (200 rpm) was used to blend the RA and the base binders for 30 ±5 minutes before mixing with the aggregates and RAP. During mixing, preheated virgin aggregates were added into the mixing bucket followed by preheated RAP and were thoroughly mixed for at most two minutes before adding a heated asphalt binder. The blend was moved to the rotary mixer and mixed until all the aggregates were coated with asphalt. Asphalt mixtures prepared for volumetrics design purposes were conditioned at 275±5ºF for two hours to simulate short-term aging and to allow asphalt absorption by the aggregates per AASHTO R 30.

Table 17 shows the JMFs of the asphalt mixtures used in this study. The table shows the final ABR of the mixtures. Only the control mix (20% ABR) met the South Dakota DOT specifications for Q2 traffic. Both the 35% ABR and 50% ABR did not meet the VMA criterion (>14.5). As mentioned previously, one should note that these mixes were designed using aggregate stockpiles originally intended for a mix that did not contain RAP. Several blends were tried, but it was not possible to meet the VMA criteria with the stockpiles provided and still keep the gradations within the primary control points of a 12.5 mm NMAS mix. The trial mixes closer to meeting the VMA criteria and all the other volumetric requirements were presented to the technical panel for approval to move forward. Note that the 50% ABR had in reality 44.4% ABR only since other trial blends with higher ABR% yielded VMA's even lower than 14. The technical panel approved to include the mixes presented in Table 17 for further testing.

Table 17. Job mix Formula for Asphalt Mixtures.

Property	20 ABR	35 ABR	50 ABR
% Passing sieve size			
3/4" (19.0 mm)	100.0	100.0	100.0
5/8" (16.0 mm)	98.9	98.9	98.8
1/2" (12.5 mm)	92.7	92.7	92.6
3/8" (9.5 mm)	84.4	83.9	84.3
#4 (4.75 mm)	68.6	68.6	70.0
#8 (2.36 mm)	48.0	54.4	54.8
#16 (1.18 mm)	33.1	39.8	40.2
#40 (0.420 mm)	21.7	26.0	26.4
#200 (0.075 mm)	4.7	5.3	5.7
Mix design information			
N_{design}, gyrations	50		
NMAS, mm	12.5		
Optimum AC @ 4% V_a	5.7	5.4	5.2
P_{be} @4 %V_a	5.1	4.6	4.3
VMA @ 4% V_a	15.5	14.4	14.0
VFA @ 4% V_a	74.1	72.6	71.4
D/B Ratio @ 4% V_a	0.92	1.15	1.31
RAP binder replacement information			
RAP Content (%)	20.0	34.0	42.0
RAP ABR (%)	19.3	34.6	44.4

Asphalt Mixture Performance Testing

Table 18 presents the mixture performance tests conducted in this study. The loose mixtures for performance testing were subjected to two aging conditions: short-term oven aging (STOA) and long-term oven aging (LTOA), both at 275°F. The loose asphalt mixtures were conditioned for STOA for four hours after mixing. For LTOA, the conditioned STOA loose asphalt mixtures were further aged for six hours but at a reduced layer thickness (less than ¾ to 1 inch thick) prior to compaction. The APA was conducted on STOA specimens to evaluate the rutting resistance of the asphalt mixtures because asphalt mixtures are most vulnerable to rutting right after construction. IDEAL-CT and DCT tests were performed on LTOA mixtures considering that asphalt mixtures tend to be more susceptible to cracking after aging due to increased mix embrittlement and reduced relaxation properties. Except for APA specimens, which were compacted to height (115±2.0 mm); specimens for IDEAL-CT and DCT were prepared to meet a target air void content of 7.0±0.5 %.

Table 18. Mixture Performance Testing.

Mixture Property	Test	Aging Condition	Performance Parameter
Rutting Resistance	APA (AASHTO T340)	STOA	Rut Depth
Intermediate Temp. Cracking Resistance	IDEAL-CT (ASTM D8225-19)	LTOA	CT_{Index}
Thermal Cracking Resistance	DCT (ASTM D7313)	LTOA	Fracture energy (G_f)

<u>Asphalt Pavement Analyzer (APA) Test</u>

The APA was conducted per AASHTO T 340-10 to evaluate the rutting resistance of the mixtures. Asphalt mixture specimens for APA were compacted to N_{design} (i.e., 50 gyrations) to a height of 115±2.0 mm and diameter of 150±2.0 mm. A total of six specimens were prepared per asphalt mix, and they were randomly divided into three pairs to reflect the testing positions (left, center, and right) in the APA setup [Figure 33 (a)]. After compaction, specimens were left to cool at room temperature overnight; then, air void contents were determined to check whether they were within the 3.0 to 5.0% air voids range, as the mixes were designed to meet a requirement of 4.0% air void contents. They were left to dry under a fan for (at most) a week. Before testing, specimens were conditioned at 58°C (high-temperature PG of the base binder) for a minimum of six hours. During testing, 100 lb. wheels are loaded onto pressurized linear hoses (100 psi.) and tracked back and forth over testing specimens for about 8000 cycles to induce rutting. The APA was run in automatic mode; the 25 initial seating cycles were first applied then the software was restarted for the actual test. Rut depths are accumulated at an interval of 100 cycles and the final value after all the cycles are complete is taken as a rut depth for that mix [Figure 33(b)]. A lower rut depth is desired for a mix resistant to rutting.

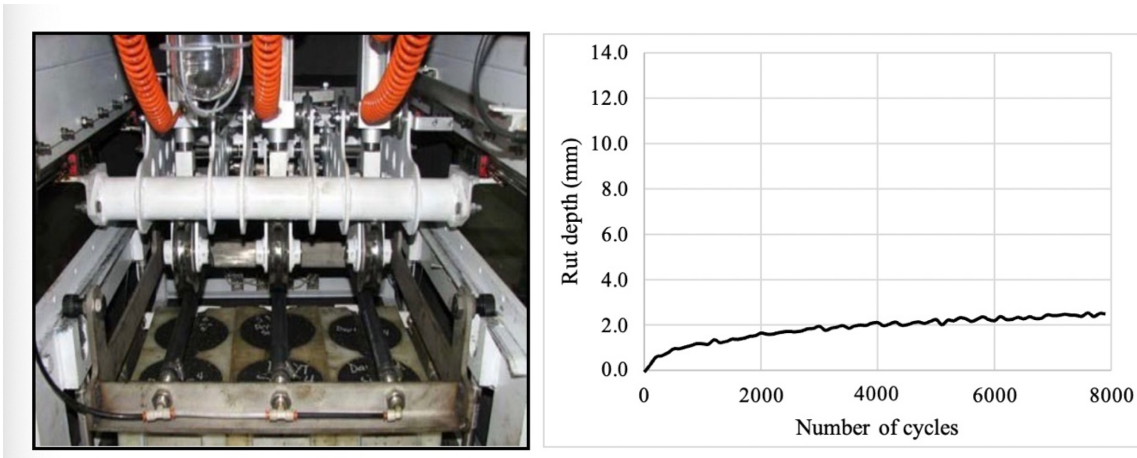


Figure 33. (a) APA experiment setup, (b) APA data analysis.

Indirect Tensile Asphalt Cracking Test (IDEAL-CT)

The IDEAL-CT was used to evaluate the intermediate-temperature cracking resistance of the asphalt mixtures. The test was conducted per ASTM D8225-19; the number of replicates per mixture ranged from five to six specimens. Before testing, specimens were conditioned in an environmental chamber at 25 ± 1.0°C for two hours ± 10 minutes. During the test, a monotonic load was applied on a gyratory specimen at a constant displacement rate of 50 mm/min as shown in Figure 34(a). Once the testing was complete, the load-displacement curve was analyzed to determine the work of fracture, which refers to the total area under the curve, and the slope of the curve at a 25 percent reduction from the peak load [Figure 34(b)]. The final test parameter, cracking tolerance index (CT_{index}), was then calculated using Equation 3. A higher CT_{index} value is desired for asphalt mixtures with better cracking resistance.

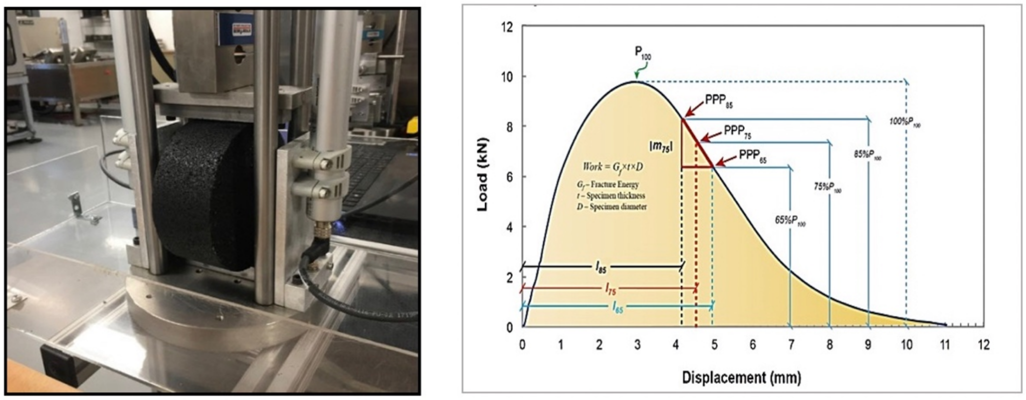


Figure 34. (a) IDEAL-CT experiment setup, (b) IDEAL-CT data analysis.

$$CT_{index} = \frac{t}{62} * \frac{l_{75}}{D} * \frac{G_f}{|m_{75}|} * 10^6 \qquad \text{Equation 3}$$

Where,

t = specimen thickness;
l_{75} = displacement at 75% of peak load;
D = specimen diameter;
G_f = fracture energy; and
$|m_{75}|$ = slope at 75% peak load.

Disc-shaped Compact Tension (DCT) Test

The DCT test was conducted following ASTM D7313-13 to assess the low-temperature cracking resistance of the mixes. Test specimens were prepared by saw-cutting a 160 mm-high by 150 mm-diameter specimen compacted to 7.5% air void contents into two halves each measuring 50 ± 5 mm thick. The halved specimens were then trimmed to possess a flat edge on one side of the specimen for studs. Then, a notch 62.5 ± 2.5 mm long was saw-cut at the center of the flat edge, followed by coring two 1-inch diameter holes on each side of the notch. The final testing specimen had a target air void content of 7.0 ± 0.5%; the number of replicates ranged from four to six specimens. Since the asphalt binders had a low-temperature PG of -34 °C, the test was conducted at -24 °C, as ASTM D 7313-13 recommends running the test at 10°C above the low PG temperature of the asphalt binder. Before testing, the specimens were conditioned in an environmental chamber for eight to sixteen hours. The test began by loading a DCT specimen in tension using metal rods that were inserted through core holes, as shown in Figure 35(a). A clip gauge was then installed over the crack mouth prior to the start of the experiment to control and record the crack mouth opening displacement (CMOD). The test was conducted in CMOD control mode with the clip gauge opening at a constant rate of 0.017 mm/sec. Figure 35(b) presents an example of the load versus CMOD behavior. For data analysis, the fracture energy (G_f) was calculated using Equation 4, where the area under the load-CMOD curve was determined through numerical integration using the trapezoidal rule. A higher G_f value is desired for asphalt mixtures with better resistance to low-temperature cracking.

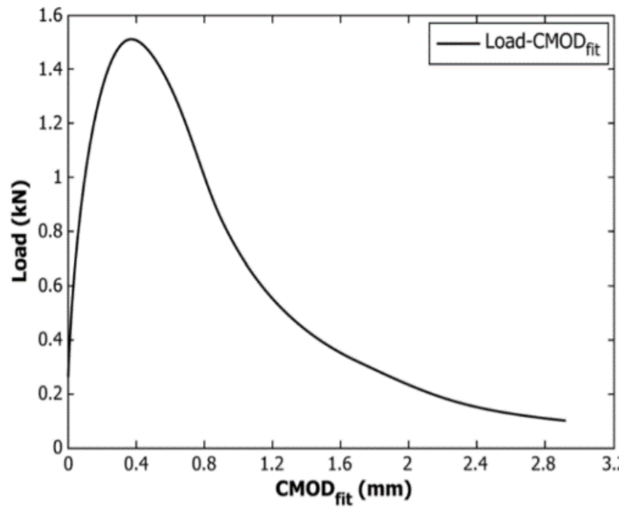


Figure 35. (a) DCT experiment setup, (b) DCT data analysis.

$$G_f = \frac{Area}{B*(W-a)}$$ Equation 4

Where,

G_f = fracture energy (J/m^2);
Area = area under load-CMOD curve;
B = specimen thickness (m); and
W-a = initial ligament length (m).

6 ANALYSIS OF LABORATORY TEST RESULTS AND FINDINGS

6.1 Analysis of Rheological and Chemical Results

This section presents the rheological and chemical testing results associated with the evaluation of the effectiveness of rejuvenators on decreasing or dismissing the detrimental effects of aging on the rheological properties of the resultant binder. Results from this evaluation will be used for the development of a procedure for the SDDOT to perform a screening process of rejuvenators for potential approval in asphalt mixtures. For simulation of oxidative aging, the Rolling Thin-Film Oven (RTFO, AASHTO T 240) was utilized for short-term aging followed by a single protocol of 60-hour in the Pressure Aging Vessel (PAV, AASHTO R 28) for simulation of long-term aging.

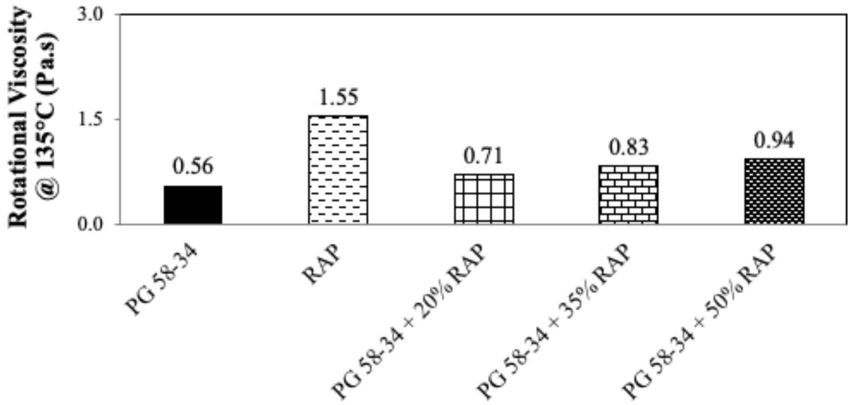
6.1.1 Rotational Viscosity

Viscosity denotes the fluid property of materials and is a measure of its resistance to flow. In this study, the viscosity measurements were performed in accordance with AASHTO T316. The rotational viscosity test measures the torque required to maintain a constant rotational speed (20 RPM) of a cylindrical spindle while submerged in the asphalt binder at a constant temperature of 135°C. This torque is then converted to a dynamic viscosity value that is used to ensure that the asphalt binder is sufficiently fluid for pumping and mixing. Table 19 presents the obtained viscosity results, before and after aging.

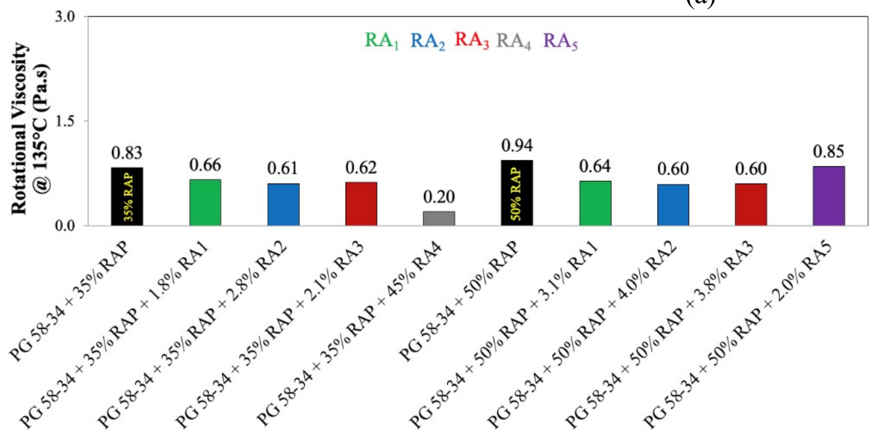
Table 19. Rotational Viscosity Results @ 135°C.

Asphalt Sample	Rotational Viscosity @ 135°C (Pa.s)	
	Unaged	RTFO plus 60-h PAV
PG 58-34	0.56	1.80
RAP	1.55	79.50
PG 58-34 + 20% RAP	0.71	8.34
PG 58-34 + 35% RAP	0.83	8.75
PG 58-34 + 50% RAP	0.94	9.13
PG 58-34 + 35% RAP + 1.8% RA_1	0.66	4.13
PG 58-34 + 35% RAP + 2.8% RA_2	0.61	4.15
PG 58-34 + 35% RAP + 2.1% RA_3	0.62	3.48
PG 58-34 + 35% RAP + 45% RA_4	0.20	1.19
PG 58-34 + 50% RAP + 3.1% RA_1	0.64	4.30
PG 58-34 + 50% RAP + 4.0% RA_2	0.60	4.55
PG 58-34 + 50% RAP + 3.8% RA_3	0.60	4.05
PG 58-34 + 50% RAP + 2.0% RA_5	0.85	8.39

The Superpave typical dynamic viscosity values for unaged asphalt binders at 135°C are 0.2 to 3 Pa.s, and all tested asphalt samples and asphalt blends showed viscosity values within this range. As expected, all samples showed an increase in viscosity after RTFO plus 60-hour PAV aging. Figure 36(a) shows that the viscosity of the RAP binder was significantly higher than the base binder PG 58-34. Moreover, it can be seen that an increase in the viscosity of the asphalt base binder PG 58-34 occurred with the addition of the RAP binder, and that this increase in viscosity was directly related to the content of the recycled material (i.e., from lower to higher viscosity @ 135°C: PG 58-34 + 20% RAP < PG 58-34 + 35% RAP < PG 58-34 + 50% RAP). As indicated in Figure 36(b), when the rejuvenators were added to the recycled asphalt binder blends, a decrease in viscosity was observed. This decrease in viscosity was found as dependent of both rejuvenator chemical composition and dosage, and influenced by the content of the recycled asphalt material.



(a)



(b)

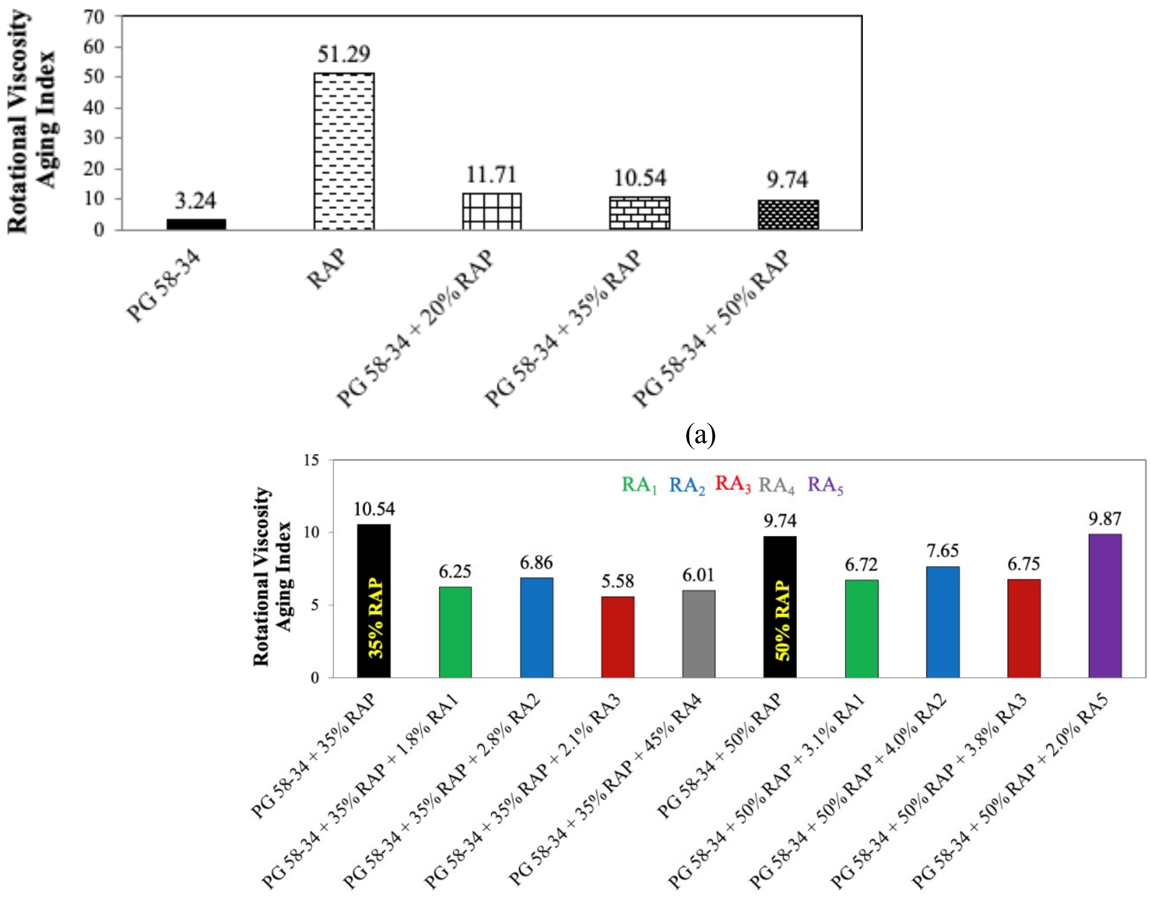
Figure 36. Rotational viscosity results @ 135°C for: (a) Base binder PG 58-34, RAP, and recycled asphalt blends, and (b) Recycled asphalt blends with and without rejuvenators.

To address the durability of the recycled asphalt blends with and without rejuvenators, an aging index was determined per Equation 5 and the results are presented in Figure 37.

$$\text{RRRRRRRRRRRRRRRRRRRR VVRRVVVVRRVVRRRRVV AAAARRRRAA IIRRIIIIII @ 135°CC PPAAPP} = \frac{\text{VVVVVVVVVVVVVVVVV}_{\text{AAAAAAAAA A RRRRRRR R + 60-h}}}{\text{VVVVVVVVVVVVVVVVV}_{\text{UUUUUUUUAAUU}}} \qquad \text{Equation 5}$$

As indicated in Figure 37(a), the base binder PG 58-34 showed the lowest aging index in comparison to the RAP binder and recycled asphalt blends. These results indicated the increased aging susceptibility of asphalts after the addition of recycled material. Figure 37(b) indicates that the addition of rejuvenators resulted in a decrease in the aging susceptibility of the control recycled asphalt blends, in terms of the viscosity aging index with one exception (PG 58-34+50%RAP+2.0%RA_5). Moreover, as observed for the unaged blends, the effect of each rejuvenator was found as dependent of both additive chemical composition and dosage, and influenced by the content of the recycled asphalt material. For example, for the recycled asphalt blends with 35% RAP, even though a dosage of 45% per weight of binder was used for the petroleum-based rejuvenator RA_4 (i.e., asphalt flux), the aging index of the PG 58-34+35%RAP+45%RA_4

asphalt blend was higher than the aging index of the of same base binder with 2.1% of bio-based rejuvenator RA_3 (aging index of 6.01 and 5.58, respectively). Furthermore, as the content of the recycled asphalt binder increased from 35% to 50%, the aging susceptibility of the recycled asphalt blends with rejuvenators also increased, regardless of the rejuvenator chemical composition or dosage.



(b)

Figure 37. Rotational viscosity aging index @ 135°C for: (a) Base binder PG 58-34, RAP, and recycled asphalt blends, and (b) Recycled asphalt blends with and without rejuvenators.

6.1.2 Superpave Performance Grade Classification

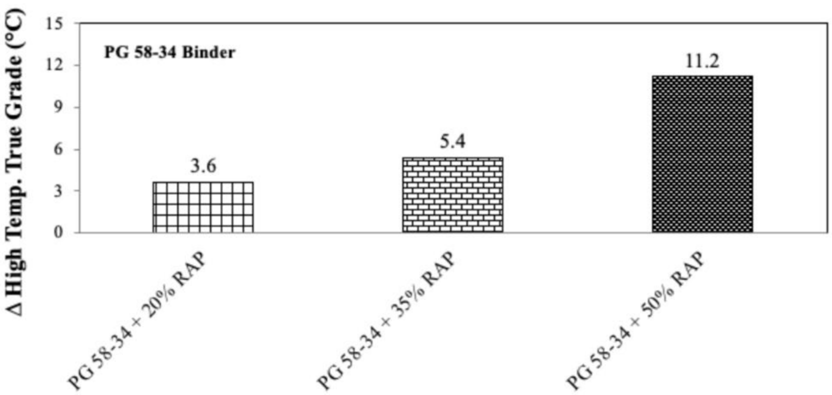
The final performance grade (PG) classifications of all asphalt binders evaluated in this study are presented in Table 20. In summary, the addition of 20, 35 and 50% recycled binder to the PG 58-34 base binder improved the rutting resistance but had a negative effect on the fatigue and thermal cracking resistance, and stress relaxation property after RTFO plus 60-hour oxidative aging in the PAV. The incorporation of the five rejuvenators evaluated in the study counterbalanced these negative effects. With exception of the blend PG 58-34 + 50% RAP + 2.0% RA_5, the addition of rejuvenators resulted in a decrease in the temperature at which the limiting fatigue parameter [|G*|.sin(δ)] was satisfied based on AASHTO M320, in comparison to the control blends (i.e., PG 58-34 + 35% RAP and PG 58-34 + 50% RAP). It is important to mention that the dosage of each rejuvenator was determined by the manufacturer by targeting the low temperature PG of the 20%RAP blend after RTFO plus 60-hour PAV aging. The only exception occurred for the bio- based rejuvenator RA_5, for which the manufacturer decided that: (a) adding RA_5 to the PG 58-34 + 35%

RAP blend was not necessary to improve the performance of the blend, and (b) for the PG 58-34 + 50% RAP recycled blend, the dosage of RA_5 should be performed targeting a PG -22 instead of -27.2.

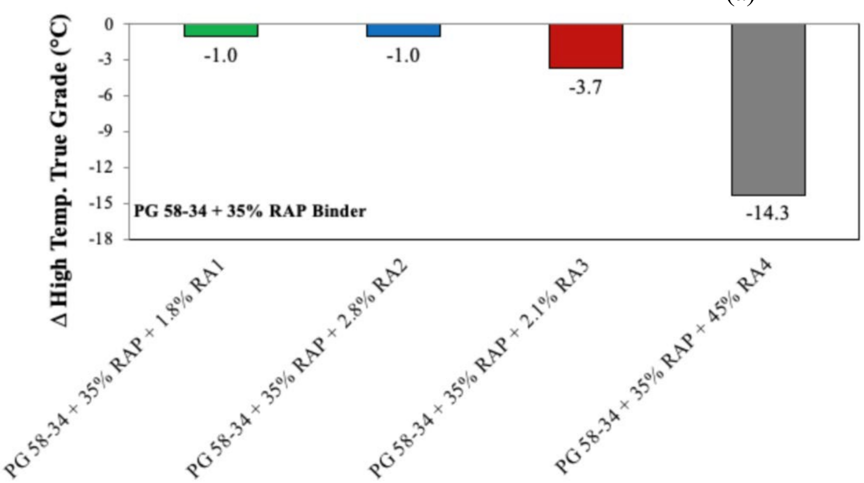
Table 20. Performance Grade Classification of Asphalt Binders at High and Low Temperatures.

Sample	Aging (RTFO + x-PAV hrs)	T_{cont}, High, Unaged °C	T_{cont}, High, RTFO °C	T_{cont}, Intermediate, °C	T_{cont}, Low S, °C	T_{cont}, Low m-value, °C	ΔT_c	PG HT	PG LT
PG 58-34	20	64.3	63.2	8.6	-36.4	-36.7	0.4	58	-34
	60			11.5	-33.8	-28.2	-5.7		-28
RAP	0	88.4	85.0	26.7	-24.7	-22.4	-2.3	82	-22
	60			36.3	-18.6	-9.1	-9.4		-4
PG 58-34 + 20% RAP	60	68.6	66.8	15.8	-31.3	-27.2	-4.1	64	-22
PG 58-34 + 35% RAP		71.9	70.2	22.8	-30.0	-23.4	-6.5	70	-22
PG 58-34 + 50% RAP		75.9	74.4	21.4	-29.0	-21.6	-7.4	70	-16
PG 58-34 + 35% RAP + 1.8% RA_1		69.5	67.6	19.9	-32.2	-28.6	-3.5	64	-28
PG 58-34 + 35% RAP + 2.8% RA_2		68.1	67.6	18.1	-33.6	-28.5	-5.1	64	-28
PG 58-34 + 35% RAP + 2.1% RA_3		68.2	64.9	20.1	-32.3	-29.4	-3.0	64	-28
PG 58-34 + 35% RAP + 45% RA_4		56.6	54.3	12.8	-35.7	-31.1	-4.5	52	-28
PG 58-34 + 50% RAP + 3.1% RA_1		69.1	69.2	18.3	-33.6	-28.0	-5.6	64	-28
PG 58-34 + 50% RAP + 4.0% RA_2		68.4	68.5	19.5	-33.3	-28.0	-5.3	64	-28
PG 58-34 + 50% RAP + 3.8% RA_3		68.6	66.0	20.9	-31.3	-28.4	-2.9	64	-28
PG 58-34 + 50% RAP + 2.0% RA_5		75.0	74.0	22.1	-30.5	-22.2	-8.2	70	-22

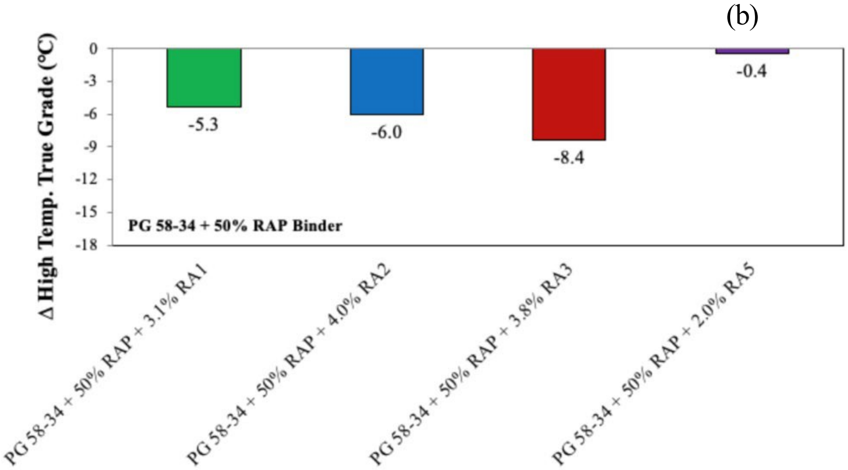
Figure 38 presents the observed change in the continuous high temperature true grade of all samples evaluated in this study, after addition of the recycled binder and rejuvenators. As indicated in Figure 38(a), the addition of 20, 35 and 50% recycled binder resulted in an increase of the high temperature true grade by 3.6, 5.4 and 11.2°C, respectively, improving the rutting resistance of the base binder 58-34. When the rejuvenators were added to the PG 58-34 + 35% RAP recycled asphalt blend, a decrease in the high pass/fail temperature was observed: RA_1 and RA_2 performed similarly decreasing the high temperature true grade by 1°C; RA_3 and RA_5 decreased the high temperature true grade by 3.7 and 14.7°C, respectively. It is important to mention that the dosage of RA_5 was 45% by weight of the binder, since this rejuvenator was petroleum-based and acted only as a softener to the recycled asphalt blend. When adding the rejuvenators to the PG 58-34 + 50% RAP recycled asphalt blend, the decrease in the high pass/fail temperature by RA_1, RA_2, RA_3 and RA_5 was by 5.3, 6.0, 8.4, and 0.4°C, respectively.



(a)



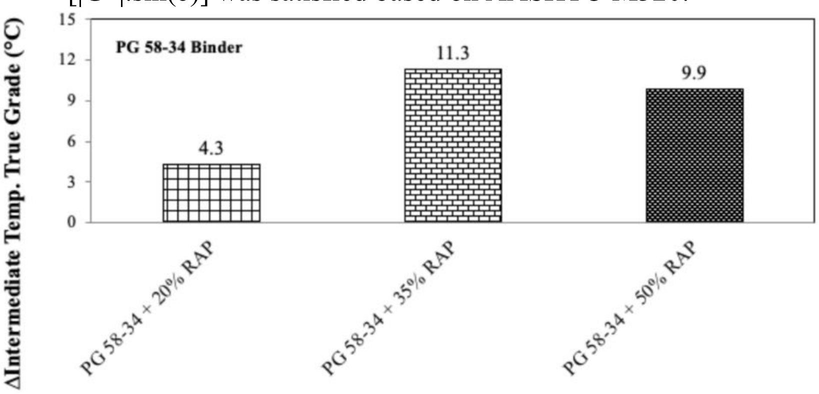
(b)



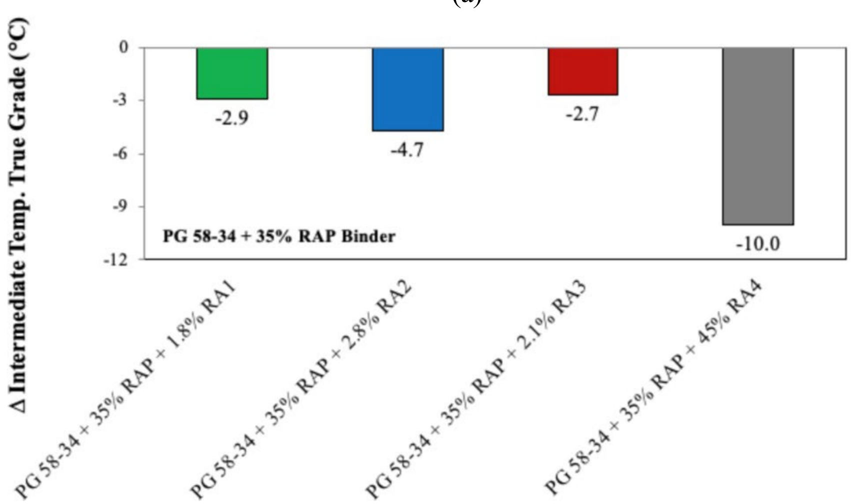
(c)

Figure 38. Change in High Temperature True Grade by Addition of: (a) 20, 35 and 50% RAP to the Base Binder PG 58-34; (b) Rejuvenators to the PG 58-34 + 35% RAP Recycled Asphalt Blend, and (c) Rejuvenators to the PG 58-34 + 50% RAP Recycled Asphalt Blend.

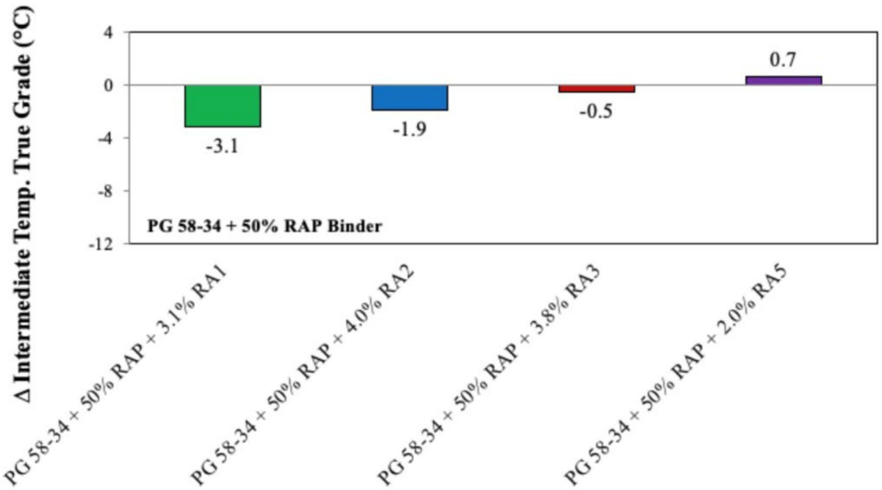
Figure 39 presents the observed change in the continuous intermediate temperature true grade, after addition of the recycled binder and rejuvenators. It is important to remember that the Superpave intermediate temperature performance grade was performed after RTFO plus 60-h PAV aging. As indicated in Figure 39(a), the addition of 20, 35 and 50% recycled binder resulted in an increase of the intermediate temperature true grade by 4.3, 11.3 and 9.9°C, respectively, decreasing the fatigue resistance of the base binder PG 58-34. When adding the rejuvenators to the PG 58-34 + 35% RAP recycled asphalt blend, the decrease in the intermediate pass/fail temperature by RA_1, RA_2, RA_3 and RA_4 was by 2.9, 4.7, 2.7, and 10.0°C, respectively. When adding the rejuvenators to the PG 58-34 + 50% RAP recycled asphalt blend, the decrease in the intermediate pass/fail temperature by RA_1, RA_2, RA_3 and RA_4 was by 3.1, 1.9, 0.5°C, respectively. On the other hand, RA_5 did not improve the fatigue resistance of the PG 58-34 + 50% RAP recycled blend. At the dosage of 2.0%, RA_5 increased by 0.7°C the temperature at which the limiting fatigue parameter [|G*|.sin(δ)] was satisfied based on AASHTO M320.



(a)



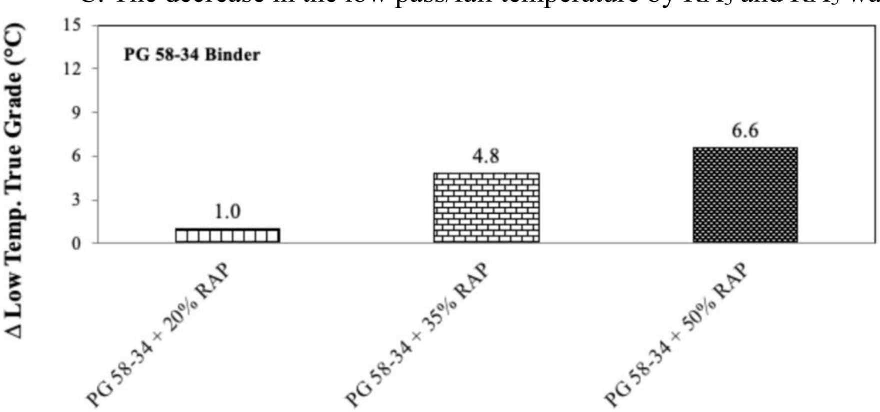
(b)



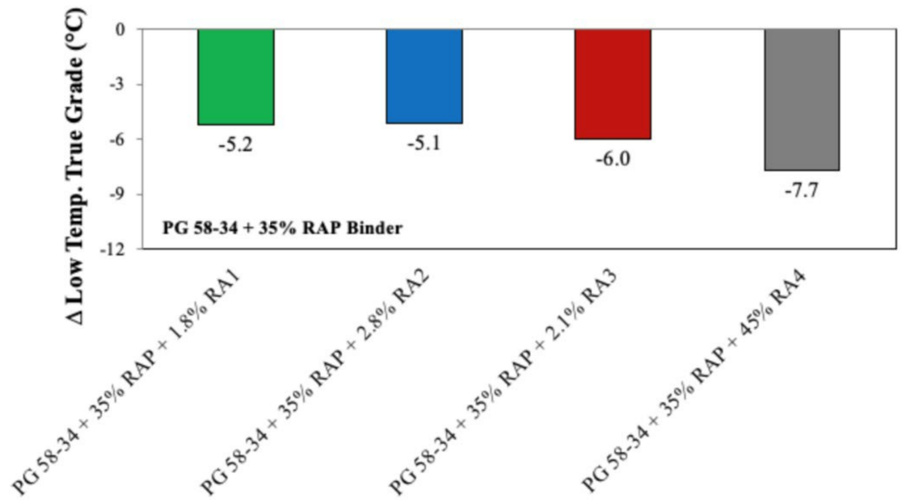
(c)

Figure 39. Change in Intermediate Temperature True Grade by Addition of: (a) 20, 35 and 50% RAP to the Base Binder PG 58-34; (b) Rejuvenators to the PG 58-34 + 35% RAP Recycled Asphalt Blend, and (c) Rejuvenators to the PG 58-34 + 50% RAP Recycled Asphalt Blend.

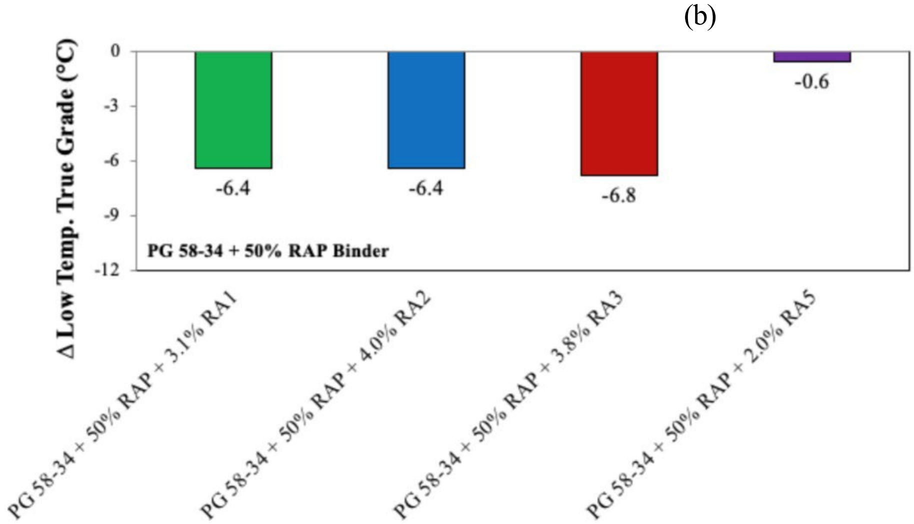
Figure 40 presents the observed change in the continuous low temperature true grade, after addition of the recycled binder and rejuvenators. It is important to remember that the Superpave low temperature performance grade was performed after RTFO plus 60-h PAV aging. As expected, the addition of 20, 35 and 50% recycled binder had a negative effect on the low temperature performance of the PG 58-34 base binder, after RTFO plus 60-hour oxidative aging in PAV. An increase of the low temperature true grade by 1.0, 4.8 and 6.6°C was observed after the addition of 20, 35 and 50% recycled binder, respectively. When the rejuvenators were added to the PG 58-34 + 35% RAP recycled asphalt blend, a decrease in the low pass/fail temperature was observed: RA_1 and RA_2 performed similarly decreasing the low temperature true grade by 5.2 and 5.1°C, respectively; RA_3 and RA_5 decreased the low temperature true grade by 6.0 and 7.7°C, respectively. When adding the rejuvenators to the PG 58-34 + 50% RAP recycled asphalt blend, it was also observed that RA_1 and RA_2 performed similarly decreasing the low temperature true grade by 6.4 °C. The decrease in the low pass/fail temperature by RA_3 and RA_5 was by 6.8 and 0.6°C, respectively.



(a)



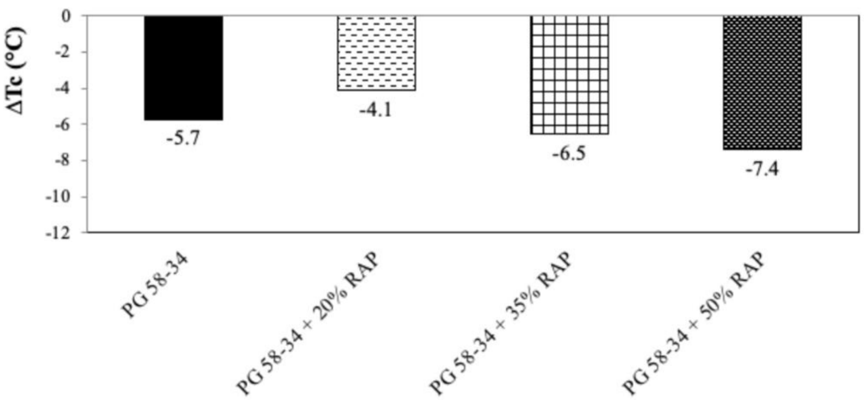
(b)



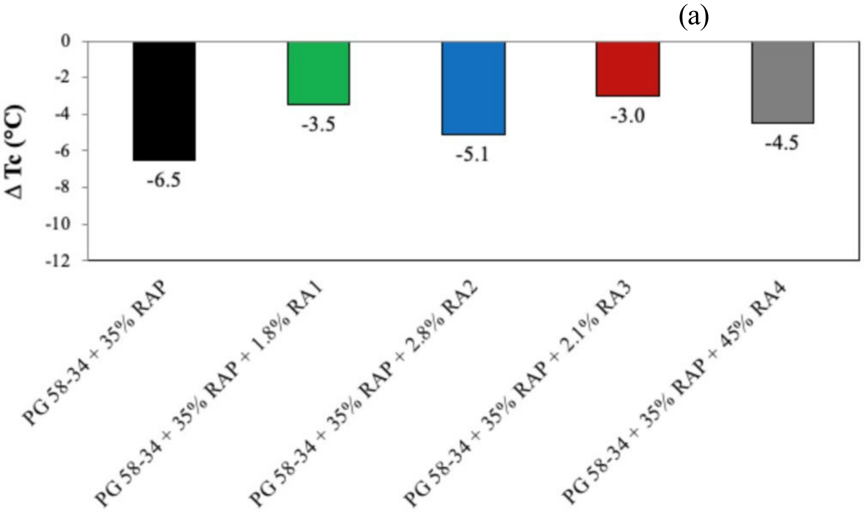
(c)

Figure 40. Change in Low Temperature True Grade by Addition of: (a) 20, 35 and 50% RAP to the Base Binder PG 58-34; (b) Rejuvenators to the PG 58-34 + 35% RAP Recycled Asphalt Blend, and (c) Rejuvenators to the PG 58-34 + 50% RAP Recycled Asphalt Blend.

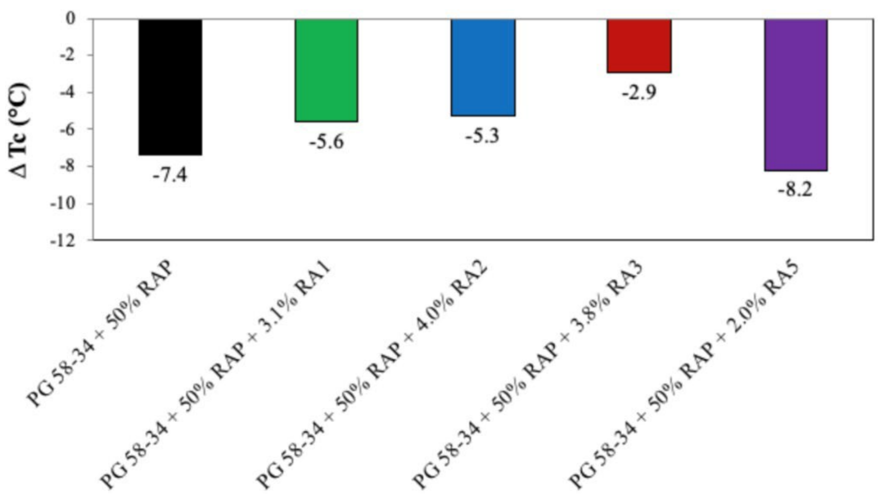
Lastly, the effect of the addition of the recycled binder and rejuvenators was evaluated in terms of the ΔT_c parameter, after RTFO plus 60-hour PAV aging. The ΔT_c parameter consists of the evaluation of the non-load associated cracking potential of asphalt binders, and is calculated using the BBR Stiffness (S) and the BBR m-slope (m-value) ($\Delta T_c = T_{c,S} - T_{c,m}$). It has been suggested that asphalt binders with low (i.e., more negative) ΔT_c have less ductility and reduced relaxation properties than asphalt binders with higher (less negative or positive) ΔT_c. A minimum ΔT_c threshold of -5°C after 40 hrs of PAV aging has been suggested to minimize the risk of age-related block cracking (Anderson et al., 2011). Figure 41(a) indicates that the addition of 20% RAP recycled binder to the base binder PG 58-34 was beneficial, resulting in a less negative ΔT_c (increase of 1.6°C), while the addition of 35% and 50% RAP resulted in a more negative ΔT_c, with a decrease of 0.8 and 1.7°C, respectively. ΔT_c is intended to provide an indication of loss of ductility, indicating when the asphalt binder cannot relax the stresses fast enough to prevent breaking. However, certain features of polymer modification may have a worsening effect on ΔT_c and therefore make it appear as if polymer modified binders exhibit diminished durability, which could be the case of the ΔT_c = -5.7°C for the polymer modified PG 58-34 base binder (Kluttz, 2019). Figure 41(b) indicates that when the rejuvenators were added to the PG 58-34 + 35% RAP recycled asphalt blend, an improvement in the ΔT_c parameter was observed: RA_1, RA_2, RA_3, and RA_4 increased the ΔT_c by 3.0, 1.4, 3.5, and 2.0°C, respectively. When adding the rejuvenators to the PG 58-34 + 50% RAP recycled asphalt blend, it was also observed an improvement in the ΔT_c parameter for RA_1, RA_2, and RA_3, (increase in the ΔT_c by 1.8, 2.1, and 4.5°C, respectively). However, the addition of RA_5 resulted in a slightly more negative ΔT_c, with a decrease of 0.8°C.



(a)



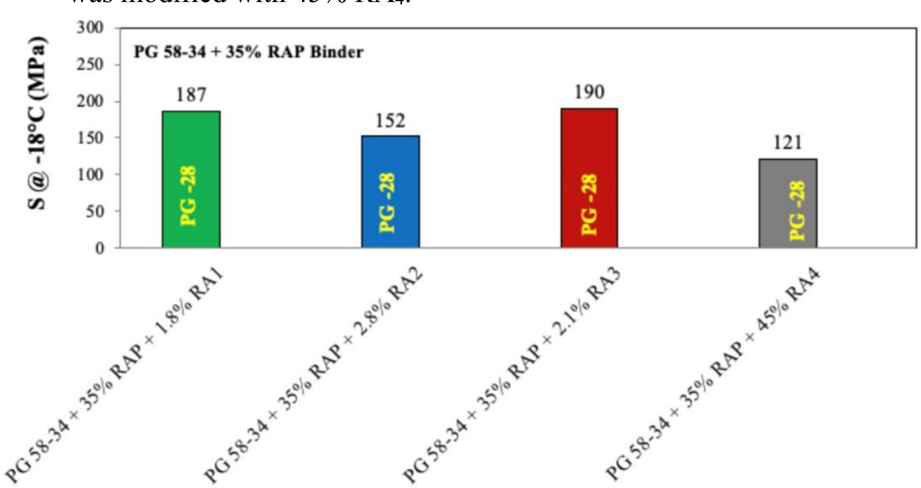
(b)



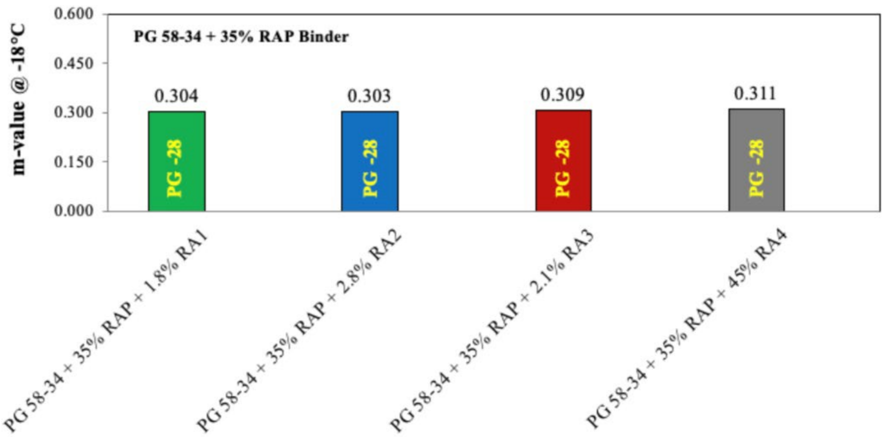
(c)

Figure 41. ΔT_c Parameter of: (a) Base Binder PG 58-34 Neat and Modified with 20, 35 and 50% RAP; (b) PG 58-34 + 35% RAP Recycled Asphalt Blend with and without Rejuvenators, and (c) PG 58-34 + 50% RAP Recycled Asphalt Blend with and without Rejuvenators.

Figure 42 presents the BBR creep stiffness (related to stresses in an asphalt pavement due to thermal contraction) and m-value (related to the ability of an asphalt pavement to relieve these stresses) at same testing temperature (i.e., -18°C) for the PG 58-34 + 35% RAP recycled asphalt blends with rejuvenators. The ranking from lower to higher stiffness was: $RA_4 < RA_2 < RA_1 < RA_3$. However, the ranking from lower to higher m-value was: $RA_2 \approx RA_1 < RA_3 < RA_4$. These results reinforce the aforementioned finding that the petroleum-based rejuvenator RA_4 acted only as a softener to the recycled asphalt blend. When considering resistance to thermal cracking, asphalt binders with lower stiffness and higher m-value usually present good overall performance. In accordance with the results presented in Figure 41, the combination of lowest stiffness and highest m-value was obtained when the PG 58-34 + 35% RAP recycled asphalt blend was modified with 45% RA_4.



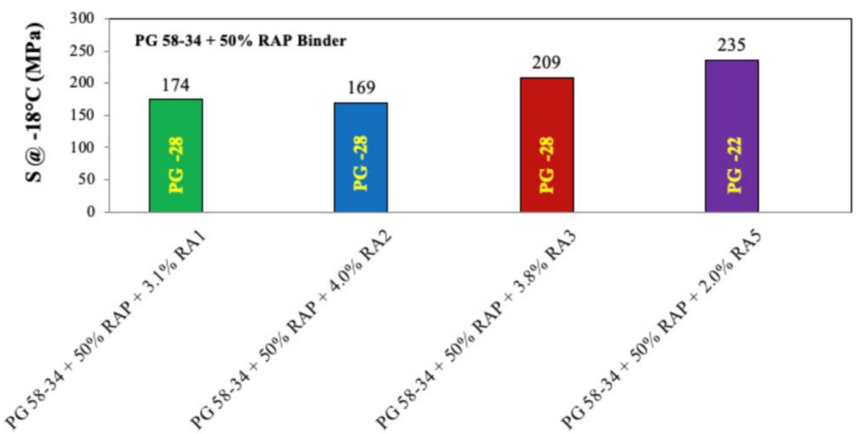
(a)



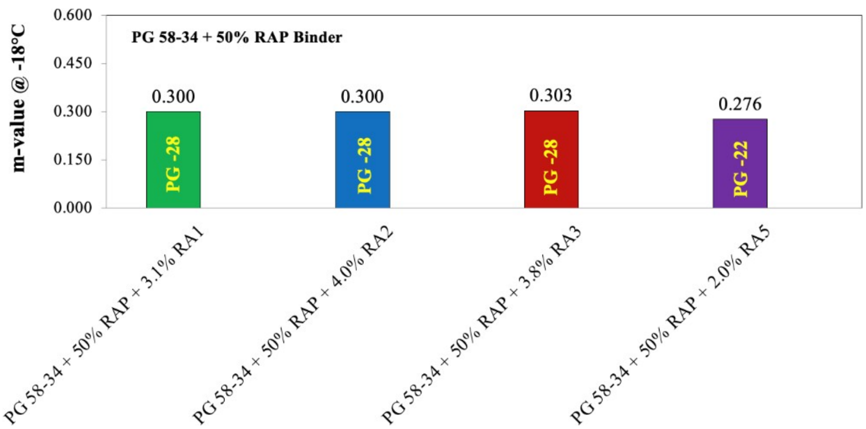
(b)

Figure 42. BBR Data at -18°C for PG 58-34 + 35% RAP Recycled Asphalt Blend with and without Rejuvenators: (a) S, (b) m-value.

Figure 44 shows the analysis of BBR creep stiffness and m-value at -18°C for the PG 58-34 + 50% RAP recycled asphalt blends with rejuvenators. The ranking from lower to higher stiffness was: $RA_2 < RA_1 < RA_3 < RA_4$. However, the ranking from lower to higher m-value was: $RA_5 < RA_1 = RA_2 < RA_3$. An interesting finding was that, regardless of the content of recycled binder (i.e., 35% or 50%), RA_1 and RA_2 behaved similarly in terms of m-value (related to stress relaxation) at the testing temperature of -18°C. Moreover, the combination of lowest stiffness and highest m-value was obtained when the PG 58-34 + 50% RAP recycled asphalt blend was modified with 4.0% RA_2.



(a)

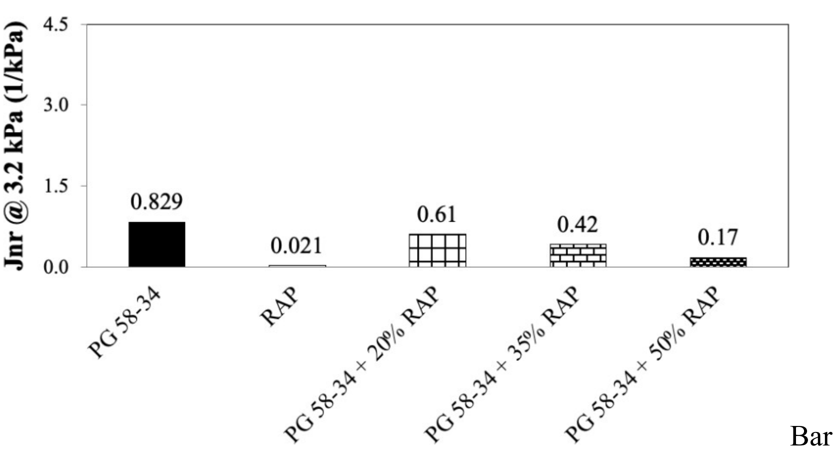


(b)

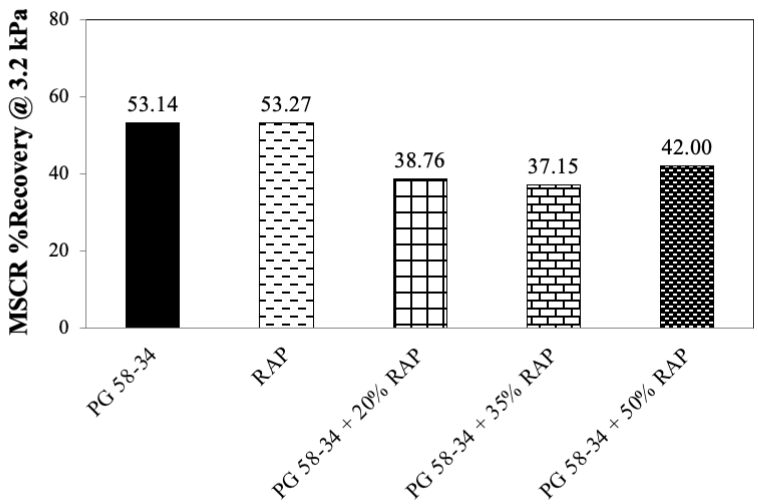
Figure 43. BBR Data at -18°C for PG 58-34 + 50% RAP Recycled Asphalt Blend with and without Rejuvenators: (a) S, (b) m-value.

6.1.3 High Temperature Performance in Terms of the Multiple Stress Creep and Recovery Test

MSCR testing was performed on all samples after RTFO oxidative aging. Although SDDOT does not specify testing temperature, 58°C was selected to better differentiate the rutting resistance of the evaluated materials based on climate. Figure 44 presents the MSCR testing parameters J_{nr} at 3.2 kPa and %Recovery after the addition of 20, 35 and 50% recycled binder to the base binder PG 58-34. Figure 44(a) indicates the Very Heavy (V) classification for PG 58-34, and PG 58-34 + 20% RAP recycled asphalt blend; while the RAP and both the PG 58-34 + 35% RAP and PG 58-34 + 50% RAP recycled asphalt blends were classified as Extreme (E). Moreover, it can be seen that as the percentage of recycled binder increased (i.e., from 20% towards 50%), the creep compliance of the recycled binder blends decreased, as the blend became stiffer. Figure 45(b) shows that the behavior of the %Recovery parameter was found to be highly influenced by the creep compliance J_{nr} of the binders, regardless of the presence and content of polymer. For example, the %Recovery of the RAP recycled binder (i.e., 49.28%) was similar to the PG 58-34 base binder (i.e., 53.14%), when it is known that this base binder was polymer modified. Thus, the %Recovery of the RAS recycled binder is due to the extremely low J_{nr} of the material (i.e., 0.021 kPa^{-1}).



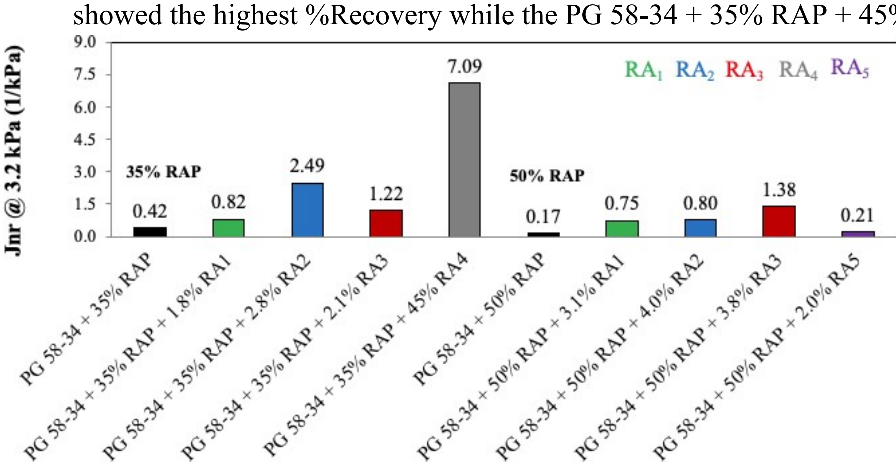
(a)



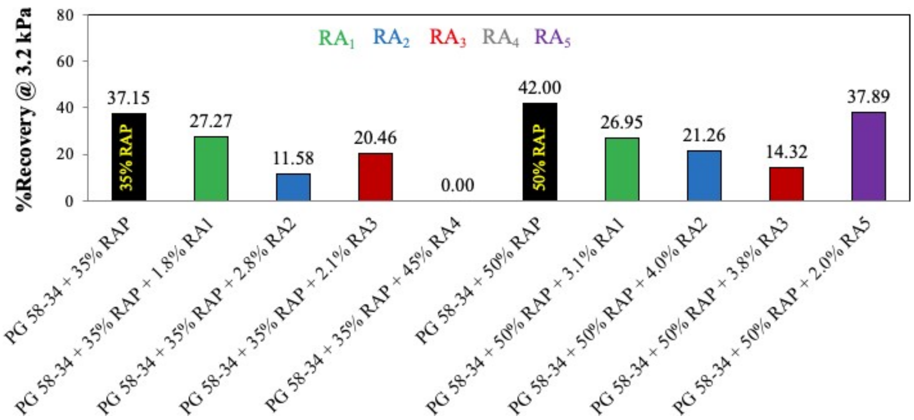
(b)

Figure 44. Effect of Binder Replacement on: (a) J_{nr} at 3.2 kPa and 58°C, and (b) %Recovery at 3.2 kPa and 58°C.

Figure 45 presents the MSCR testing parameters J_{nr} at 3.2 kPa and %Recovery after the addition of rejuvenators to the recycled binder blends. Figure 45(a) indicates that the rejuvenators seem to have an effect on increasing the overall magnitude of J_{nr}; and the effectiveness of each rejuvenator seemed to be related to both additive chemical composition and dosage, and influenced by the content of the recycled asphalt material. The very high J_{nr} observed for the PG 58-34 + 35% RAP + 45% RA_4 was due to the large dosage (i.e., 45% by weight of binder) of the petroleum-based (asphalt flux) rejuvenator, which acted as a softener. As expected, Figure 45(b) indicates that the addition of rejuvenators decreased the %Recovery of the recycled binder blends. Among the blends with rejuvenators, the PG 58-34 + 50% RAP + 2% RA_5 blend showed the highest %Recovery while the PG 58-34 + 35% RAP + 45% RA_4 showed no recovery.



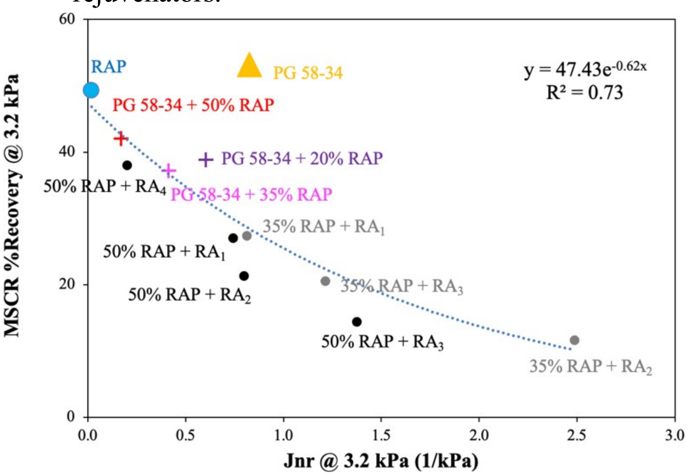
(a)



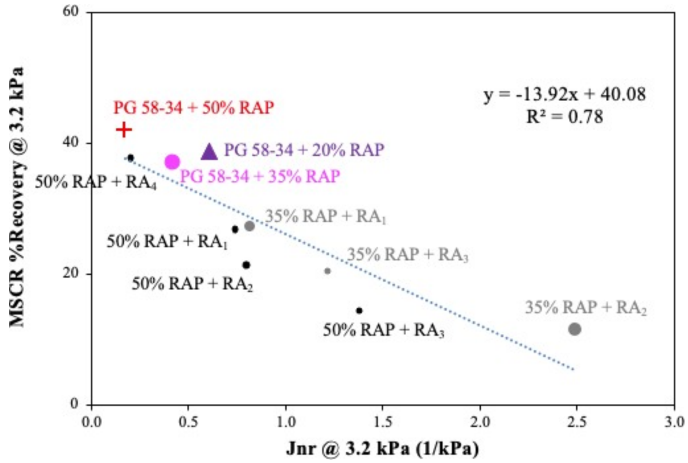
(b)

Figure 45. Effect of Binder Replacement and Rejuvenators on: (a) J_{nr} at 3.2 kPa and 58°C, and (b) %Recovery at 3.2 kPa and 58°C.

Figure 46(a) illustrates the aforementioned relationship between J_{nr} and %Recovery, where %Recovery for a given binder blend is associated to the J_{nr} of the binder. Moreover, it can be seen that base binder type, recycled binder type and percentage, and RA type and dosage played a role in the overall MSCR results of the binders, which agreed with the findings of Bahia et al. (2018). Figure 46(b) indicates the strong correlation existing between the MSRC J_{nr} and %Recovery parameters for the recycled binder blends with rejuvenators.



(a)



(b)

Figure 46. J_{nr} *versus* %Recovery at 3.2 kPa and 58°C for: (a) All the Evaluated Asphalt Binders, and (b) Recycled Binder Blends with Rejuvenators.

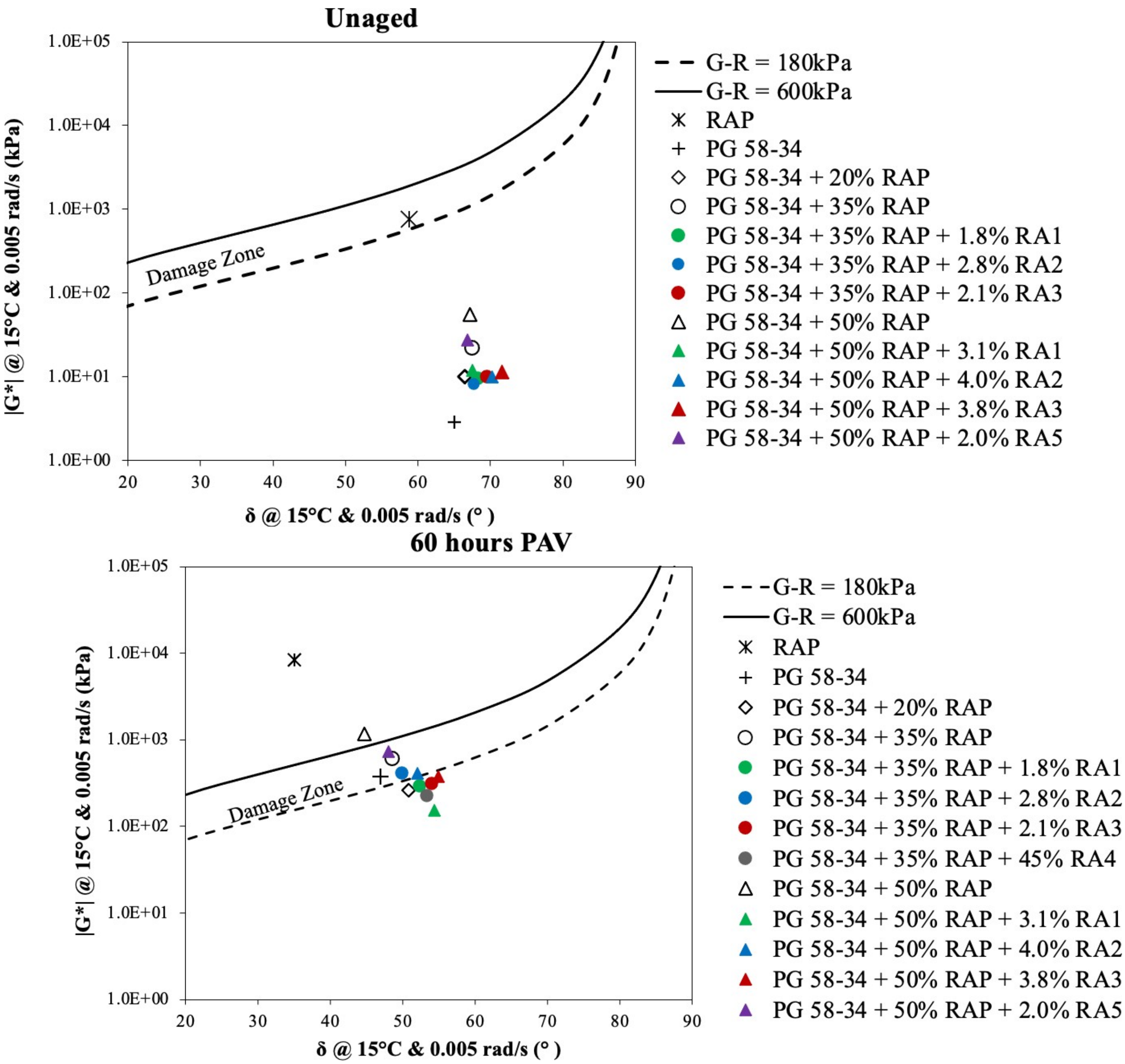
6.1.4 Glover-Rowe Parameter and Black Space Diagram

The Glover-Rowe (*G-R*) parameter considers both binder stiffness and embrittlement and offers an indication of the cracking potential at intermediate temperature (Rowe, 2011). The |*G**| and *δ* at 15°C and 0.005 rad/s as well as the *G-R* parameter results of the base binder PG 58-34 and recycled asphalt binders' blends with and without rejuvenators, at unaged and after RTFO plus 60-hour of PAV aging conditions, are presented in Table 21. With exception of the PG 58-34 + 20% RAP blend, the blends of recycled asphalt binders consistently showed higher *G-R* parameters than the base binder PG 58-34 after long-term aging. These results highlighted the binder stiffening effect due to the use of recycled asphalt materials. Moreover, after RTFO plus 60-hour PAV aging, the following samples exceeded the preliminary *G-R* parameter criterion of 180 kPa for the onset of block cracking: PG 58-34, PG 58-34 + 35% RAP, PG 58-34 + 50% RAP, PG 58-34 + 35% RAP + 2.8% RA_2, PG 58-34 + 50% RAP + 4.0% RA_2, PG 58-34 + 50% RAP + 2.0% RA_5. The possibility of these binder blends having block cracking in the field should be considered. However, debate exists in the validity of using the *G-R* thresholds for evaluating polymer modified binders.

Table 21. |*G| and *δ* at 15°C and 0.005 rad/s, and *G-R* Parameter Results, Before and After Aging.**

Sample	15°C, 0.005 rad/s (unaged)			15°C, 0.005 rad/s (RTFO plus 60-hour of PAV aging)		
	|G*| (kPa)	δ (°)	G-R (kPa)	|G*| (kPa)	δ (°)	G-R (kPa)
PG 58-34	2.9	65.0	0.6	377.2	47.0	240.1
RAP	759.9	58.7	239.4	8462.7	35.0	9889.6
PG 58-34 + 20% RAP	10.1	66.4	1.8	263.3	50.8	135.9
PG 58-34 + 35% RAP	21.8	67.4	3.5	602.2	48.6	350.2
PG 58-34 + 50% RAP	55.5	67.1	9.1	1172.5	44.7	840.5
PG 58-34 + 35% RAP + 1.8% RA_1	9.6	68.3	1.4	292.0	52.4	137.0
PG 58-34 + 35% RAP + 2.8% RA_2	8.1	67.7	1.3	409.5	50.0	221.3
PG 58-34 + 35% RAP + 2.1% RA_3	9.9	69.5	1.3	308.1	54.1	131.0
PG 58-34 + 35% RAP + 45% RA_4	Sample was too soft for testing			227.6	53.4	100.7
PG 58-34 + 50% RAP + 3.1% RA_1	12.0	67.5	1.9	154.5	54.4	64.4
PG 58-34 + 50% RAP + 4.0% RA_2	10.0	70.2	1.2	407.7	52.1	195.1
PG 58-34 + 50% RAP + 3.8% RA_3	11.5	71.5	1.2	373.6	54.9	150.7
PG 58-34 + 50% RAP + 2.0% RA_5	27.7	66.8	4.7	736.5	48.0	443.9

Figure 47 presents the *G-R* parameter results on a Black Space diagram for the asphalt samples, where the binder |*G**| at 15°C and 0.005 rad/s is plotted on the y-axis *versus* δ at the same condition on the x-axis at both unaged and RTFO plus 60-hour PAV aged conditions. The bold and dashed curves in the figure represent the two preliminary *G-R* parameter criteria of 180 kPa and 600 kPa for the onset of block cracking and visible surface cracking, respectively. As shown in Figure 47(a), before aging, the base binder PG 58-34 and all the recycled binder blends were located below the *G-R* 180 kPa limit, with exception of the RAP binder. As aging increased for each binder, the |*G**| and δ data migrated from the lower right corner [i.e., low stiffness (|*G**|) and high ductility (δ)] to the upper left corner [i.e., increased stiffness (|*G**|) and increased brittleness (δ)] of the Black Space diagram [Figure 47(b)]. It can be observed that the addition of 50% RAP binder replacement exceeded the preliminary *G-R* parameter criterion of 600 kPa for the significant block cracking of the resultant binder blend after long-term aging. Furthermore, after RTFO plus 60-h PAV aging, the following asphalt samples were located within the "cracking damage zone" on the Black Space diagram: PG 58-34, PG 58-34 + 35% RAP, PG 58-34 + 35% RAP + 2.8% RA_2, PG 58-34 + 50% RAP + 4.0% RA_2, PG 58-34 + 50% RAP + 2.0% RA_5. Moreover, the following blends did not reach the damage zone after long-term aging: PG 58-34 + 20% RAP, PG 58-34 + 35% RAP + 1.8% RA_1, PG 58-34 + 35% RAP + 2.1% RA_3, PG 58-34 + 35% RAP + 45% RA_4, PG 58-34 + 50% RAP + 3.1% RA_1, and PG 58-34 + 50% RAP + 3.8% RA_3. When investigating the potential binder rejuvenation of the rejuvenators, it was observed that the addition of the five RAs to the recycled binder blends decreased the stiffness of all blends, before and after PAV aging. Moreover, for the aged blends, it can be seen that this decrease in stiffness was followed by an increase in the phase angle (δ). When comparing the performance of the recycled binder blends with rejuvenators after long-term aging, it was observed that the recycled asphalt blends with the rejuvenators RA_2 and RA_5 were located within the "cracking damage zone" on the Black Space diagram. For the RA_2, this behavior occurred regardless of the content of recycled binder (i.e., 35 or 50% RAP). All recycled binder blends rejuvenated with RA_1, RA_3 and RA_4 were located below the damage zone and thus, are not likely to experience premature block cracking in the field.



(a)

(b)

Figure 47. |*G| and *δ* results on a Black Space diagram of: (a) Unaged, and (b) RTFO plus 60-hour PAV Aged.**

An aging index in terms of *G-R* was calculated to evaluate the effect of the rejuvenators on the aging behavior of the recycled binder blends. For each binder, the *G-R* Aging Index was calculated as the fraction of the *G-R* parameter of the RTFO plus 60-h PAV-aged sample over that of the unaged sample. As shown in Figure 48, the addition of the rejuvenators RA_2, RA_3, and RA_5 increased the *G-R* aging index of all the recycled binder blends, indicating increased susceptibility to oxidative aging in terms of binder stiffness and embrittlement. This behavior was much pronounced for the recycled binder blends containing RA_2. On the other hand, the bio-based rejuvenator RA_1 decreased the aging susceptibility of the recycled binder blends, regardless of the content of recycled binder (i.e., 35 or 50% RAP). It is important to mention that the difference observed between the rotational viscosity aging index and the *G-R* aging index could be related to the fact that the first relates to flow properties while the second relates to dynamic material properties (such as complex modulus and phase angle obtained from an oscillation test).

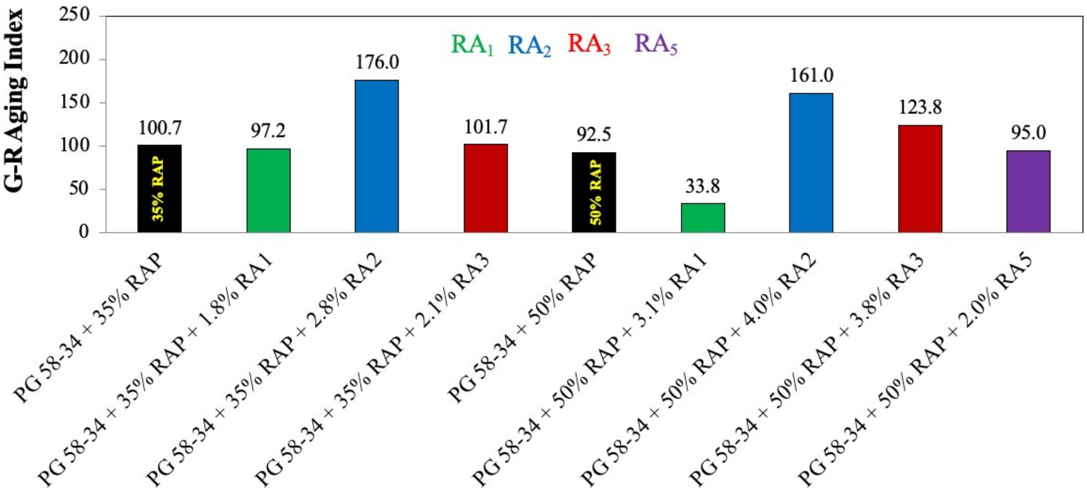
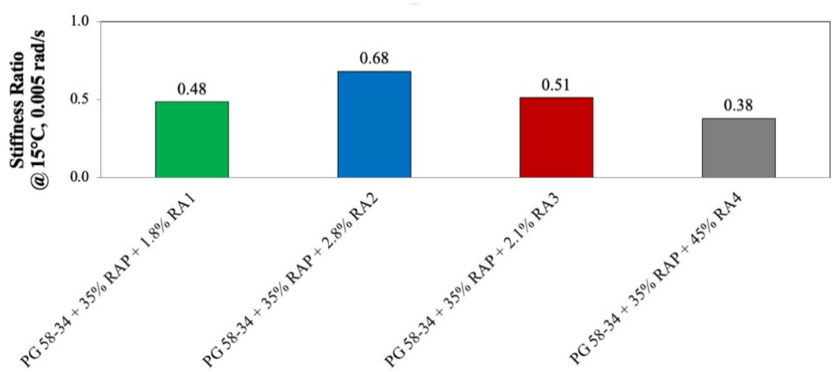


Figure 48. *G-R* Aging Index Results.

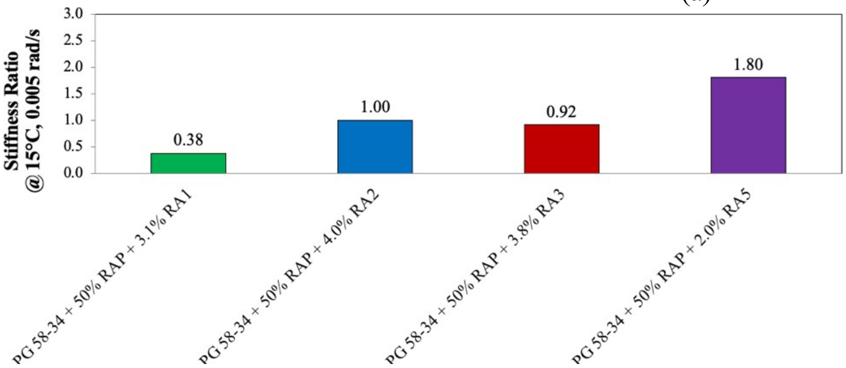
Even though some recycled binder blend with rejuvenator showed higher binder stiffness and embrittlement rate than the control recycled binder, to address the longevity of the effectiveness of the rejuvenator products in decreasing the stiffness of the control recycled binder blends after long-term aging, a stiffness ratio was determined per Equation 6 and the results are presented in Figure 49.

$$\text{Stiffness Ratio @ 15°C, 0.005 rad/s} = \frac{RRRRVVVVVVRRRRRR\ BBVVBBRRRRBB\ BBRRRRBBRR\ ww\text{VVVV}h\ RRRRRRRRRRRRBBRRVVVVBB\ (|GG^*|)_{RRRRRRR}\ pppppppp\ 60\text{–}h\ PPAAPP}{CCVVBBVVBBVVRR\ RRRRVVVVVVRRRRRR\ BBVVBBRRRRBB\ BBRRRRBBRR\ (|GG^*|)_{RRRRRRR}\ pppppppp\ 60\text{–}h\ PPAAPP} \quad \text{Equation 6}$$

As indicated in Figure 49(a), all four rejuvenator products showed stiffness ratio below 1.0 after RTFO plus 60-h PAV, indicating that the stiffness of the rejuvenated recycled binder blends contained 35% RAP remained softer than the control recycled binder, measured after long-term aging. The ranking from lower to higher stiffness was: $RA_4 < RA_1 < RA_3 < RA_2$. Figure 49(b) indicates that, when considering the PG 58-34 + 50% RAP recycled binder blend, the rejuvenator products that presented stiffness ratio below 1.0 after RTFO plus 60-h PAV were RA_1 (stiffness ratio = 0.38) and RA_3 (stiffness ratio = 0.92), indicating that the stiffness of the rejuvenated recycled binders remained below the stiffness of the control. When comparing among the rejuvenators, the ranking from lower to higher stiffness was: $RA_1 < RA_3 < RA_2 < RA_5$.



(a)



(b)

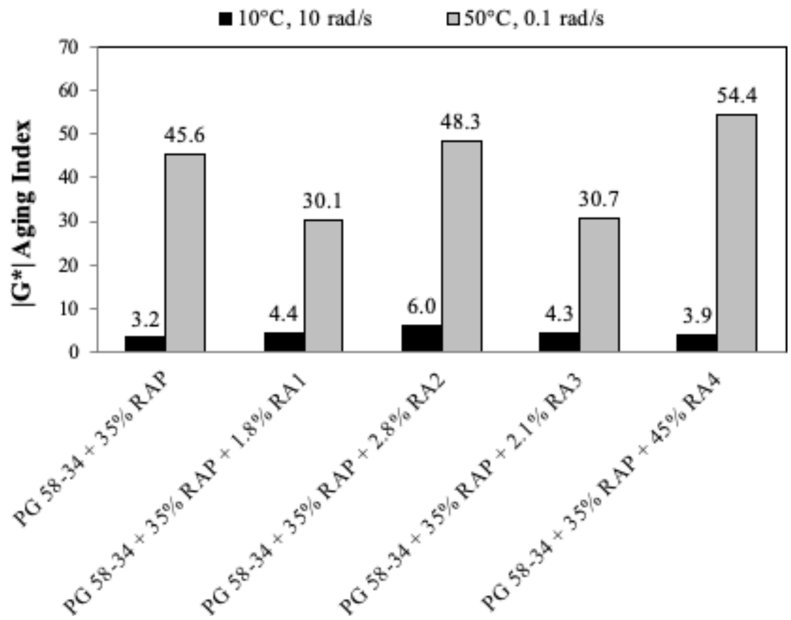
Figure 49. Stiffness Ratio for: (a) Rejuvenated PG 58-34 + 35% RAP Recycled Binder Blends, and (b) Rejuvenated PG 58-34 + 50% RAP Recycled Binder Blends.

6.1.5 Complex Modulus |G| Aging Indexes*

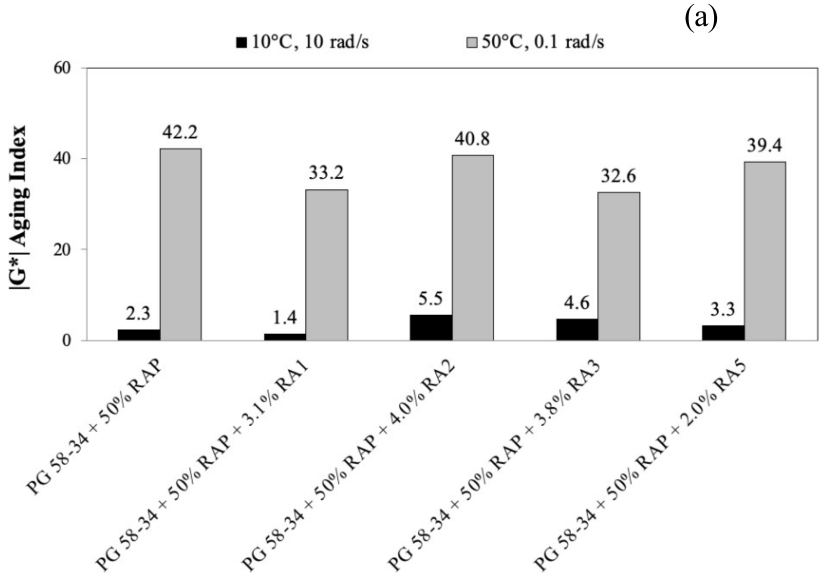
In order to investigate the relationship between oxidative aging and the potential binder rejuvenation of the rejuvenators, a complex modulus aging index was calculated using |G*| at 10°C and 10 rad/s (representing lower temperatures or higher traffic speed) and at 50°C and 0.1 rad/s (representing higher temperatures or lower traffic speed). The aging indexes were calculated in accordance with Equation 7.

$$\text{Aging Index } (|GG*|) = \frac{(|G^*|)_{\text{After RTFO plus 60-h PAV}}}{(|G^*|)_{\text{Unaged}}} \qquad \text{Equation 7}$$

As can be seen in Figure 50(a), the presence of the rejuvenators RA_1 and RA_3 decelerated the age hardening of the PG 58-34 + 35% RAP recycled binder blend at 50°C and 0.1 rad/s (representing higher temperatures or lower traffic speed), while RA_2 and RA_4 showed higher aging susceptibility than the control recycled binder blend. The highest aging susceptibility was observed for the petroleum-based rejuvenator RA_4. At 10°C and 10 rad/s (representing lower temperatures or higher traffic speed) none of the rejuvenators seemed to decrease the aging susceptibility of the control recycled binder blend. Figure 50(b) shows that all rejuvenators (i.e., RA_1, RA_2, RA_3, and RA_5) decelerated the age hardening of the PG 58-34 + 50% RAP recycled binder blend at 50°C and 0.1 rad/s. At 10°C and 10 rad/s, only RA_1 seemed effective on the binder rejuvenation in terms of |G*| after long-term aging.



(a)



(b)

Figure 50. |G*| Aging Indexes at 10°C and 10 rad/s and at 50°C and 0.1 rad/s for: (a) PG 58-34 + 35% RAP Recycled Asphalt Blend with and without Rejuvenators, and (b) PG 58-34 + 50% RAP Recycled Asphalt Blend with and without Rejuvenators.

Also, in accordance with Equation 7, another complex modulus aging index was calculated using |G*| at 60°C and 10 rad/s [representing higher temperatures and the shearing action corresponding to a traffic speed of about 55 mph (90 km/h)], and the results are presented in Figure 51. As previously observed, the rejuvenators RA_1 and RA_3 decelerated the age hardening of the PG 58-34 + 35% RAP recycled binder blend, while the rejuvenators RA_2 and RA_4 were unsuccessful against aging through monitoring the complex modulus aging index (ratio of the complex modulus before and after aging). Moreover, RA_4 was found as the rejuvenator most susceptible to aging, confirming that this additive only acted as a softener to the recycled binder blend and did not avoid further aging of the recycled binder. For the PG 58-34 + 50% RAP recycled binder blend, only RA_3 decelerated the age hardening after long-term aging.

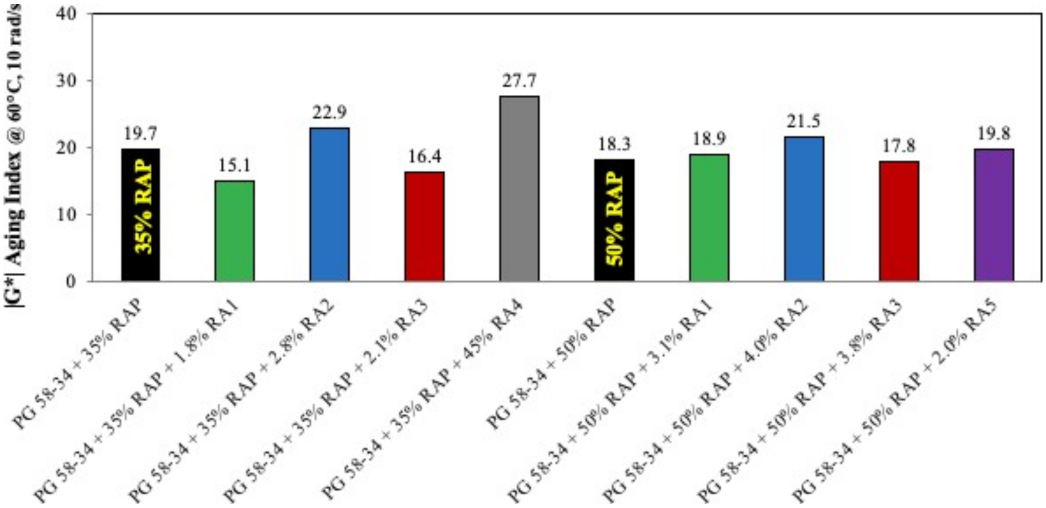


Figure 51. |G*| Aging Index at 60°C and 10 rad/s.

6.1.6 Chemical Evaluation by Means of Fourier Transform Infrared (FTIR) Spectroscopy

Since the aging behavior of recycled blended binders is influenced by the chemical composition of the individual binders, FTIR analysis was performed to investigate the impact of recycled binders and rejuvenators on the chemical composition of the resulting binder blends. The change of chemical structure of asphalt binders can be obtained by the calculation of functional and structural indices of some groups from FTIR spectra, since with oxidative aging the absorbance bands representing oxygen containing functionalities (e.g., carbonyl and sulfoxide groups) of asphalt increases (Milton et al., 1998). In this study, to quantify oxidation-related changes collected by means of infrared absorption, band areas rather than peak absorbance values were used. The absorption spectrum carbonyl area (C=O) was calculated for all samples by integrating the area of the spectrum between the wavelengths of 1660 and 1740 cm^{-1} and using the magnitude of the absorption at 1740 cm^{-1} as the baseline.

Figure 52 shows the absorption spectrum carbonyl and sulfoxide (C=O+S=O) area of the base binder PG 58-34, RAP and recycled binder blends before oxidative aging. As expected, the base binder PG 58-34 showed the lowest aging characteristics (C=O+S=O area = 0.79) while the RAP binder showed the highest (C=O+S=O area = 1.52). Moreover, as the percentage of recycled binder increased (i.e., from 20% towards 50%) in the recycled binder blends, the C=O+S=O area also increased, as the availability of the aged binder increased in the recycled binder blend.

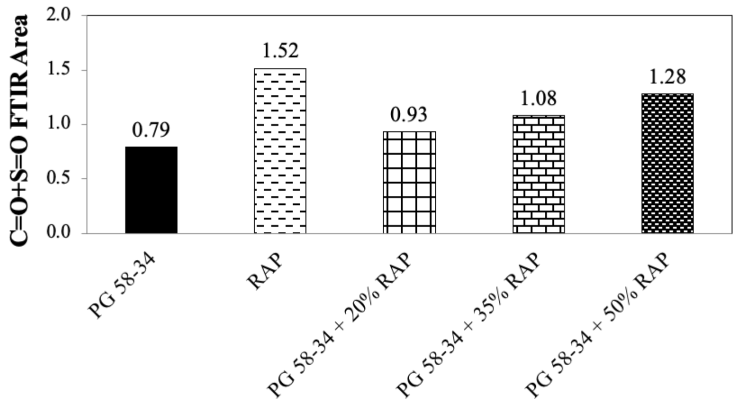
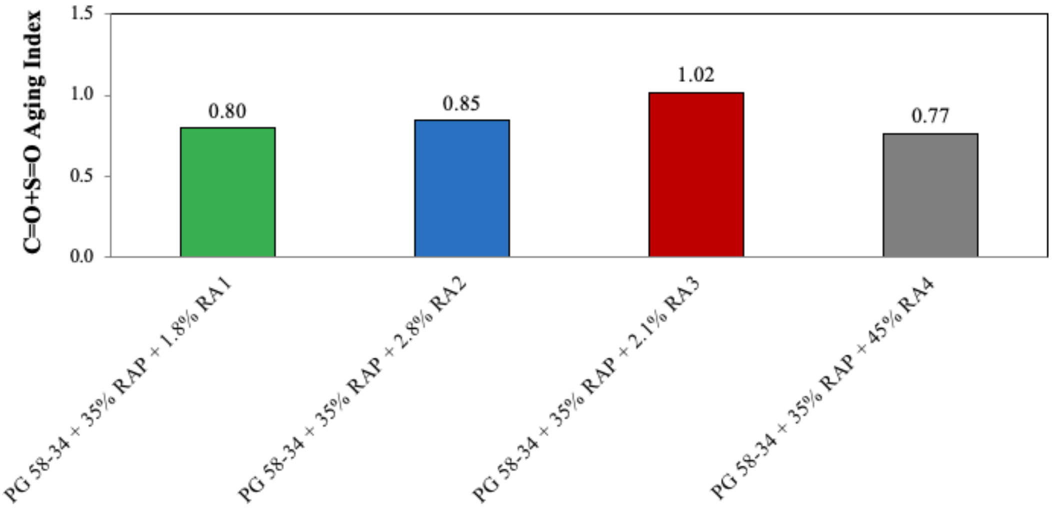


Figure 52. Effect of Binder Replacement on C=O+S=O Area for PG 58-34 Binder.

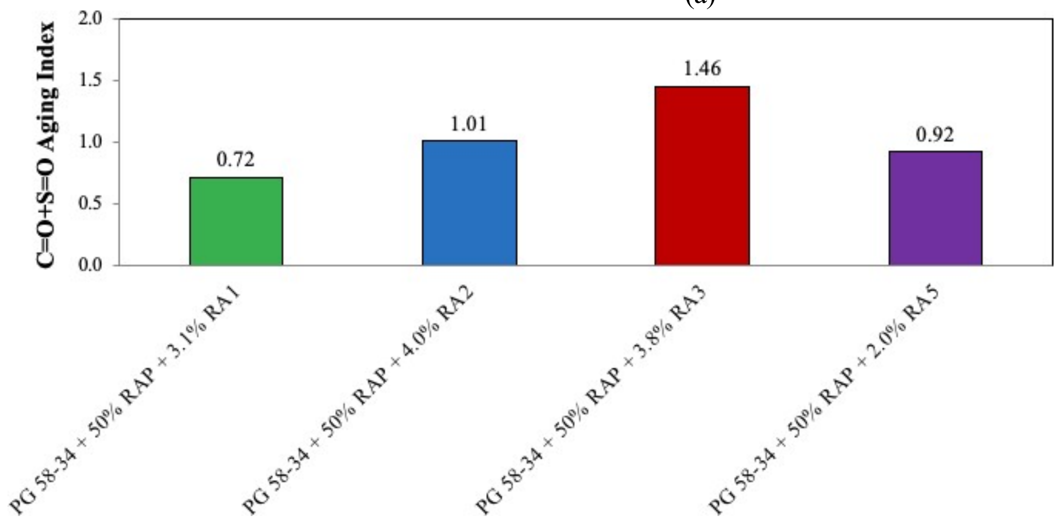
To address the effectiveness of the rejuvenator products in decreasing the growth of oxygen containing functionalities (i.e., carbonyl and sulfoxide groups) of the recycled binder blends PG 58-34 + 35% RAP and PG 58-34 + 50% RAP, an aging index was determined per Equation 8 and the results are presented in Figure 53.

$$CC_{PPAAPP} = 00 + SS = 00\ AAAARRRRAA\ IIRRIIIIII = \frac{\text{RRRRVVVVVVRRRRR R BBVVBBRRRRB B BBRRRRBBR R wwVVVV}h\text{ RRRRRRRRRRRRBBRRVVVVBB}(CC=00+SS=00)_{\text{RRRRRRR R ppp[illegible]ppp p 60-h}}}{\text{CCVVBBVVBBVVRR RRRRVVVVVVRRRRRR BBVVBBRRRRBB BBRRRRBBRR }(CC=00+SS=00)_{\text{RRRRRRRR ppppppp 60-h PPAAPP}}} \quad \text{Equation 8}$$

As indicated in Figure 53(a), the RA_1, RA_2, and RA_4 rejuvenator products showed C=O+S=O aging index below 1.0 after RTFO plus 60-h PAV aging. These results indicated that, with exception of RA_3, the oxidation of the rejuvenated recycled binder blends remained below the oxidation of the PG 58-34 + 35% RAP control recycled binder. The ranking from lower to higher oxidation was: $RA_4 < RA_1 < RA_2 < RA_3$. Figure 53(b) shows that the overall oxidation of the rejuvenated recycled binder blends containing RA_1 and RA_5 remained below the oxidation of the PG 58-34 + 50% RAP control recycled binder measured after the RTFO plus 60-h PAV aging. For the PG 58-34 + 50% RAP + 4.0% RA_2 and PG 58-34 + 50% RAP + 3.8% RA_3 blends, the rejuvenators RA_2 and RA_3 presented higher oxidation in terms of the C=O+S=O area (i.e., aging index higher than 1.0) than the recycled binder blend PG 58-34 + 50% RAP. Therefore, it was observed that the reduction in C=O+S=O area due to the addition of rejuvenators was dependent on the RA type (i.e., composition) and dosage, and recycled binder content, where the imparted reduction of the oxygen-containing compounds varied between the recycled binder blends.



(a)



(b)

Figure 53. C=O+S=O Aging Index for: (a) Rejuvenated PG 58-34 + 35% RAP Recycled Binder Blends, and (b) Rejuvenated PG 58-34 + 50% RAP Recycled Binder Blends.

6.2 Asphalt Mixture Performance Testing Analysis

Before an in-depth statistical analysis of the data set, outliers were removed following a procedure described in ASTM E178. Analysis of variance (ANOVA) and Tukey-Kramer tests were used to assess the statistically significant differences in the mean values of the performance testing results (multiple groups). While ANOVA shows whether the differences in the mean values of the groups are statistically significant, Tukey's HSD indicates the exact groups with mean values as statistically significant (if any). Both tests were conducted with a significance level (α) of 0.05.

6.2.1 Asphalt Pavement Analyzer (APA) Test

Six replicates were prepared per each mix type to get three pairs, one per wheel path. Figure 54 presents the APA rut depth results for both 35% ABR and 50%ABR mixes along with the control mix (20% ABR). As shown in Figure 54, all mixtures both with and without rejuvenators had less rut depth compared to the control mixture (20% ABR), except mixture 35 ABR/RA_4 (mix with the flux). This mix had the highest mean rut depth (3.2 mm) followed by the control mix (2.4 mm); other mixtures' mean rut depth ranged from 2.2 mm to 1.8 mm with mixtures 35% ABR/No RA having the lowest. None of the mixtures exceeded the 7 mm rut depth threshold required by South Dakota DOT for Q2 roadways. Therefore, these high RAP mixes with and without RAs are expected to perform equivalent to the control mix as long as they are subjected to similar conditions. In addition, there was no difference in the rutting performance of the mixes rejuvenated with different rejuvenators, with the exception of the mix with RA_4.

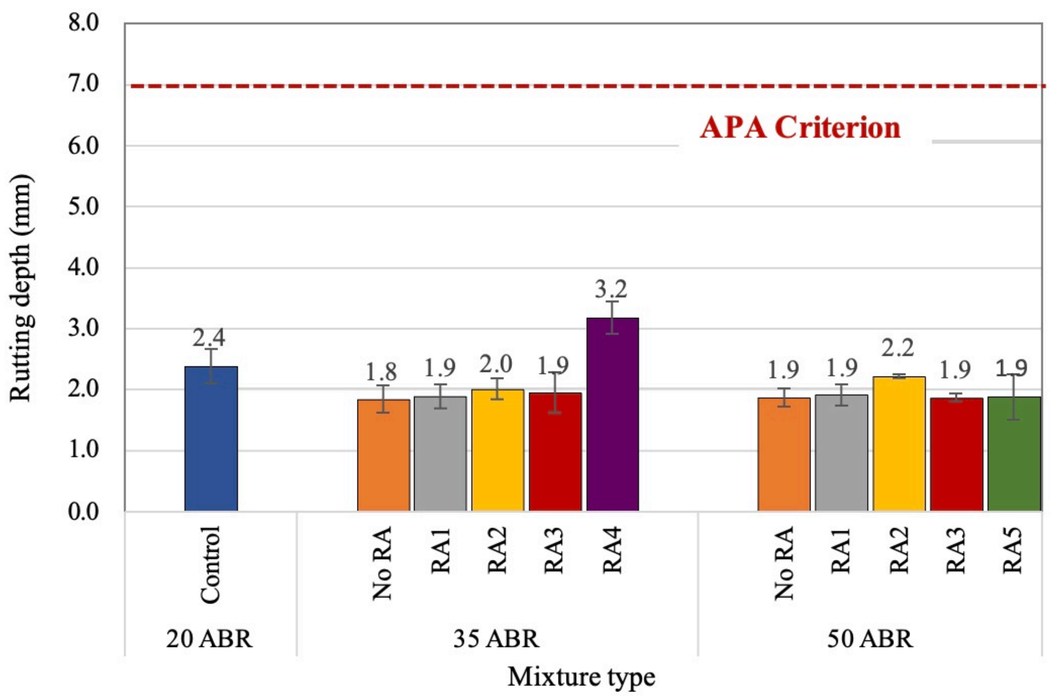


Figure 54. APA Rut Depth Results.

6.2.2 *Indirect Tensile Asphalt Cracking Test (IDEAL-CT)*

Figure 55 and Figure 56 present the average IDEAL-CT load versus displacement curves of the control mix, 35% ABR and 50% ABR mixes with and without rejuvenators after LTOA. From Figure 55 the 35% ABR mix with RA_4 had the lowest peak load and one of the lowest post peak slopes among the mixes. The 20% ABR mix had similar peak load to the peak load of the rejuvenated mixes (35%ABR and 50%ABR) with RA_2. The 35% ABR and 50% ABR mixes without rejuvenators had the highest peak load with steeper post-peak slopes.

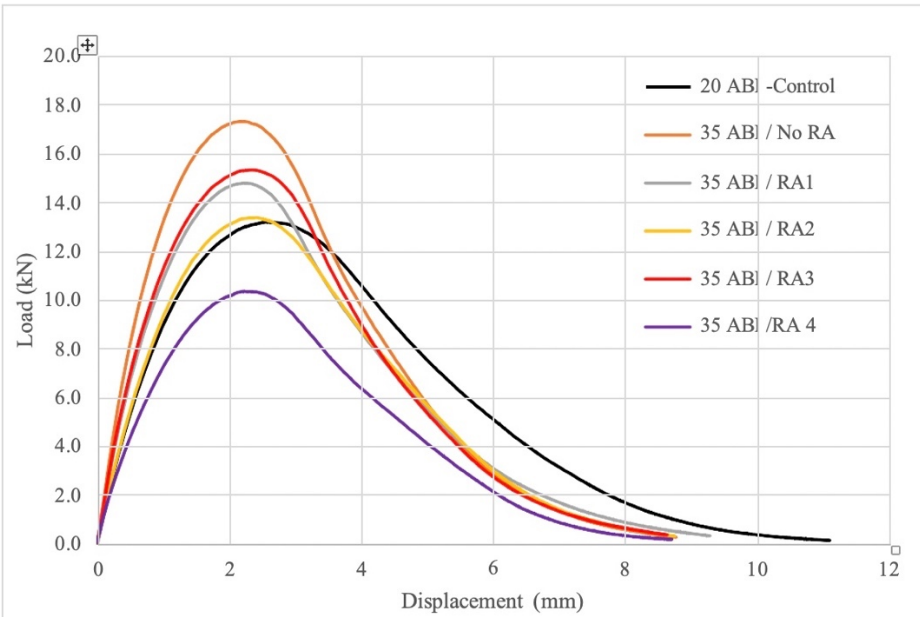


Figure 55. Load-Displacement Curves for the 35 ABR Mixes.

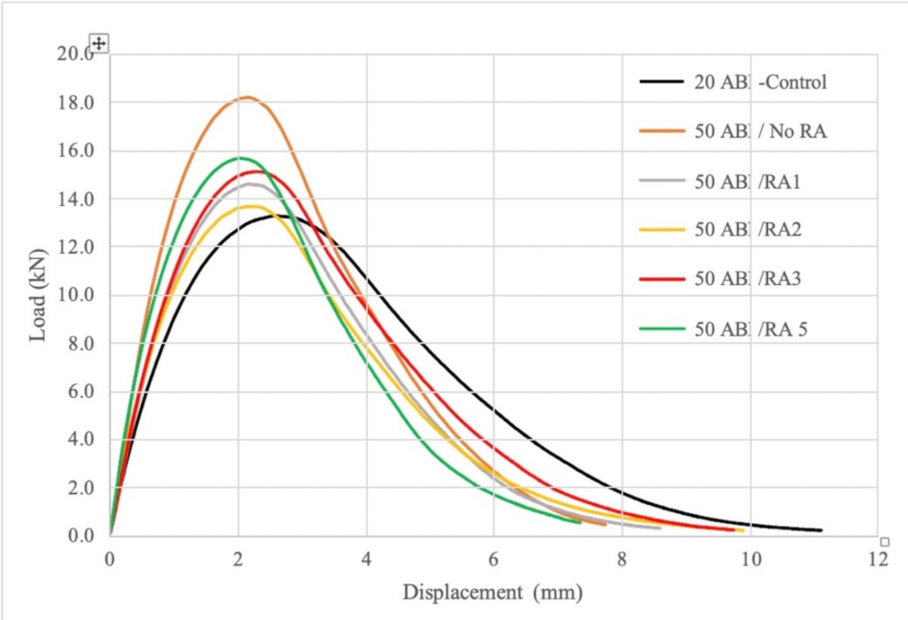


Figure 56. Load-Displacement Curves for the 50 ABR Mixes

The mean CT_{index} values of all the mixes are presented in Figure 57. As shown in this Figure, the 20%ABR control mix had the highest CT_{index} among all mixes. Tukey Kramer showed that the CT_{index} values of the 20% ABR control mix were statistically different than all 35% ABR and 50% ABR mixes, indicating that regardless of whether the mix was rejuvenated or not, their intermediate temperature cracking resistance assessed with CT_{Index} was not better compared to the control mix.

The average CT_{Index} values of non-rejuvenated mixes (35% ABR and 50% ABR) had lower CT_{index} values than the rejuvenated mixes, except for the 50% ABR /RA_5 mixture. The performance of the 50% ABR/RA_5 mixture was unexpected since it was anticipated that all the rejuvenated mixes would yield higher CT_{Index} values than mixes without rejuvenators at the same RAP content. Despite these differences observed in the average CT_{index} values of non-rejuvenated mixes vs. rejuvenated mixes, the statistical analysis showed that significant statistical differences only exist between CT_{index} values of mix 35% ABR/No RA versus 35% ABR/RA_2 , and mix 50% ABR/No RA vs 50% ABR/RA_3. Hence, when considering the variability of the IDEAL-CT test, at the selected dosage rate and RAP contents, mixes with RA_2 and RA_3 are the only mixes expected to have better intermediate temperature cracking resistance than non-rejuvenated mixes.

Since IDEAL-CT is very sensitivity to asphalt content changes (Zhou, et al. 2019) it is important to assess the effective binder content (P_{be}) of the mixes under evaluation. Table 17 showed that P_{be} was higher for the control 20% ABR mix when compared to the 35% ABR and 50% ABR mixes, 5.1% for the 20%ABR control mix compared to 4.6% and 4.3% for the 35%ABR and 50%ABR mixes, respectively. Therefore, the higher CT_{Index} of the control 20%ABR mix when compared to the results of the high RAP mixes with and without rejuvenators may be partially attributed to the higher effective binder content of the control mix.

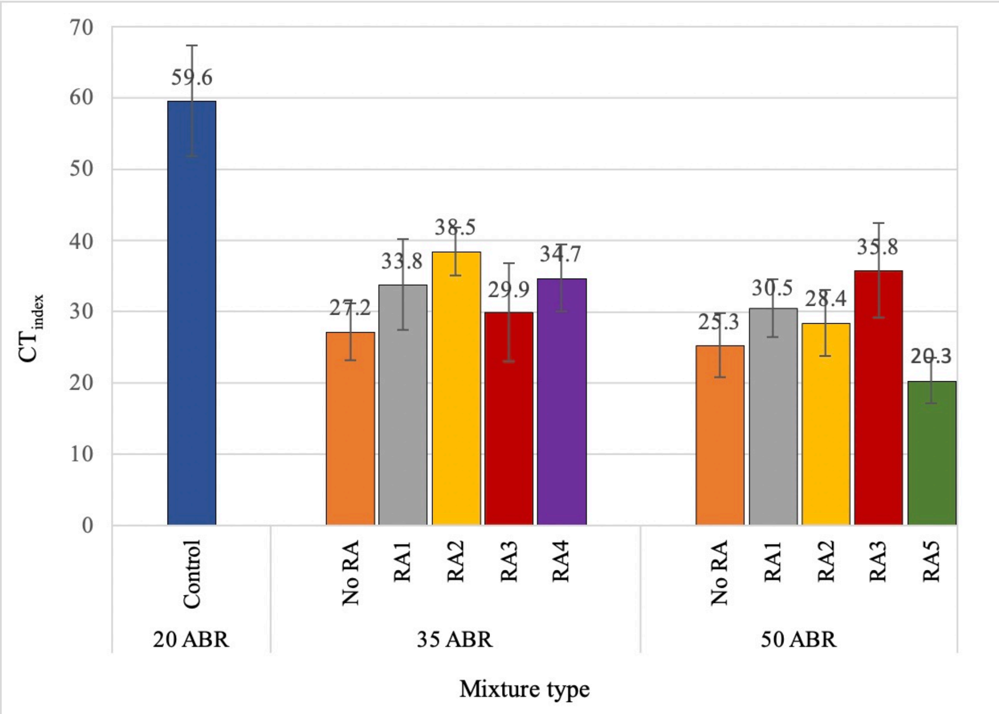


Figure 57. CT$_{index}$ Results.

To further investigate the unexpected results in Figure 57, additional IDEAL-CT tests not included in the original experimental plan were conducted. These additional tests attempted to address three possible reasons for the results obtained with the IDEAL-CT test:

1. The rejuvenator dosages recommended by the suppliers may be too low. The research team considered that selecting a rejuvenator dosage to target the low-temperature PG grade (-34°C) of the virgin binder (after RFTO plus 60-hour PAV aging) would be more appropriate. This approach would yield higher rejuvenator dosages and could potentially produce mixes with better intermediate-temperature cracking performance.
2. The cracking tests used in this study were conducted with a more aggressive long-term aging procedure than the one currently specified in AASHTO R30. The 6-hour 135°C loose mixture aging protocol is expected to better simulate the field aging of asphalt pavements than the current procedure, and it is considered to be crucial for evaluating the long-term cracking resistance of high RAP content mixes with RAs because some of these additives could significantly affect the aging characteristics of the rejuvenated binders and mixtures.
3. When a 100% blending between aged binder and virgin asphalt binder is assumed, rejuvenators alone may not be effective to significantly improve the cracking resistance of high RAP content mixes. A combination of adding rejuvenators and additional binder content may be a more effective approach. The additional binder to be added can be determined by estimating a RAP binder availability factor (BAF), which is defined as the percentage of recycled asphalt binder in RAP that contributes to a given performance property. States like Georgia and Illinois have implemented this approach. For the purpose of this evaluation, an assumed BAF of 75% for RAP binder was used.

Figure 58 summarizes the results of IDEAL-CT tests conducted for the 35% ABR mix with no RA and with RA_1 at two different dosages; the first dosage (1.8%) corresponds to the dosage recommended by the supplier (IDEAL-CT results presented previously), and the second dosage (3.6%) was determined by targeting a LT-PG of -34°C. The statistical analysis indicated that a significant difference exists between the CT_{Index} results of the 35%ABR mix with no RA versus with 3.6% RA_1. This indicates that at the higher dosage, RA_1 was effective at improving the intermediate temperature cracking resistance of the 35% ABR mix. The difference in CT_{Index} values between the mixes with 1.8% and 3.6% RA_1 was not statistically significant.

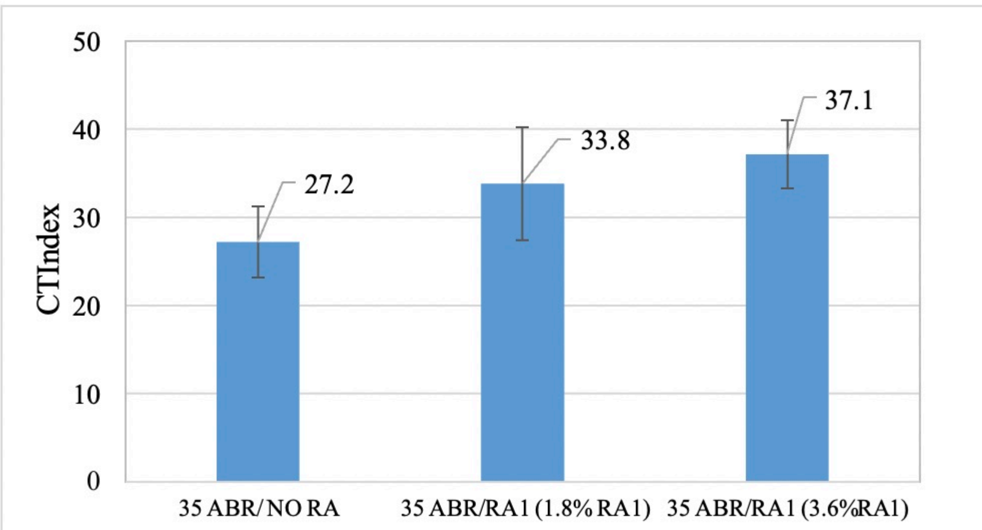


Figure 58. IDEAL-CT Results for 35%ABR Mixes with and without RA1 at Different Dosages.

Figure 59 summarizes the IDEAL-CT test results conducted on samples after Short-Term Oven Aging (STOA) (loose mix aging for 4 hours at 135°C) for the 35% ABR mix with no RA and with RA_1 at the dosage recommended by the supplier. The IDEAL-CT results obtained after LTOA reported previously in Figure 57 are included again in this figure for comparison purposes. The statistical analysis shown that a significant difference exists between their CT_{Index} results. These results suggest that the effectiveness of the rejuvenators may be diminished with the long-term aging of the mix.

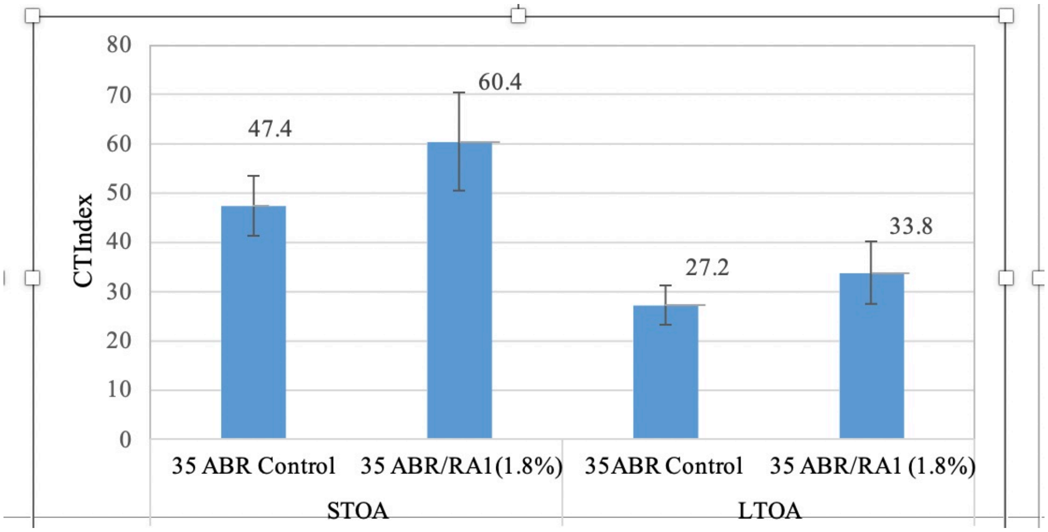


Figure 59. IDEAL-CT Results after STOA and LTOA for 35%ABR Mixes with and without RA_1.

Finally, IDEAL-CT Finally, IDEAL-CT tests were conducted after LTOA for the 35% ABR mix with RA_1 at the dosage recommended by the supplier assuming a 75% BAF. These results were compared to the 35%ABR mix with no rejuvenator as presented in Figure 60. The statistical analysis indicated that there is a significant difference in the CT_{Index} results. These results suggest that when a 75%BAF was used, RA_1 was effective at improving the performance of the 35% ABR mix at intermediate temperature.

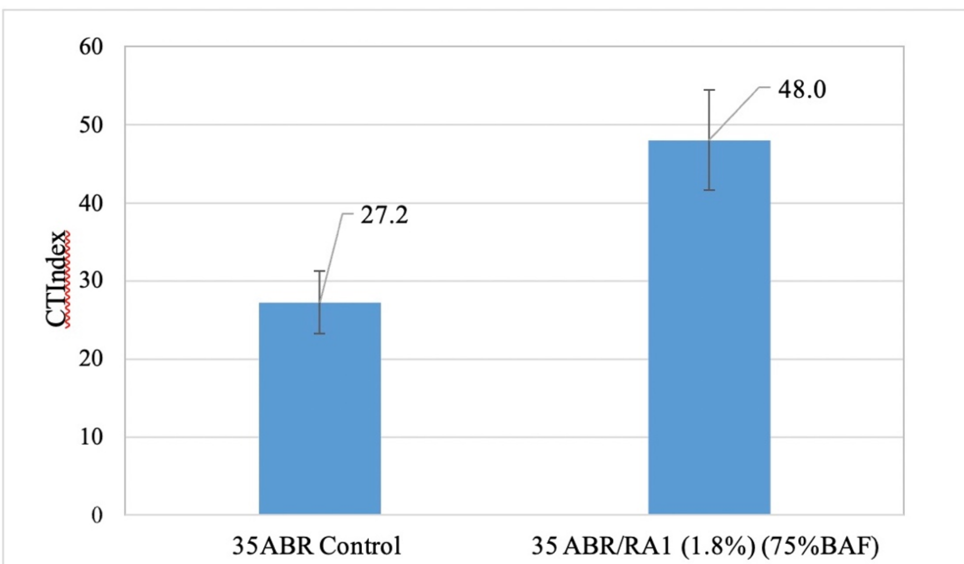


Figure 60. IDEAL-CT Results after LTOA for 35%ABR Control Mix and 35% ABR Mix at the Recommended RA Dosage assuming a 75% BAF.

6.2.3 Disc-shaped Compact Tension (DCT) Test

The DCT fracture energy values of the mixes after LTOA are shown in Figure 61. A higher fracture energy value is desired for a mix to have a better resistance to thermal cracking. As shown in this figure, for the 35%ABR mixes, all the mixes except the non-rejuvenated mix and the mix with RA_3 had higher mean fracture energy values compared to the 20%ABR mix. The non-rejuvenated mix had the lowest mean fracture energy. For the 50% ABR mixes, all the mixes had lower mean fracture energy values compared to the 20%ABR mix. In addition, except for 50% ABR/RA_3 mix, the mean fracture energy values of all rejuvenated mixes were higher compared to the fracture energy of the corresponding non-rejuvenated mix.

While ANOVA showed that the fracture energy values of the 20% ABR mixes and 35% ABR mixes are statistically different, the 50% ABR mixes were not. For the 35% ABR mixes, the Tukey-Kramer test showed that a statistical difference exists between the non-rejuvenated mix and mixes with RA_2 and RA_4. Therefore, at the selected dosage rate and RAP content, mixes with RA_2 and RA_4 are expected to have better thermal cracking resistance than the non-rejuvenated mix, but comparable performance to that of the control mix. For the 50% ABR mixes, all mixes (rejuvenated, and non-rejuvenated) are expected to have an equivalent resistance against thermal cracking.

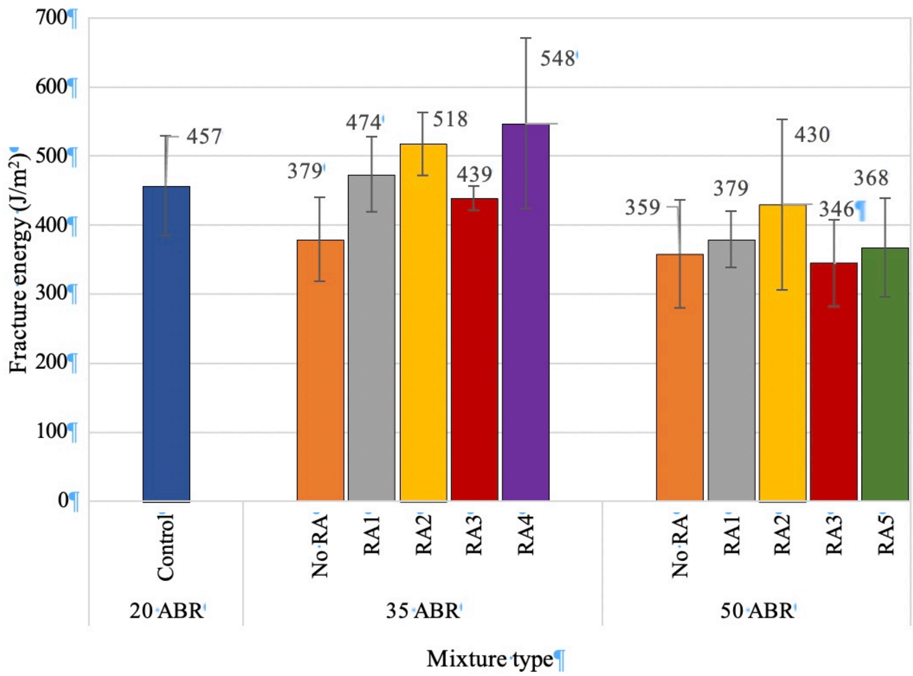


Figure 61. DCT Fracture Energy Results.

7 CONCLUSIONS AND RECOMMENDATIONS

7.1 Conclusions

This research evaluated the effectiveness of rejuvenators in restoring the properties of asphalt blends and mixtures with high RAP contents. The evaluation of binder blends included the following rheological and chemical tests: viscosity at 135°C, PG grading, Multiple Stress Creep Recovery (MSCR), and Fourier Transform Infrared Spectroscopy (FTIR). Asphalt blends with different RAP contents were tested with and without different rejuvenators. In addition to binder performance testing, this research also included mixture performance testing using the Indirect Tensile Asphalt Cracking Test (IDEAL-CT) for evaluating intermediate-temperature cracking resistance, the Disc-Shaped Compact Tension Test DCT for evaluating low-temperature cracking resistance, and the Asphalt Pavement Analyzer Test (APA) for evaluating rutting resistance. The APA test is conducted on short-term aged specimens while the IDEAL-CT and DCT tests are conducted on specimens subjected to short-term aging plus additional long-term aging.

The results of this study are summarized below.

Binder Testing

Rotational Viscosity

- The viscosity of the asphalt base binder PG 58-34 increased with the addition of RAP binder, and this increase in viscosity was directly related to the RAP content.
- The addition of rejuvenators decreased the viscosity of the recycled binder blends. This decrease in viscosity was influenced by the rejuvenators' composition and dosage.
- The viscosity aging index results indicated that the addition of rejuvenators decreased the aging susceptibility of the recycled asphalt blends, with exception of the PG 58-34+50%RAP+2.0%RA_5 blend.

Performance Grading and ΔTc Parameter

- The addition of recycled asphalt binder to the PG 58-34 base binder increased the stiffness of the asphalt binders but reduced their fatigue resistance, thermal cracking resistance, and stress relaxation property after RTFO plus 60-hour of oxidative aging in the PAV.
- The incorporation of rejuvenators counterbalanced these negative effects with the exception of the blend PG 58-34 + 50% RAP + 2.0% RA_5.
- When considering 35% ABR, an improvement in the ΔTc parameter after RTFO plus 60-hour PAV was observed for all rejuvenated recycled binder blends. When considering 50% ABR, an improvement in the ΔTc parameter was observed for RA_1, RA_2, and RA_3. The addition of RA_5 resulted in a slightly more negative ΔTc.

MSCR

- J_{nr} and % Recovery MSCR testing parameters were found dependent on the constituents of the binder blends (i.e., base binder type, recycled binder percentage, and RA type and dosage).
- % Recovery parameter was found to be highly influenced by the creep compliance J_{nr} of the binders, regardless of the presence and dosage of rejuvenators.

- Rejuvenators increased the magnitude of J_{nr}. This increase varied depending on the chemical composition and dosage of the additives as well as the RAP content.

G-R Parameter and Black Space Diagram

- *G-R* parameter results highlighted the binder stiffening effect from RAP, since the recycled asphalt binder blends showed consistently higher *G-R* parameters than the control recycled binders.
- The addition of 50% RAP binder replacement to the PG 58-34 base binder resulted in failing the *G-R* parameter criterion of 600 kPa for the significant block cracking after RTFO plus 60-hour PAV aging.
- The addition of rejuvenators to the recycled binder blends decreased the stiffness of all blends, before and after aging. For the recycled binder blends after aging, this decrease in stiffness was accompanied by an increase in the phase angle (δ).
- After RTFO plus 60-hour PAV aging, regardless of the content of recycled binder (i.e., 35 or 50% RAP), the binder blends rejuvenated with RA_1, RA_3 and RA_4 were located below the G-R damage zone and thus, are not likely to experience premature block cracking in the field.
- G-R Aging Index calculated as the fraction of the G-R parameter of the RTFO plus 60-h PAV-aged sample over that of the unaged sample indicated that the bio-based rejuvenator RA_1 decreased the aging susceptibility of the recycled binder blends, regardless of the content of recycled binder (i.e., 35 or 50% RAP).
- Stiffness ratio results after long-term aging indicated that the all the rejuvenated recycled binder blends contained 35% RAP remained softer than the control recycled binder. When considering the rejuvenated recycled binder blends containing 50% RAP, rejuvenators RA_2 and RA_5 did not improve the stiffness of the control recycled binder.

FTIR

- As expected, as the percentage of RAP binder increased (i.e., from 20% towards 50%) in the recycled binder blends, the growth of oxygen containing functionalities (i.e., carbonyl and sulfoxide groups) also increased.
- When considering 35% ABR, the oxidation of the rejuvenated recycled binder blends containing RA_1, RA_2, and RA_4 remained below the oxidation of the PG 58-34 + 35% RAP control recycled binder. The opposite was observed for the rejuvenated recycled binder blends containing RA_3.
- When considering 50% ABR, rejuvenated recycled binder blends containing RA_1 and RA_5 presented a lesser degree of oxidation than the PG 58-34 + 35% RAP control recycled binder; while recycled binder blends containing RA_2 and RA_3 presented a higher degree of oxidation.
- The reduction in oxygen-containing compounds due to the addition of rejuvenators was found dependent on the RA type (i.e., composition) and dosage as well as the RAP binder content.

Mixture Performance Testing

APA

- All high RAP mixes with and without rejuvenators had less rut depth compared to the 20% ABR control mix, except for 35%ABR/RA_4 mix.
- Since none of the mixtures exceeded the 7 mm rut depth threshold required by South Dakota DOT, it is expected that all the mixtures would have adequate rutting performance.

IDEAL-CT

- CT_{index} results showed that the 20% ABR control mix had better intermediate temperature cracking resistance than the non-rejuvenated and rejuvenated 35% ABR and 50% ABR mixes. This can be partially attributed to the higher asphalt content of the control mix.
- The use of rejuvenators showed an improvement in the average CT_{Index} values for rejuvenated mixes when compared to non-rejuvenated mixes at the same ABR content. However, when the variability of the test was considered, the difference in CT_{Index} did not show a statistically significant improvement for the 35% ABR and 50% ABR mixes except for two cases: mix 35% ABR/No RA versus 35% ABR/RA_2 and mix 50% ABR/No RA vs 50% ABR/RA_3.
- Limited additional IDEAL-CT tests conducted with the 35%ABR mix and one rejuvenator suggested the following:
  - For this specific study, results have indicated that the rejuvenator dosages recommended by the suppliers may not be sufficient to improve the intermediate temperature cracking resistance of high RAP mixes. This could be a consequence of the approach followed by the suppliers to determine the rejuvenator dosage being too conservative. Selecting the rejuvenator dosage by targeting the low-temperature PG grade (-34°C) of the virgin binder (after RFTO plus 60-hour PAV aging) could be more appropriate but warrant further investigation.
  - Tests conducted on samples after STOA and LTOA suggested that the effectiveness of rejuvenators may diminish with aging.
  - When a 75% binder availability Factor (BAF) was used, RA_1 was effective on improving the cracking performance of the 35% ABR mix, at intermediate temperature. This could be an indication that the use of rejuvenators in combination with higher asphalt content may produce mixes with improved intermediate temperature cracking performance.

DCT

- Fracture energy results showed that for the 35%ABR mixes, all the mixes except the non-rejuvenated mix and the mix with RA_3 had higher mean values compared to the 20%ABR mix. The non-rejuvenated mix had the lowest mean fracture energy. For the 50% ABR mixes, all the mixes had lower mean fracture energy values compared to the 20%ABR mix. In addition, except for 50% ABR/RA_3 mix, the mean fracture energy of all rejuvenated mixes were higher compared to the fracture energy of the corresponding non-rejuvenated mix.
- Statistical analysis including the control mix showed that for the 35% ABR mixes, significant differences only exist between fracture energy of the non-rejuvenated mix versus mixes with RA_2 and RA_4.On the other hand, statistical analysis showed that all the 50% ABR mixes with and without rejuvenators had similar fracture energy results and thus, are expected to have equivalent resistance against thermal cracking.

7.2 Recommendations

7.2.1 Proposed Testing Procedure to Evaluate Rejuvenators

The results of this research were used to develop a procedure to evaluate the effectiveness of rejuvenators in improving the performance of high recycled mixtures. It is important to point out that this procedure was developed based on binder and mixture test results corresponding to a limited set of raw materials (i.e., one virgin binder, one aggregate source, and one RAP source). Therefore, it is highly recommended that this procedure be re-verified using tests results conducted with additional materials used in South Dakota.

1) Select a control 20% ABR mix with satisfactory performance.

2) Select virgin binder and RAP material and determine their high-temperature (HT) and low-temperature (LT) performance grade (PG) per AASHTO M 320.

3) Select the rejuvenator and high RAP mix design to be evaluated.

4) The rejuvenator dosage should be indicated by the additive manufacturer using the results in step 2 along with the information of the mix design to be evaluated. For this project, the suppliers provided dosages to target the low temperature PG grade of the 20% ABR mix. Since the results of this study indicated that the dosages selected by the suppliers following this approach were too low, other approaches that have been recommended to select rejuvenator dosages should be considered. Examples of these approaches include the selection of rejuvenator dosages to target either the continuous high temperature PG grade or the low temperature PG grade to that of the target asphalt binder that satisfies both climate and traffic requirements (Epps Martin et al., 2018, Rodezno et al., 2021).

5) Conduct mixture performance tests to evaluate mixture performance with aging.

— For APA, samples are prepared from loose mix aged for four hours at 135°C (STOA).

— For IDEAL-CT and DCT, samples are prepared from STOA conditioned mix further aged for six hours at 135°C (LTOA).

a. Prepare samples with the selected RA dosage and RAP proportion combination to conduct the IDEAL-CT test.

b. Compare the IDEAL-CT results of the proposed mix to the results obtained for the control 20%ABR mixture.

c. If the IDEAL-CT results of the proposed mix meet the performance of the control mix, verify that the mix meets the APA threshold of 7 mm rut depth (specified by SDDOT), and the DCT results obtained for the control 20%ABR mix.

d. If the IDEAL-CT criterion is not satisfied, increase the RA dosage and/or increase the binder content of the mix and retest IDEAL-CT, APA, and DCT.

8 REFERENCES

Abbas, A.R., Mannan, U.A., and Dessouky, S. (2013). Effect of Recycled Asphalt Shingles on Physical and Chemical Properties of Virgin Asphalt Binders. Construction and Building Materials, Vol. 45, pp.162-172.

Anderson, A. and Marasteanu. M. (1999). Physical Hardening of Asphalt Binders Relative to Their Glass Transition Temperatures. Transportation Research Record: Journal of the Transportation Research Board, N. 166, pp. 27-34.

Anderson, D., Christensen, D., Bahia, H., Dongre, R., Sharma, M., Antle, C., and Button, J. (1994). Binder Characterization and Evaluation, Volume 3: Physical characterization, SHRP A-369. Washington DC: National Research Council.

Anderson, M., G. N. King, D. I. Hanson, and P. B. Blankenship. (2011). Evaluation of the Relationship between Asphalt Binder Properties and Non-Load Related Cracking. Journal of the Association of Asphalt Paving Technologists, Vol. 80, pp. 615-664.

Arámbula-Mercado, E., Kaseer, F., Martin, A. E., Yin, F., and Cucalon, L. G. (2018). Evaluation of recycling agent dosage selection and incorporation methods for asphalt mixtures with high RAP and RAS contents. Construction and Building Materials, 158, 432-442.

Asphalt Institute. (1996). Asphalt Hot-Mix Recycling, The Asphalt Institute Manual Series No. 20 (MS-20), Second Edition.

Bahia, H. (1991). Low-Temperature Isothermal Physical Hardening of Asphalt Cements. Ph.D. Thesis. Pennsylvania State University.

Bahia, H., Swiertz, D., Kilger, A. (2018). Modified Binder (PG+) Specifications and Quality Control Criteria. Project WHRP (0092-14-20) TPF-5(302) Extension Final Report.

Bonaquist, R. (2011a). Effect of Recovered Binders from Recycled Shingles and Increased RAP Percentages on Resultant Binder PG. Wisconsin Highway Research Program, Report No. WHRP 11-13.

Bonicelli, A., Calvi, P., Martinez-Arguelles, G., Fuentes, L., Giustozzi, F. (2017). Experimental Study on the Use of Rejuvenators and Plastomeric Polymersfor Improving Durability of High RAP Content Asphalt Mixtures. Construction and Building Materials, Vol. 155, pp. 37-44.

Castorena, C., Pape, S., & Mooney, C. (2016). Blending measurements in mixtures with reclaimed asphalt: use of scanning electron microscopy with X-ray analysis. Transportation Research Record: Journal of the Transportation Research Board, No. 2574, 57-63.

Churchill, E. V., S. N. Amirkhanian, J. L. Burati Jr. (1995). HP-GPC Characterization of Asphalt Aging and Selected Properties. Journal of Materials in Civil Engineering, Vol. 7, pp. 41-49.

Christensen, D. W. and Anderson, D. A. Interpretation of Dynamic Mechanical Test Data for Paving Grade Asphalt Cements. J. Assoc. Asphalt Pav. Technol., Vol. 61, pp. 67–116, 1992.

Coffey, S., DuBois, E., Mehta, Y., Nolan, A., & Purdy, C. (2013). Determine the Impact of Degree of Blending and Quality of Reclaimed Asphalt Pavement on Predicted Pavement Performance using Mechanistic-Empirical Design Guide (MEPDG). In Proceedings of the international conferences on the bearing capacity of roads, railways and airfields, pp. 445-454.

Cooper Jr, S. B., Mohammad, L. N., Elseifi, M. A., and Medeiros Jr, M. S. (2015). Effect of recycling agents on the laboratory performance of asphalt mixtures containing recycled asphalt shingles. Transportation Research Record, 2506(1), 54-61.

Copeland, A. (2011). Reclaimed asphalt pavement in asphalt mixtures: State of the practice

Corbett, L. W. (1969). Composition of Asphalt Based on Generic Fractionation Using Solvent Deasphalteneing, Elution-Adsorption Chromatography and Densiometric Characterization. Analytical Chemistry, Vol. 41, pp. 576-579.

Epps Martin et al. (2018). The Effects of Recyling Agents on Asphalt Mixtures with High RAS and RAP Binder Ratios. NCHRP 9-58 Draft Final Report.

Espinoza-Luque, A.F., Al-Qadi, I.L., and Ozer. H. (2018). Optimizing rejuvenator content in asphalt concrete to enhance its durability, Constr. Build. Mater. 179 (2018) 642–648.

Glover, C., R. R. Davison, C. H. Domke, Y. Ruan, P. Juristyarini, D. B. Knorr, and S. H. Jung. (2005). Development of a New Method for Assessing Asphalt Binder Durability with Field Validation. Publication FHWA/TX-05/1872-2, Report 0-1872-2, U.S. Department of Transportation.

Grilli, A., Gnisci, M., and Bocci, M. (2017). Effect of Ageing Pocess on Bitumen and Rejuvenated Bitumen. Construction and Building Materials, Vol. 136, pp. 474-481.

Haghshenas, H., Nabizadeh, H., Kim, Y-R., Santosh, K. (2016). Research on High-RAP Asphalt Mixtures with Rejuvenators and WMA Additives. Report No. SPR- P1(15) M016, Nebraska Department of Roads, Lincoln, NE.

Hajj, E., Souliman, M., Alavi, M., and Salazar, L. (2013). Influence of Hydrogreen Bio-Asphalt on Viscoelastic Properties of Reclaimed Asphalt Mixtures. Transportation Research Record: Journal of the Transportation Research Board, 2371, 13-22.

Hintz, C., R. Velasquez, Z. Li, and Bahia, H. (2011). Effect of Oxidative Aging on Binder Fatigue Performance. Journal of Association of Asphalt Paving Technologists, Vol. 80, pp. 527-548.

Huang, B., Li, G., Vukosavljevic, D., Shu, X., & Egan, B. K. (2005). Laboratory investigation of mixing hot-mix asphalt with reclaimed asphalt pavement. Transportation Research Record, 1929(1), 37-45.

Im, S., and Zhou, F. (2014). Field performance of RAS test sections and laboratory investigation of impact of rejuvenators on engineering properties of RAP/RAS mixes. Rep. No. FHWA/TX-14/0-6614-3, Texas Transportation Institute, College Station, TX.

Jennings, P. W., Pribanic, P. W., Campbell, W., Dawson, K., Shane, S., and Taylor, R. (1980). High Pressure Liquid Chromatography as a Method of Measuring Asphalt Composition. FHWA-MT-79-30 Final Report, Bozeman, Montana.

Jiang, Y., Gu, X., Zhou, Z., Ni, F., & Dong, Q. (2018). Laboratory Observation and Evaluation of Asphalt Blends of Reclaimed Asphalt Pavement Binder with Virgin Binder using SEM/EDS. Transportation Research Record, 2672(28), 69-78.

Johnson, K.-A. N., and Hesp, S. A. M. (2014). Effect of Waste Engine Oil Residue on Quality and

Durability of SHRP Materials Reference Library Binders. Transportation Research Record: Journal of the Transportation Research Board, No. 2444, pp. 102-109.

Kandhal, P. S. (1977). Low-Temperature Ductility in Relation to Pavement Performance. Low-Temperature Properties of Bituminous Materials and Compacted Bituminous Paving Mixtures, ASTM STP 628. C. R. Marek, Ed. American Society for Testing and Materials, pp. 95-106.

Karlsson, R. and Isacsson, U. (2003). Application of FTIR-ATR to Characterization of Bitumen Rejuvenator Diffusion. Journal of Materials in Civil Engineering, Vol. 15, pp.157-165.

Kaseer, F., Arámbula-Mercado, E., Cucalon, L. G., and Martin, A. E. (2018). Performance of asphalt mixtures with high recycled materials content and recycling agents. International Journal of Pavement Engineering, 1-15.

Kaseer, F., Arámbula-Mercado, E., & Martin, A. E. (2019). A Method to Quantify Reclaimed Asphalt Pavement Binder Availability (Effective RAP Binder) in Recycled Asphalt Mixes. Transportation Research Record, 0361198118821366.

Kaseer, F., Cucalon, L., Arámbula-Mercado, E., Martin, A., Epps, J. (2018). Practical tools for Optimizing Recycled Materials Content and Recycling Agent Dosage for Improved Short- and Long-Term Performance of Rejuvenated Binder Blends and Mixtures. Journal of the Association of Asphalt Paving Technologists, Vol. 87.

Kaseer, F., Yin, F., Arámbula-Mercado, E., Epps Martin, A. (2017). Characterization of Asphalt Mixtures with High RAP/RAS Contents and Recycling Agents. Presentation at the 96th Annual Meeting of the Transportation Research Board, Washington, DC.

Kelli-Anne, N. J., and Hesp, S. A. M. (2014). Effect of Waste Engine Oil Residue on the Quality and Durability of SHRP MRL Binders. 93rd Annual Meeting Transportation Research Board.

Kluttz, R. Q., Prepared Discussion on Relationships Between Mixture Fatigue Performance and Asphalt Binder Properties. Journal of the Association of Asphalt Paving Technologists, Vol. 88, 2019.

Lee, H., Mokhtari, A., and Williams, C. (2018). Development of quality standards for inclusion of high recycled asphalt pavement content in asphalt mixtures-phase III

Li, Y., Moraes, R., Lyngdal, E., and Bahia, H. (2016). Effect of Polymers and Oils Modification on the Aging Susceptibility of Asphalt Binders. Transportation Research Record: Journal of the Transportation Research Board, No. 2574, Vol. 1, pp. 28-37.

Loeber, L., Sutton, O., Morel, J., Valleton, J. M., and Muller, G. (1996). New Direct Observation of Asphalts and Asphalt Binders by Scanning Electron Microscopy and Atomic Force Microscopy. Journal of Microscopy, Vol. 182, pp. 32-39.

Mallick, R., Tao, M., O’sullivan, K., and Frank, R. (2010). Why not (use rejuvenator for) 100% RAP Recycling? Procedings, 89th TRB Annual Meeting, Transportation Research Board, Washington, DC.

Marasteanu, M., Zofka, A., Turos, M., Li, X., Velasquez, R., Buttlar, W., Paulino, G., Braham, A., Dave, E., Ojo, J., Bahia, H., Williams, C., Bausano, J., Gallistel, A., and McGraw, J. (2007). Investigation of Low Temperature Cracking in Asphalt. Pavements National Pooled Fund Study 776: Report N. MN/RC 2007-43, Minnesota Department of Transportation, St. Paul, MN.

McDaniel, R., and Anderson, M. (2001). Recommended Use of Reclaimed Asphalt Pavement in the Superpave Mix Design Method: Technician's Manual. National Cooperative Highway Research Program (NCHRP) Report 452.

Menapace, I., Cucalon, L., Kaseer, F., Arámbula-Mercado, E., Martin, A., Masad, E., and King, G. (2018). Effect of Recycling Agents in Recycled Asphalt Binders Observed with Microstructural and Rheological Tests. Construction and Building Materials, Vol. 158, pp. 61-74.

Milton, L., J. M. Chaffin, R. R. Davison, C. J. Glover, and J. A. Bullin. Changes in Corbett Fraction Composition during Oxidation of Asphalt Fractions. Transportation Research Record: Journal of the Transportation Research Board, No. 1638, pp. 40-46, 1998.

Mogawer, W., Bennert, T., Daniel, J. S., Bonaquist, R., Austerman, A., and Booshehrian, A. (2012). Performance characteristics of plant produced high RAP mixtures. Road Materials and Pavement Design, 13(sup1), 183-208.

Mogawer, W. S., Booshehrian, A., Vahidi, S., and Austerman, A. J. (2013). Evaluating the effect of rejuvenators on the degree of blending and performance of high RAP, RAS, and RAP/RAS mixtures. Road Materials and Pavement Design, 14(sup2), 193-213.

Mogawer, W., Bennert, T., Austerman, A., and Ericson, C. (2015). Using Binder and Mixture Space Diagrams to Evaluate the Effect of Re-Refined Engine Oil Bottoms on Binders and Mixtures after Ageing. Road Materials and Pavement Design, 16(2), in press.

Moraes, R. (2014). Investigation of Mineral Filler Effects on the Aging Process of Asphalt Mastics. Ph.D. Thesis, University of Wisconsin-Madison.

Moraes, R., and Bahia, H. (2015a). Effect of Mineral Filler on Changes in Molecular Size Distribution of Asphalts during Oxidative Ageing. Road Materials and Pavement Design, Vol. 16, Supplement 2, pp. 55-72.

Moraes, R., and Bahia, H. U. (2015b). Effect of Mineral Fillers on the Oxidative Aging of Asphalt Binders: A Laboratory Study Using Mastics. Transportation Research Record: Journal of the Transportation Research Board, No. 2506, pp. 19-31.

O'Sullivan, K. (2011). Rejuvenation of Reclaimed Asphalt Pavement (RAP) in Hot Mix Asphalt Recycling with High RAP Content. Master's thesis, Worcester Polytechnic Institute (WPI), Worcester, MA.

Ouyang, C., Wang, S., Zhang, Y. (2006). Improving the Aging Resistance of Styrene-Butadiene-Styrene Tri-block Copolymer Modified Asphalt by Addition of Antioxidants. Polymer Degradation and Stability, Vol. 91, p.795-804.

Pauli, A. T., Robertson, R. E., Eggleston, C. M., Branthaver, J. F., and Grimes, W. (2001). Atomic Force Microscopy Investigation of SHRP Asphalts. American Chemical Society, Vol. 46, pp. 104-110.

Petersen, J. C. (2009). A Review of the Fundamentals of Asphalt Oxidation: Chemical, physicochemical, Physical Property, and Durability Relationships. Washington, DC, United States: Transportation Research Board.

Petersen, J. C., Branthaver, J. F., Robertson, R. E., Harnsberger, P. M., Duvall, J. J., and Ensley, E. K. (1993). Effects of Physicochemical Factors on Asphalt Oxidation Kinetics. Transportation Research Record: Journal of the Transportation Research Board, No. 1391, pp. 1-10.

Pfeifer, B. 2018. “Illinois Department of Transportation’s Practices for Balanced Mix Design.” TRB Workshop 124, Balanced Asphalt Mixture Design: Implementation Efforts and Success Stories.

Read, J. and Whiteoak, D. (2003). The Shell Bitumen Handbook. 5th Edition, Edited by R. Hunter.

Reinke, Gerald. (2017). The Relationship of Binder Delta Tc to Mixture Fatigue. Southeastern Asphalt User Producer Group, Jacksonville, Florida.

RILEM. (2012). Proposal for a Pre-Normative FTIR Method. International Union of Laboratories and Experts in Construction Materials, Systems and Structures, TC SIB-TG5-Recycling.

Rodezno, C. R. Moraes, M. Fortunatus, and F. Yin. *Asphalt Binder Study*. WHRP 0092-19-04 Final Draft Report, 2021.

Rowe, G. M. (2011). Evaluation of the Relationship between Asphalt Binder Properties and Non-Load Related Cracking (prepared discussion). Journal of the Association of Asphalt Paving Technologists, Vol. 80, pp. 615-664.

Shen, J., Huang, B., and Hachiya, Y. (2004). Validation of Performance-Based Method for Determining Rejuvenator Content in HMA, Int. J. Pavement Eng. 5 (2) (2004) 103–109.

Tabatabaee, H., Kurt, T. (2017). Analytical Investigation of the Impact of a Novel Bio-Based Recycling Agent on the Colloidal Stability of Aged Bitumen. Road Materials and Pavement Design, DOI: 10.1080/14680629.2017.1304257, pp. 131-140.

Tran, N., Taylor, A., and Willis, R. (2012). Effect of rejuvenator on performance properties of HMA mixtures with high RAP and RAS contents. Rep. No. 12-05, National Center for Asphalt Technology (NCAT), Auburn, AL, 70 pp, 2012.

Turner, T. F., and Branthaver, J. F. (1997). DSC Studies of Asphalts and Asphalt Components. Asphalt Science and Technology, Arthur Usmani: Editor, New York, Marcel Dekker.

Van Deusen, D. (2018). Update on the Cracking Group Experiment: North Experiment on Thermal Cracking. 2018 Fall Sponsor Meeting, MnROAD/NCAT Partnership, Minneapolis, MN.

Wada, Y., and Hirose, H. (1960). Glass Transition Phenomenon and Rheological Properties of Petroleum Asphalt. Journal of the Physical Society of Japan, Vol. 15, No.10, pp. 1885-1894.

West, R. C., and Copeland, A. (2015). High RAP Asphalt Pavements: Japan Practice-Lesson Learned.

West, R., Rodezno, C., Leiva, F., and Yin, F. (2018). Development of a Framework for Balanced Mix Design. NCHRP 20-07/Task 406 Final Report http://onlinepubs.trb.org/onlinepubs/nchrp/docs/NCHRP20-07(406)_Revised_final_report.pdf.

Xie, Z., Tran, N., Julian, G., Taylor, A., and Blackburn, L. D. (2017). Performance of asphalt mixtures with high recycled contents using rejuvenators and warm-mix additive: field and lab experiments. Journal of Materials in Civil Engineering, 29(10), 04017190.

Yan, J., Zhang, Z., Zhu, H., Li, F., and Liu, Q. (2014). Experimental study of hot recycled asphalt mixtures with high percentages of reclaimed asphalt pavement and different recycling agents, ASTM J. Test. Eval. 42 (5).

Yin, F., Kaseer, F., Arámbula-Mercado, E., Martin, A. (2017). Characterising the Long-Term Rejuvenating Effectiveness of Recycling Agents on Asphalt Blends and Mixtures with High RAP and RAS

Contents. Road Materials and Pavement Design, pp. 273-292.

Yu, S., Shen, S., Zhang, C., Zhang, W., & Jia, X. (2017). Evaluation of the blending effectiveness of reclaimed asphalt pavement binder. Journal of Materials in Civil Engineering, 29(12), 04017230.

Yu, X., Zaumanis, M., Santos, S. Poulikakos, L. (2014). Rheological, Microscopic, and Chemical Characterization of the Rejuvenating Effect on Asphalt Binders. Fuel, Vol. 135, pp. 162-171.

Zaumanis, M., Mallick, R. B., and Frank, R. (2013). Evaluation of rejuvenator's effectiveness with conventional mix testing for 100% reclaimed asphalt pavement mixtures, Transp. Res. Rec.: J. Transp. Res. Board, 17–25. No. 2370.

Zaumanis, M., Mallick, R. B., and Frank, R. (2013). Use of rejuvenators for production of sustainable high content RAP hot mix asphalt. Paper presented at the The XXVIII International Baltic Road Conference.

Zaumanis, M., Mallick, R. B., and Frank, R. (2014). Determining optimum rejuvenator dose for asphalt recycling based on Superpave performance grade specifications. Construction and Building Materials, 69, 159-166.

Zaumanis, M., Mallick, R. B., and Frank, R. (2015). Evaluation of different recycling agents for restoring aged asphalt binder and performance of 100% recycled asphalt. Materials and Structures, 48(8), 2475-2488.

Zhou, F., Im, S., Morton, D., Lee, R., Hu, S., and Scullion, T. (2015). Rejuvenator Characterization, Blend Characteristics, and Proposed Mix Design Method. Journal of the Association of Asphalt Paving Technologists, Vol. 84, pp. 675-704.

Zhou, F., Karki, P., Xie, S., Yuang, J., Sun, L., Lee, R., Barborak, R. (2018). Toward the Development of Performance-related Specification for Bio-Rejuvenators. Journal of Construction and Building Materials, Vol. 174, 2018, pp. 443-455.

APPENDIX A: SURVEY QUESTIONNAIRE OF STATE HIGHWAY AGENCIES

The National Center for Asphalt Technology (NCAT) is conducting a research project for the South Dakota Department of Transportation, entitled "*Evaluation of Asphalt Binder Rejuvenators for Use in Recycled Asphalt*." Dr. Carolina Rodezno (mcr0010@auburn.edu) is the principal investigator. The primary objective of this project is to develop a laboratory testing protocol or procedure for the evaluation of asphalt binder rejuvenators.
As part of this research, we are conducting a survey to determine how state highway agencies evaluate and use asphalt binder rejuvenators in hot mix asphalt. The survey has a maximum of 14 questions and should take approximately 5 to 7 minutes to complete. Your response is important for the success of this project.

Thank you in advance for your participation.
NCAT

Q1. Please provide your contact information in case we need to contact you with follow-up questions regarding your responses.

a. Name
b. Agency
c. Title
d. E-mail
e. Phone Number

Q2. Does your agency allow the use of RAP and/or RAS in mainline asphalt mixtures?

a. Yes, allow RAP only
b. Yes, allow RAP and RAS
c. No

Q3. If "Yes" to Q2, what are the average and maximum allowable RAP and/or RAS percentages in mainline asphalt mixtures? Please indicate the percentages by weight of aggregate, by weight of total mix, or asphalt binder replacement.

Layer Type	**Surface Layer**	**Intermediate Layer**	**Binder Layer**
State Average			
Maximum Allowable			

Q4. What is your agency's current practice for approving RAP and/or RAS materials? (check all that apply)

a. Fractionate RAP
b. Grade extracted binder
c. Determine binder content
d. Determine gradation
e. Measure aggregate specific gravity
f. No requirement for RAP or RAS materials
g. Others, please indicate

Q5. Does your agency require performance testing on asphalt mixtures containing RAP and/or RAS?

a. Yes
b. No

Q6. If "Yes" to Q5, what performance tests and criteria are used?

Pavement Distress	**Performance Test**	**Test Criteria**
Rutting		
Cracking		
Moisture Damage		

Q7. Does your agency allow or require the use of rejuvenators in asphalt mixtures with RAP and/or RAS?

a. Yes
b. No

Q8. If "Yes" to Q7, what are the criteria to decide when asphalt rejuvenators should be used?

Q9. If "Yes" to Q7, how does your agency evaluate and approve asphalt rejuvenators?

Q10. If "Yes" to Q7, does your agency have a list of approved rejuvenators?

a. Yes, please add a link to the location of the approved products and the specifications
b. No

Q11. If "Yes" to Q7, does your agency have an approach for determining the dosage of rejuvenators to be used in the mixture?

Q12. Would your agency accept testing from AASHTO NTPEP (National Transportation Product Evaluation Program) in lieu of conducting testing in-house for rejuvenators?

a. Yes
b. No

Q13. Has your agency constructed any field sections using asphalt mixtures with rejuvenators?

a. Yes
b. No

Q14. If "Yes" to Q10, are there any technical reports or papers documenting the performance of these sections? Please provide a link or a copy of the publication.