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Novel Methods for Adding Rejuvenators in Asphalt Mixtures with High Recycled Binder Ratios

NRRA FLEXIBLE TEAM

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The overall objective of this study was to explore three novel rejuvenator application methods using the emulsion and foaming technologies and determine their impacts on the workability and long-term cracking resistance of high-reclaimed asphalt pavement (RAP) asphalt mixtures. To that end, a comprehensive experimental plan was developed, which consisted of four supplementary experiments focusing on rejuvenator characterization, foaming measurements of rejuvenators and rejuvenated asphalt binders, RAP pretreatment and marination evaluations, and mixture performance testing, respectively. Test results indicated that adding rejuvenators for RAP pretreatment improved the overall quality characteristics of RAP. Among the three rejuvenator application methods evaluated for RAP pretreatment, the emulsion method was found more effective than the spray-on and foaming methods. Marinating the pretreated RAP had a notable impact on the rheological and chemical properties of the extracted RAP binders, but it did not significantly affect the workability, appearance, and color consistency of RAP. Adding rejuvenators, in general, improved the workability and cracking resistance of high-RAP mixtures, although the improvement in mixture performance test results, in some cases, was not statistically significant. Among the different rejuvenator application methods, pre-blending the rejuvenator into the virgin binder (with or without foaming) provided slightly better or equivalent rejuvenating effectiveness and thus, mixture performance properties, than adding the rejuvenator for RAP pretreatment. Based on the findings of the study, it was recommended that asphalt contractors continue to use the pre-blending method of adding rejuvenators for the design and production of high-RAP mixtures due to performance and ease of operation considerations.

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NOVEL METHODS FOR ADDING REJUVENATORS IN ASPHALT MIXTURES WITH HIGH RECYCLED BINDER RATIOS

FINAL REPORT

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LIST OF ABBREVIATIONS

%R	Percent Recovery
|G*|	Shear Complex Modulus
$\|m_{75}\|$	Post-peak Slope at 75% of Peak Load
BBR	Bending Beam Rheometer
CII	Colloidal Instability Index
CMOD	Crack Mouth Opening Displacement
CT_{index}	Cracking Tolerance Index
DCT	Disc-shaped Compact Tension
DSR	Dynamic Shear Rheometer
DWT	Dongre Workability Test
ER	Expansion Ratio
ER_{max}	Maximum Expansion Ratio
FI	Foamability Index
G_f	Fracture Energy
G-R Parameter	Glover-Rowe Parameter
IDEAL-CT	Indirect Tensile Asphalt Cracking Test
I-FIT	Illinois Flexibility Index Test
J_{nr}	Non-recoverable Creep Compliance
l_{75}	Post-peak Displacement at 75% of Peak Load
MnDOT	Minnesota Department of Transportation
MSCR	Multiple Stress Creep Recovery
NCAT	National Center for Asphalt Technology
NRRA	National Road Research Alliance
OT	Overlay Test
PAV	Pressure Aging Vessel
RAP	Reclaimed Asphalt Pavement
RAS	Recycled Asphalt Shingle
RBR	Recycled Binder Ratio
RTFO	Rolling Thin Film Oven

SARA	Saturate, Aromatic, Resin, and Asphaltene
SGC	Superpave Gyratory Compactor
$t_{1/2}$	Half Life
TAP	Technical Advisory Panel
WMA	Warm Mix Asphalt
δ	Phase Angle
ΔT_c	Delta T_c
Θ	Contact Angle

EXECUTIVE SUMMARY

The use of reclaimed asphalt pavement (RAP) in asphalt mixtures provides significant economic and environmental benefits. From a performance perspective, adding RAP has an overall stiffening impact on the asphalt mixture due to the heavily aged nature of the asphalt binder in RAP. As a result, asphalt mixtures containing RAP, especially those with a high RAP content, typically have good rutting resistance, but they could be highly susceptible to cracking and other durability-related distresses. Over the years, many laboratory studies and field trials have demonstrated the potential of using rejuvenators to abate the cracking challenges of high-RAP asphalt mixtures, although the rejuvenating effectiveness has been found to vary greatly depending on the type and dosage of the rejuvenator, the source and quality of the virgin and RAP binder, and the RAP content, among other mix design variables.

The most common method of adding rejuvenators when preparing rejuvenated RAP mixtures in the laboratory is to pre-blend the rejuvenator into the virgin binder prior to mixing with the virgin aggregate and RAP. For production of these mixtures in the field, the rejuvenator can be either pre-blended into the virgin binder at the asphalt terminal or in-line blended with the virgin binder at the asphalt plant. Although this pre-blending method works well, there is an alternative rejuvenator application method that adds the rejuvenator into the RAP instead of the virgin binder, which is often referred to as the RAP pretreatment method. This alternative method appears conceptually promising because it allows the rejuvenator to directly contact and interact with the RAP binder, which has the potential to enhance the rejuvenating effectiveness from the mixture performance perspective. However, several existing studies that evaluated the RAP pretreatment method did not obtain favorable results. Some of those studies reported that it was challenging to achieve a uniform dispersion of the rejuvenator with the RAP due to the small amount of rejuvenator used, which was hypothesized to result in the lack of improvement in mixture performance from adding the rejuvenator via the RAP pretreatment method.

This study explored three novel rejuvenator application methods based on the traditional pre-blending and RAP pretreatment methods. The first two methods used the emulsion and foaming technology, respectively, to apply the rejuvenator for RAP pretreatment. Compared to the traditional RAP pretreatment method, adding the rejuvenator in an emulsion or foaming form was expected to provide better dispersion due to the significantly increased volume and surface area. The third application method was adapted from the pre-blending method by applying the rejuvenated virgin binder for mixing with the virgin aggregate and RAP through foaming. With foaming, the rejuvenated virgin binder underwent volume expansion and viscosity reduction, which was anticipated to provide better coating and mixing of the virgin aggregate and RAP with the rejuvenated virgin binder.

The overall objective of the study was to evaluate the impacts of different rejuvenator application methods, including the three proposed exploratory methods and the traditional pre-blending method, on the performance properties of high-RAP asphalt mixtures. To that end, four supplementary experiments were conducted, which focused on rejuvenator characterization, foaming measurements of rejuvenators and rejuvenated asphalt binders, RAP pretreatment and marination evaluations, and mixture performance testing, respectively.

The first experiment focused on characterizing the wettability and viscosity of two bio-based rejuvenators evaluated in the study (i.e., RA1 and RA2), using the Sessile Drop and Rotational Viscosity tests, respectively. The two rejuvenators, in both their original and emulsion forms, showed good wettability with a PG 67-22 and PG 58S-28 binder. The two rejuvenators had similar viscosity at the unaged and Rolling Thin Film Oven (RTFO) aged conditions but distinctly different viscosity at the standard and extended Pressure Aging Vessel (PAV) conditions, which highlighted their varying susceptibility to oxidative aging due to differences in chemical compositions.

The second experiment was to optimize the foaming conditions of the two rejuvenators and two rejuvenated asphalt binders (i.e., PG 67-22 binder with RA1 and PG 58S-28 binder with RA2). Foaming was performed using the Wirtgen foamer at different foaming conditions (i.e., a combination of temperature and water content). Quantitative foaming measurements were taken with a laser distance meter, which recorded the height of the foam throughout the entire foaming process. The foam height data was then processed to generate the volume expansion curve and calculate several foaming index parameters. Overall, both the rejuvenators and rejuvenated asphalt binders exhibited good foaming characteristics at most of the selected foaming conditions. Based on the foamability index (FI) parameter, the optimum foaming conditions were selected as follows: 120°C (248°F) and 3% water content for RA1; 130°C (266°F) and 3% water content for RA2; and 150°C (302°F) and 2% water content for both the rejuvenated PG 67-22 binder with RA1 and the rejuvenated PG 58S-28 binder with RA2. These optimum foaming conditions were further evaluated for RAP pretreatment and preparation of high-RAP mixtures in the last two experiments of the study.

The third experiment included two sub-experiments to determine the impacts of RAP pretreatment and RAP marination, respectively, on the quality characteristics of RAP. The first sub-experiment evaluated three different rejuvenator application methods for RAP pretreatment: spray-on, emulsion, and foaming. Quality characterization of the untreated and pretreated RAP was conducted primarily based on the Dongre Workability Test (DWT) and grayscale-based image analysis. Furthermore, the moisture content of the RAP was measured before and after pretreatment with the rejuvenator. The results indicated that the emulsion method was more effective than the spray-on and foaming methods for RAP pretreatment as it yielded the resultant RAP sample with better overall quality characteristics from the workability, appearance, and color consistency perspectives. Based on this finding, the pretreated RAP with the emulsified rejuvenator was selected for further evaluation in the sub-experiment focusing on RAP marination. This second sub-experiment evaluated four RAP marination conditions after pretreatment: 1.5 hours at 135°C (275°F), 3 hours at 135°C (275°F), 3 days at room temperature, and 7 days at room temperature. At each marination condition, the pretreated RAP was characterized using the DWT and grayscale-based image analysis, and the extracted RAP binder was characterized through Dynamic Shear Rheometer (DSR), Bending Beam Rheometer (BBR), and Saturate, Aromatic, Resin, and Asphaltene (SARA) Fraction testing. Test results indicated that although marination had a notable impact on the rheological properties and SARA fractions of the extracted binders, it did not significantly affect the overall quality characteristics of the pretreated RAP in terms of workability, appearance, or color consistency.

Finally, the last experiment focused on mixture performance testing of high-RAP mixtures with and without rejuvenators. The experimental plan included two high-RAP mix designs, two virgin binders, two rejuvenators, and four rejuvenator application methods. A total of 10 mixtures, including two control mixtures without rejuvenator and eight rejuvenated mixtures, were tested with the DWT for workability evaluation and the Indirect Tensile Asphalt Cracking Test (IDEAL-CT) and Disc-shaped Compact Tension (DCT) test to evaluate mixture cracking resistance. Test results indicated that adding rejuvenators, in general, improved the mixture workability and cracking resistance, although in some cases, the improvement in test results was not statistically significant if considering the variability of the test. Among the different rejuvenator application methods evaluated in the study, the pre-blending methods provided slightly better or equivalent rejuvenating effectiveness and resultant mixture performance properties than the RAP pretreatment methods.

Based on the test results and findings of this study, it is recommended that asphalt contractors continue to use the pre-blending method of adding rejuvenators for the design and production of high-RAP asphalt mixtures because of performance and ease of operation considerations. Future research is suggested to further evaluate the use of the DWT as a quick tool to evaluate the overall quality and consistency of RAP stockpiles for asphalt mixture design and production.

CHAPTER 1: INTRODUCTION

1.1 BACKGROUND

The use of RAP in asphalt mixtures provides significant economic and environmental benefits. However, because the heavily aged binders in RAP are stiffer and more brittle than virgin binders, asphalt mixtures with high RAP contents tend to be more susceptible to cracking and durability-related distresses. Asphalt researchers and practitioners have explored the use of asphalt recycling agents (RAs), including rejuvenators, to help mitigate the stiffening impact of RAP. RAs are defined as organic materials with chemical and physical characteristics selected to restore the properties of aged asphalt to achieve target specification limits (Asphalt Institute, 1986). The optimal rejuvenation of the RAP binder depends not only on the viscosity-reducing capacity of the rejuvenator but also on its chemical composition. Furthermore, the degree of dispersion and interaction of the rejuvenator with the RAP is also important because it allows changes in the intermolecular agglomeration and self-assembly of the asphalt polar micelles, affecting the overall performance of the resultant RAP binder and mixture.

Currently, the most common method for adding a rejuvenator into RAP mixtures is to pre-blend it into the virgin binder prior to mixing with the virgin aggregate and RAP, which is often referred to as the pre-blending method. For mixture production in the field, the rejuvenator can be either pre-blended into the virgin binder at the asphalt terminal or in-line blended with the virgin binder at the asphalt plant. This practice has been successfully used in many field projects in the United States (West et al., 2018; Epps Martin et al., 2019; Vrtis, 2019; West et al., 2021). As an alternative to the pre-blending method, a rejuvenator can also be added directly to the RAP for pretreatment purposes, which is known as the RAP pretreatment method. It is hypothesized that with this alternative approach, the RAP will get largely rejuvenated prior to mixing with the virgin binder and aggregate, allowing maximum direct contact and interaction of the rejuvenator with the RAP binder. However, several studies investigated this alternative method but found no performance improvement over the traditional pre-blending method (Kaseer et al., 2019; Xie et al., 2020). One challenge reported by these studies is that due to the low dosage of the rejuvenator used (typically less than 1% by weight of the RAP), it did not have a good uniform dispersion with the RAP, as shown in Figure 1, and therefore, failed to provide the expected rejuvenating results.

Figure 1. Non-uniform Dispersion of Rejuvenator in RAP when Added using the Traditional RAP Pretreatment Method (Xie et al., 2020)

Over the years, asphalt researchers have evaluated different rejuvenator application and laboratory mixing methods of asphalt mixtures containing RAP and/or recycled asphalt shingle (RAS). For example, Xie et al. (2019) assessed the impacts of rejuvenator type and mixing method on the volumetric properties of a 50% RAP mixture, finding that the mixing method significantly impacted the design air voids of the mixture. They also found that for the mixing method that combined all the component materials (i.e., virgin binder, virgin aggregate, RAP, and rejuvenator) simultaneously for mixing without pre-blending or pre-mixing, rejuvenator type had a significant impact on the voids in the mineral aggregate (VMA) of the mixture. In another study, Xie et al. (2020) assessed the pre-blending method and the belt-spraying method of adding rejuvenators on the preheated RAP, along with 48-hour and 14-day room-temperature marination of the pretreated RAP, for a 40% RAP mixture and a 25% RAP plus 5% RAS mixture. It was found that adding rejuvenators, in general, improved the durability and intermediate-temperature cracking resistance of the RAP/RAS mixtures as characterized using the Cantabro test, Illinois Flexibility Index Test (I-FIT), and Overlay Test (OT), but there was no significant difference in the resultant mixture performance between the different rejuvenator application methods. Rathore et al. (2020) evaluated the impacts of rejuvenator application method, mixing temperature and time, and mixer equipment on the Indirect Tensile Strength and Stiffness Modulus test results of a 60% RAP mixture with a tall-oil based rejuvenator. The study found that the pre-blending method and the RAP pretreatment method (with and without 22-hour room-temperature marination) of adding rejuvenators did not significantly affect the performance test results. Furthermore, mixing temperature had a significant impact on the performance test results of the RAP mixture while mixing time did not. Finally, the study recommended a mixing procedure for preparing high-RAP mixtures containing rejuvenators in the laboratory.

In a plant study, Zaumanis et al. (2019) discussed ten potential rejuvenator addition locations at an asphalt batch plant and conducted full-scale plant evaluation for the two most promising locations: 1) spraying the rejuvenator onto the RAP conveyor belt upstream of the dryer, and 2) adding the rejuvenator directly into the pugmill for mixing with the RAP. RAP samples were collected at various locations of the plant, and asphalt binders were extracted and recovered for a battery of rheological and

chemical testing. Test results indicated that there was no significant difference in the binder properties for the two rejuvenator addition locations. This lack of discrimination was attributed to the asphalt extraction and recovery process that inherently blended the rejuvenator with the RAP binder. Because of this limitation, the study recommended conducting mixture performance tests to further evaluate the two rejuvenator addition locations in the next step of the research.

Despite the previous efforts, it is believed that the rejuvenating effectiveness of the traditional pre-blending and RAP pretreatment methods of adding rejuvenators can be further enhanced using emulsion or foaming technology. For the pre-blending method, the volume and surface area of the rejuvenated virgin binder can be expanded by up to 10 to 20 times through the mechanical foaming process, which allows better dispersion and mixing of the rejuvenator and virgin binder with the RAP. Another performance benefit associated with foaming the rejuvenated virgin binder is the reduction of binder viscosity, which is expected to provide improved workability and compactability of the resultant mixture (Newcomb et al., 2015). Because of the wide availability of plant foaming units among asphalt contractors in the United States (Williams et al., 2020), this foaming-enhanced pre-blending method of adding rejuvenators for high-RAP mixtures has good implementation potential.

For the RAP pretreatment method, better dispersion and mixing of the rejuvenator with the RAP can be achieved when the rejuvenator is applied in an emulsion or foam form. Previous experience at the National Center for Asphalt Technology (NCAT) shows that foaming a rejuvenator can provide the same volume expansion and viscosity reduction benefits as foaming an asphalt binder. For illustration purposes, Figure 2 presents the volume expansion curves of two bio-based rejuvenators foamed at 120°C (248°F) and 2% water content using the Wirtgen foamer. As shown, both rejuvenators exhibited good foaming behavior in terms of volume expansion and foam stability. Because of the increased volume and surface area, the rejuvenator after foaming is expected to provide better dispersion and mixing with the RAP, as illustrated in Figure 3. This foaming-enhanced RAP pretreatment method has been recognized as one of the major contributing factors for the successful use of high-RAP (up to 75%) asphalt mixtures in Japan (Koshi et al., 2017).

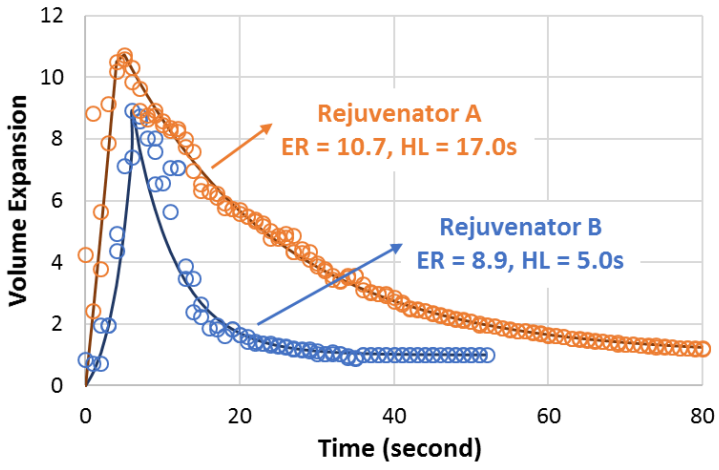


Figure 2. Volume Expansion Curves of Two Bio-based Rejuvenators

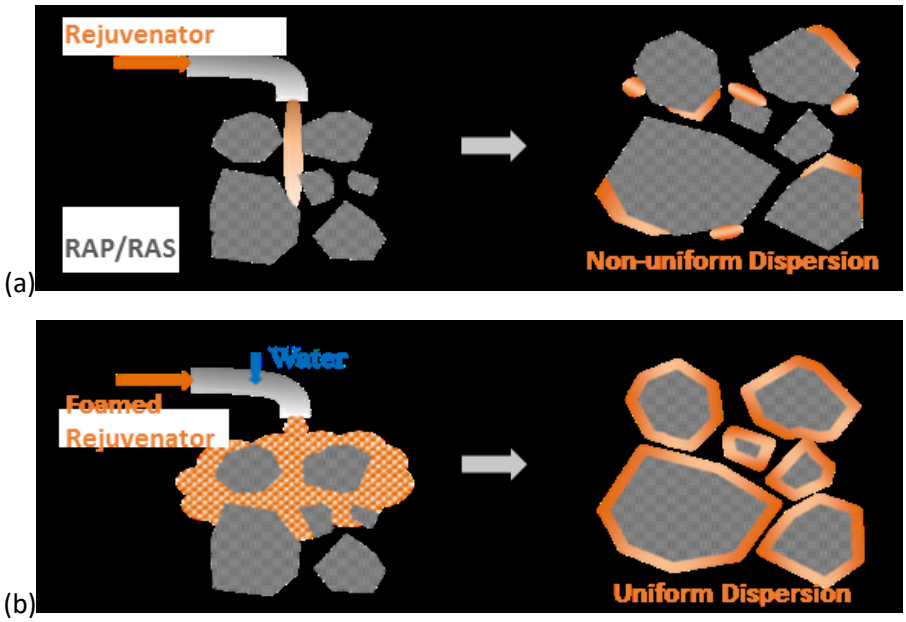


Figure 3. Illustrative Comparison of RAP Pretreatment; (a) with Rejuvenator, (b) with Foamed Rejuvenator (Koshi et al., 2017)

In addition to foaming, emulsion also allows for the volume expansion of the rejuvenator due to the addition of surfactant and water used in the emulsification and dilution process. Recently, emulsified rejuvenators have been widely used in rejuvenating seal applications as a pavement preservation treatment, where they are applied to an existing asphalt pavement surface to preserve its functional and structural integrity and delay a more costly rehabilitation treatment (Moraes, 2019). Given the success of this application, it is believed that the rejuvenating effectiveness of the RAP pretreatment method can be improved by adding the rejuvenator in an emulsion form. By increasing the physical dispersion and chemical diffusion between the rejuvenator and the RAP, this emulsion-enhanced RAP pretreatment method is expected to further improve the cracking resistance and durability of high-RAP mixtures.

This study was proposed to explore three novel methods of adding rejuvenators for high-RAP mixtures and determine whether they could provide better rejuvenating effectiveness and performance enhancement than the traditional pre-blending and RAP pretreatment methods. As compared to the traditional methods, the three proposed exploratory methods were intended to enhance the dispersion and diffusion of the rejuvenator with the RAP using the emulsion or foaming technology. The differences between the traditional and proposed methods of adding rejuvenators are summarized as follows:

- Traditional pre-blending method: the rejuvenator is pre-blended into the virgin binder, which is then mixed with the virgin aggregate and RAP for mixture production.
- Foaming-enhanced pre-blending method: the rejuvenator is pre-blended into the virgin binder, which is then foamed with water and mixed with the virgin aggregate and RAP for mixture production.

- Traditional RAP pretreatment method: the rejuvenator is directly added into the RAP as a pretreatment, which is then mixed with the virgin binder and aggregate for mixture production.
- Foaming-enhanced RAP pretreatment method: the rejuvenator is foamed with water to pretreat the RAP, which is then mixed with the virgin binder and aggregate for mixture production.
- Emulsion-enhanced RAP pretreatment method: the rejuvenator is emulsified and diluted with water to pretreat the RAP, which is then mixed with the virgin binder and aggregate for mixture production.

1.2 OBJECTIVES

The overall objective of this study was to explore three novel methods of adding rejuvenators and determine their impacts on the performance properties of high-RAP asphalt mixtures. Specifically, the study sought to: 1) characterize the wettability and viscosity of rejuvenators; 2) optimize the foaming characteristics of rejuvenators and rejuvenated asphalt binders; 3) evaluate the effects of RAP treatment and marination on the quality characteristics of RAP as well as the rheological and chemical properties of the extracted RAP binders; and 4) determine the impacts of different rejuvenator application methods on the workability, intermediate-temperature cracking resistance, and thermal cracking resistance of high-RAP asphalt mixtures.

CHAPTER 2: EXPERIMENTAL PLAN

To accomplish the objectives of the study, a comprehensive experimental plan consisting of four supplementary experiments was proposed. These experiments are briefly described below with more details provided in the following subsections.

1. The first experiment focused on characterizing the wettability and viscosity of the two rejuvenators used in the study. The Sessile Drop test was conducted to measure the contact angle of the rejuvenators, in both their original and emulsion forms, with two asphalt binders. Furthermore, the Rotational Viscosity test was conducted to measure the viscosity of the rejuvenators at various aging conditions. This experiment is referred to as the *Rejuvenator Characterization* experiment in the report.
2. The second experiment was to determine the foaming characteristics and optimize the foaming conditions of the rejuvenators and rejuvenated asphalt binders. Quantitative foaming measurements were taken using a laser distance meter and the results were analyzed to develop the volume expansion curve and calculate foaming index parameters. Based on the results, the optimum foaming conditions of the rejuvenators and rejuvenated asphalt binders were selected and further evaluated for RAP pretreatment and preparation of high-RAP mixtures for performance testing. This experiment is referred to as the *Foaming* experiment.
3. The third experiment sought to evaluate the pretreatment and marination of RAP with rejuvenators and their impacts on the RAP quality characteristics. Three rejuvenator application methods were evaluated for RAP pretreatment applications: spray-on, emulsion, and foaming. The quality characterization of untreated and pretreated RAP was conducted using the DWT and grayscale-based image analysis. Based on the results, the most effective RAP pretreatment method was selected to further investigate the effect of marination on the RAP quality characteristic. Four laboratory marination conditions were assessed, which included two accelerated, elevated-temperature marination methods and two extended, ambient-temperature marination methods. DWT and grayscale-based image analyses were conducted to determine the impact of marination on the quality characteristics of the pretreated RAP with the rejuvenator. Furthermore, DSR, BBR, and SARA fraction testing were performed to assess the rheological and chemical properties of the extracted RAP binders with selected marination conditions. This experiment is referred to as *RAP Pretreatment* and *RAP Marination* sub-experiments.
4. The last experiment focused on the performance testing of high-RAP mixtures prepared with different rejuvenator application methods, which included: 1) pre-blending the rejuvenator into the virgin binder, 2) pretreating the RAP with the emulsified rejuvenator, 3) pretreating the RAP with the foamed rejuvenator, and 4) foaming the pre-blended virgin binder with the rejuvenator. DWT was used for mixture workability evaluation while the IDEAL-CT and DCT were conducted to evaluate the mixture resistance to intermediate-temperature and thermal cracking, respectively. This experiment is referred to as the *Mixture Performance Testing* experiment.

2.1 MATERIALS SELECTION AND MIX DESIGN

Two high-RAP mix designs were used in this study to evaluate different rejuvenator application methods and their impacts on mixture performance. The mixture design information including virgin binder type, mix component proportions, and aggregate gradation is summarized in Table 1. Mix design A was a 9.5mm NMAS Superpave mixture with 45% RAP, which corresponded to a recycled binder ratio (RBR) of 0.40. Mix design B was a 12.5mm NMAS Superpave mixture with 50% RAP corresponding to a RBR of 0.51. Mix design A was evaluated with a PG 67-22 (PG 64-22) neat binder to represent asphalt mixtures in southern states, while mix design B was tested with a PG 58S-28 neat binder to represent asphalt mixtures in northern states.

Table 1. High-RAP Mix Design Summary

Properties	Mix Design A	Mix Design B
NMAS (mm)	9.5	12.5
Virgin Binder PG	PG 67-22	PG 58S-28
Recycled Binder Ratio	0.40	0.51
Total Asphalt Content (%)	5.9	5.6
Sieve (mm)	Percent Passing (%)	
19	100.0	100.0
12.5	100.0	98.2
9.5	97.0	89.5
4.75	76.0	54.8
2.36	53.4	37.1
1.18	40.9	30.4
0.6	30.6	25.3
0.3	16.5	18.8
0.15	9.7	12.4
0.075	6.3	7.0

The properties of the RAP used in each mix design are summarized in Table 2, which include the binder content of the RAP, true grade of the extracted RAP binder, and the RAP aggregate gradation. The binder content of RAP A (i.e., the RAP used in mix design A) and RAP B (i.e., the RAP used in mix design B) was determined as 5.30% and 5.72%, respectively, using the ignition method. Asphalt binders were extracted (using trichloroethylene) and recovered from the two RAP sources in accordance with AASHTO T164 and ASTM D5404, and then tested to determine their PG grades based on the Superpave binder specifications. The extracted and recovered binder from RAP A was graded to be PG 100+2 with a true grade of PG 105.0-2.1 and a delta Tc (ΔTc) of -7.3°C after RTFO plus 20 hours of PAV aging, while the extracted binder of RAP B was graded to be PG 94-4 with a true grade of PG 98.4-6.0 and a ΔTc of -13.4°C at RTFO plus 20-hour PAV aging.

Table 2. RAP Property Summary

Properties	RAP A	RAP B
NMAS (mm)	9.5	9.5
RAP Binder Content (%)	5.30	5.72
Recovered RAP Binder PG	PG 100-(+2)	PG 94-4
Sieve (mm)	Percent Passing (%)	
19	100	100
12.5	100	99
9.5	98	97
4.75	82	83
2.36	65	69
1.18	54	58
0.6	42	48
0.3	25	37
0.15	13	24
0.075	8.1	13.5

Two bio-based rejuvenators were used in this study to evaluate different application methods and their effectiveness in improving the workability and cracking resistance of high-RAP mixtures. One was used with mix design A and the PG 67-22 binder and is referred to RA1. The other was used with mix design B along with the PG 58S-28 binder and is referred to RA2. The two rejuvenators were also evaluated in an emulsion form for RAP pretreatment and marination applications. The emulsified RA1 contained 40% water and the emulsified RA2 contained 30% water. Figure 4 shows the two rejuvenators and the two emulsified rejuvenators used in the study. For mix design A, the dosage of RA1 was 16.1% by weight of the RAP binder (or 10.8% by weight of the virgin binder), which was suggested by the supplier to target 76°C as the high-temperature PG of the rejuvenated RAP binder. For mix design B, the dosage of RA2 was 6.0% by weight of RAP binder (or 6.3% by weight of the virgin binder), which was suggested by the supplier to match the low-temperature true grade of a corresponding 20% RAP binder blend (i.e., -23.7°C) based on theoretical blending chart analysis.

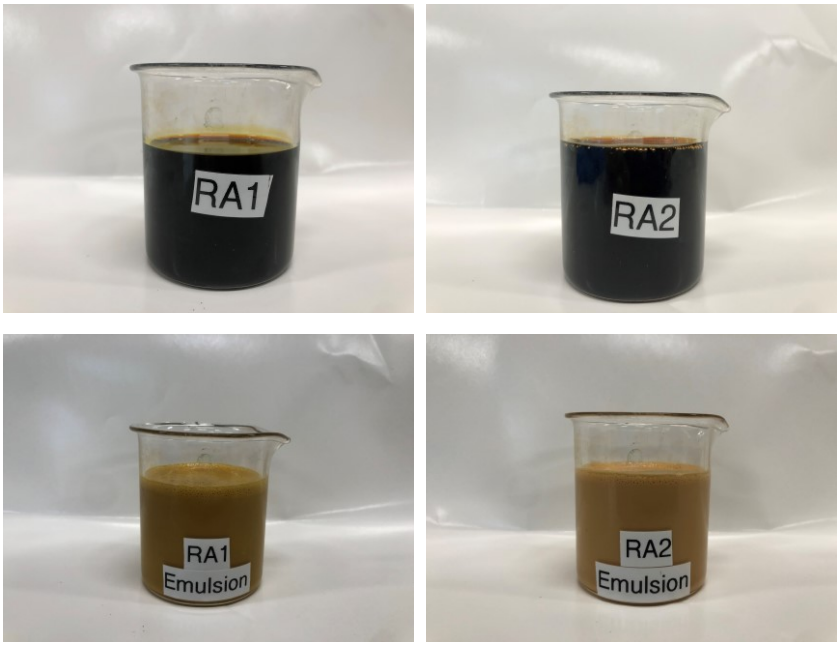


Figure 4. Rejuvenators and Emulsified Rejuvenators used in the Study

2.2 WETTABILITY AND VISCOSITY CHARACTERIZATION OF REJUVENATORS

This section discusses the experimental plan of the *Rejuvenator Characterization* experiment for determining the wettability and viscosity of the two rejuvenators used in the study.

2.2.1 Characterization of the Wettability of Asphalt Rejuvenators

Formation of a chemical or a physical bond requires the existence of a well-established contact, which involves, in the case of a liquid applied on a solid, a suitable wetting of the solid. The wetting refers to the propensity of a liquid to cover a given solid surface rather than to stay in a compact droplet that minimizes the surface contact (Shanahan, 1991). One method to quantify the surface wetting characteristics of a rejuvenator is to measure the contact angle (θ) of a drop of rejuvenator placed on the surface of a solid. A zero-contact angle is also called perfect wetting and hence, indicates spontaneous spreading. If $\theta < 90°$, the rejuvenator is said to wet the solid, and this condition reflects good wetting. If $\theta > 90°$, the rejuvenator is said to be non-wetting the surface, and this condition indicates poor wetting. Therefore, a low wetting surface would provide a high contact angle, while a high wetting surface would provide a low contact angle (Figure 5). In this study, the wettability capacity of the two selected rejuvenators was determined using the Sessile Drop test, as shown in Figure 6.

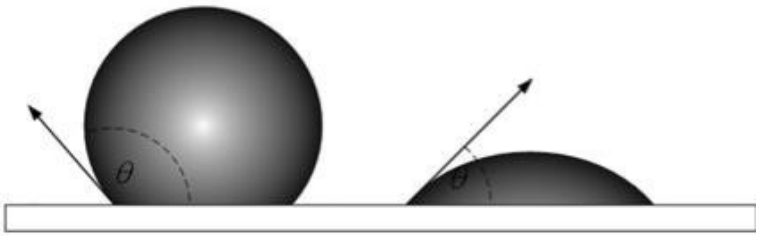


Figure 5. Illustration of the Contact Angle Concept; High Contact Angle indicating Low Wetting Surface (Left), Low Contact Angle indicating High Wetting Surface (Right)

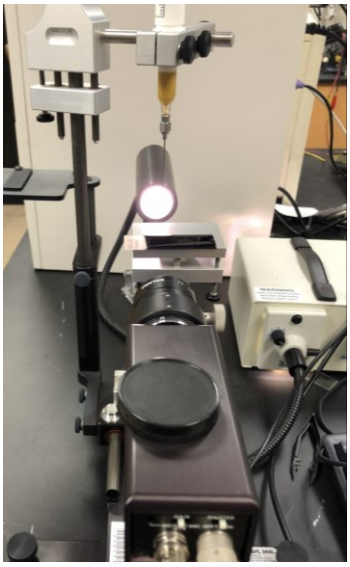


Figure 6. Sessile Drop Test Equipment

The Sessile Drop test involved placing a flat sample of the PG 67-22 or PG 58S-28 virgin binder underneath a syringe that deposited a drop of five liquids (i.e., RA1, emulsified RA1, RA2, emulsified RA2, and water) onto the asphalt binder-coated substrates, and then observing this drop in cross section, where the angle between the baseline of the drop and the tangent at the drop boundary was measured (Figure 7).

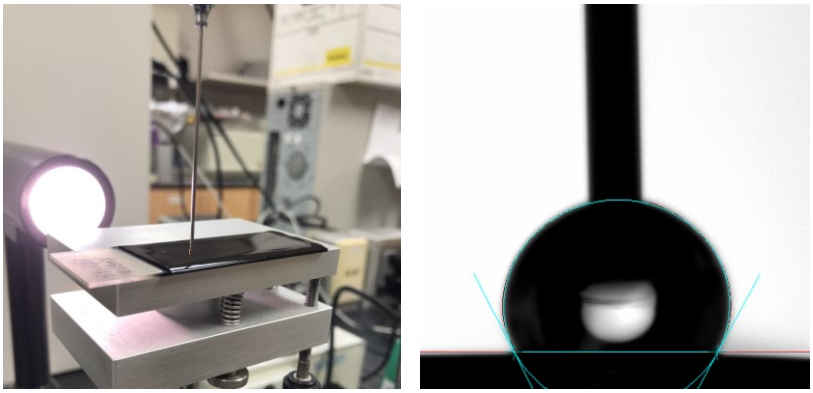


Figure 7. Sessile Drop Testing of Rejuvenators and Emulsified Rejuvenators

The preparation of asphalt binder-coated substrates for Sessile Drop testing is briefly discussed as follows. The glass slide surface used as a substrate for the asphalt binder was first degreased with acetone to remove moisture and dust. The asphalt binder was heated in an oven at 150°C (302°F) and stirred in the container before pouring a small quantity onto the glass slide surface. The quantity of asphalt binder poured was sufficient to form an area of approximately 25 mm × 75 mm in size (Figure 8). The glass slide surface covered with the binder sample was then allowed to cool to room temperature. For each of the five liquids for Sessile Drop testing (i.e., RA1, emulsified RA1, RA2, emulsified RA2, and water), an asphalt binder-coated substrate was prepared and only tested once.

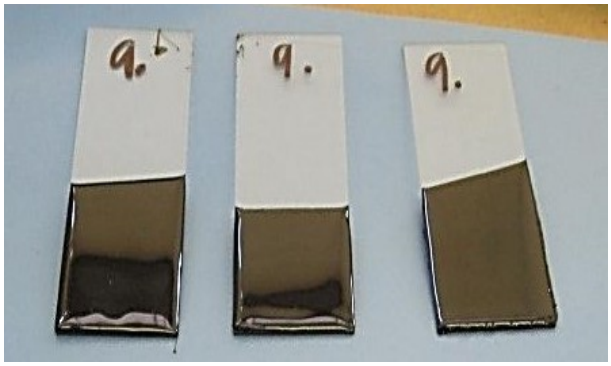


Figure 8. Preparation of Asphalt Binder-Coated Substrates for Sessile Drop Testing of Rejuvenators and Emulsified Rejuvenators

The Sessile Drop test procedure consists of the following steps: 1) Place the asphalt binder-coated substrate between the equipment light source and camera; 2) Rinse the micro syringe used for the probe liquid disposal with the liquid under evaluation; 3) Position the tip of the micro syringe needle approximately 5 mm away from the top of the sample; 4) Dispense a small drop of the probe liquid from the syringe. As more volume of the probe liquid is added, the drop on the asphalt binder surface will expand to a point where its interfacial boundary with the asphalt binder surface starts to expand. Stop adding the probe liquid at this point and capture an image of the drop using the equipment camera; 5) Analyze each image to obtain two contact angles (i.e., the left and right angles); and 6) For each asphalt-liquid interface (i.e., combination of the asphalt binder substrate and the liquid under evaluation), collect the contact angle for a minimum of three replicates and report the average of the left and right contact angles for each replicate.

2.2.2 Characterization of the Viscosity of Asphalt Rejuvenators

Viscosity is an important property of asphalt rejuvenators for foaming and emulsion applications. For foaming, the rejuvenator must be thin enough to be uniformly applied through the spray nozzle and viscous enough to trap air bubbles for volume expansion. For the emulsion application, the viscosity may affect the mixability and resulting thickness of the asphalt binder film on the aggregate surface. In this study, the viscosity of rejuvenators was evaluated at three testing temperatures [i.e., 60°C (140°F), 110°C (230°F), and 130°C (266°F)] using the Brookfield rotational viscometer (AASHTO T316) with spindle SC4-18. To assess durability and aging susceptibility, the viscosity of the rejuvenators was measured considering four aging conditions: unaged, short-term aged in the RTFO (AASHTO T 240),

RTFO plus 20-hour PAV aging (AASHTO R 28) at 100°C (212°F), and RTFO plus 40-hour PAV aging at 100°C (212°F). All the viscosity measurements were performed at 100 RPM, with an exception that the 60°C (140°F) viscosity of RA2 after RTFO plus 40-hour PAV aging was tested at 50 RPM. To evaluate the overall aging susceptibility of the two rejuvenators, an aging index was calculated following Equation 1.

$$Viscosity\ Aging\ Index\ @\ 60°C = \frac{Viscosity_{After\ RTFO\ plus\ 40-h\ PAV}}{Viscosity_{Unaged}} \quad \text{Equation 1}$$

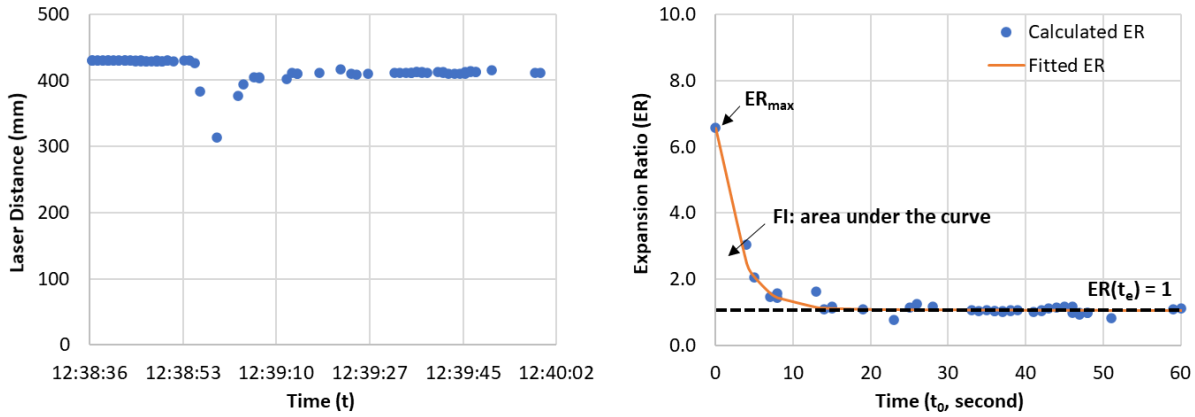
2.3 FOAMING CHARACTERIZATION OF REJUVENATORS AND REJUVENATED ASPHALT BINDERS

This section discusses the experimental plan of the *Foaming* experiment for determining the foaming characteristics of the rejuvenators and rejuvenated asphalt binders (i.e., virgin binders pre-blended with rejuvenators). The overall objective of this experiment was to optimize the foaming condition of the rejuvenator for RAP pretreatment applications, and the foaming condition of the rejuvenated asphalt binder for preparing foamed RAP mixtures. In this experiment, three foaming temperatures were considered for the rejuvenators: 110°C (230°F), 120°C (248°F), and 130°C (266°F). For the rejuvenated asphalt binders, the foaming temperatures assessed were 130°C (266°F), 140°C (284°F), and 150°C (302°F). At each foaming temperature, three water contents were evaluated: 1%, 2%, and 3%. These foaming conditions were selected based on existing literature on asphalt foaming for warm mix asphalt (WMA) applications (Newcomb et al., 2015; Yin et al., 2015) and NCAT's previous experience with foaming rejuvenators in the laboratory.

Foaming of the rejuvenators and rejuvenated asphalt binders was performed using the Wirtgen foamer, with a target of 200 g for each foaming application. Two repeatable and high-quality foaming measurements were collected for each condition, but tester discretion was used for the total number of foaming applications. For each foaming measurement, the height of the foam was continuously measured using a laser distance meter attached to the Wirgten foamer, as shown in Figure 9. The laser recorded real-time distance data and time and instantly transferred them to a computer via Bluetooth. These data [Figure 10(a)] were then processed to generate a volume expansion curve of the foam, as shown in Figure 10(b). Several quantitative foaming index parameters were then determined from the curve, which included maximum expansion ratio (ER_{max}), expansion half-life ($t_{1/2}$), and FI.

(a) (b)

Figure 9. Foaming Measurement Setup; (a) Wirtgen Foamer, (b) Laser Distance Meter



(a) (b)

Figure 10. Foaming Data Collection and Analysis; (a) Measured Laser Distance Data, (b) Volume Expansion and Collapse Curve after Data Processing

Expansion ratio (ER) is an index parameter to express the degree of volume expansion due to foaming and ER_{max} refers to the maximum volume expansion. $t_{1/2}$ is defined as the time required for the foam to collapse from its peak volume to half of it, which indicates the relative volume stability of the foam. Different from ER_{max} and $t_{1/2}$, FI considers the entire volume expansion and collapse behavior of the foam over time. It is defined as the area under the ER curve with a baseline ER value of 1. In general, higher ER_{max}, $t_{1/2}$, and FI values are desired for foam with greater volume expansion and stability. However, ER_{max} and $t_{1/2}$ often trend in opposite directions, as the foam with a high ER_{max} typically has poor stability and thus, tends to collapse at a fast rate with a low $t_{1/2}$. Previous research with asphalt foaming for WMA applications indicates that FI is a more robust parameter for characterizing the overall quality of foam than ER_{max} and $t_{1/2}$ (Newcomb et al., 2015). Therefore, in this study, FI was used as the primary index parameter to optimize the foaming conditions of rejuvenators and rejuvenated asphalt binders.

The processing of the laser distance data for calculating the quantitative foaming index parameters is briefly described as follows. First, for each distance data point in Figure 10(a), its corresponding ER was calculated using Equation 2.

$$ER(t) = \frac{h_{initial} - h(t)}{h_{initial} - h_{final}} \quad \text{Equation 2}$$

Where, *ER(t)* = ER at time t; $h_{initial}$ = initial height of the foam; *h(t)* = height of the foam at time t; and h_{final} = final height of the foam.

The highest ER value obtained by iterating Equation 2 through all the laser distance data points was selected as the ER_{max}, representing the maximum volume expansion. All the post-ER_{max} data was then fitted with an exponential function expressed in Equation 3. Figure 10(b) presents a curve fitting example of the post-ER_{max} data for illustration purposes.

$$ER(t_0) = ae^{-b(t_0)} + ce^{-d(t_0)} \quad \text{Equation 3}$$

Where, t_0 = time in second since ER_{max} occurs; and *a*, *b*, *c*, and *d* = fitting coefficients.

After determining the fitting coefficients of Equation 3, $t_{1/2}$ was then calculated by setting the left side of the equation, $ER(t_0)$, to half of the ER_{max}. Finally, FI was calculated by mathematically integrating Equation 3 to determine the area under the $ER(t_0)$ curve with a baseline ER value of 1, up to the time when the laser distance measurement was terminated (t_e). The calculation of FI is shown in Equation 4.

$$FI = \int_0^{t_e} [ER(t_0) - 1]dt = \int_0^{t_e} (ae^{-bt} + ce^{-dt} - 1)dt \quad \text{Equation 4}$$

2.4 PRETREATMENT AND MARINATION OF RAP WITH REJUVENATORS

This section discusses the experimental plan of the *RAP Pretreatment* and *RAP Marination* sub-experiments for evaluating the impacts of adding rejuvenators for RAP pretreatment and marination on the RAP quality characteristics. Note that only the RAP from mix design A (i.e., RAP A) and RA1 were evaluated for these efforts.

2.4.1 Pretreatment of RAP with Rejuvenators

The objective of the *RAP Pretreatment* sub-experiment was to characterize the quality properties of untreated and pretreated RAP using different rejuvenator application methods: spray-on, emulsion, and foaming. These methods used the same "effective" rejuvenator dosage of RA1 at 0.85% by weight of the RAP, which corresponded to 16.1% by weight of the RAP binder as discussed in Section 2.1. Prior to pretreatment with the rejuvenator, the RAP was dried and split to maintain homogeneity. Pretreatment

of the RAP was conducted using the Wirtgen pugmill, as shown in Figure 11. Approximately 20,000g to 30,000g of RAP was used for each pretreatment trial.

Figure 11. The Wirtgen Pugmill used to Pretreat RAP with Rejuvenators

For the spray-on pretreatment method, a spray bottle was used to add the rejuvenator. While the pugmill was running, the rejuvenator was sprayed on the RAP through the opening on the top cover of the pugmill. The pugmill was stopped after 120 seconds of mixing. For the emulsion method, the emulsified rejuvenator was poured on top of the RAP in the pugmill in a "zig-zag" motion. After that, the pugmill was kept running for 120 seconds. For the foaming method, the Wirtgen foamer was used to apply the foamed rejuvenator. Foaming was conducted at the optimum foaming condition that yielded the best foam quality with the highest FI value. The foamed rejuvenator was introduced to the RAP through the foaming nozzle of the Wirtgen foamer after approximately 10 seconds of running the pugmill. The mixing process was kept at 120 seconds to be consistent with the other two pretreatment methods.

After pretreatment, the RAP was tested for quality characterization using the DWT and grayscale-based image analysis. Moisture content of the pretreated RAP was also measured to determine the amount of water introduced with the emulsion and foaming pretreatment methods. Because the emulsified rejuvenator contained a plentiful amount of water (40% for the emulsified RA1), additional moisture content measurements were conducted for the pretreated RAP using the emulsion method for up to 7 days of room-temperature storage after pretreatment.

The DWT was conducted in the *RAP Pretreatment* sub-experiment to evaluate the workability of the untreated and pretreated RAP using different rejuvenator application methods. Prior to DWT testing, the RAP was preheated to the target test temperature and a 4,200g sample was placed into a Superpave Gyratory Compactor (SGC) mold. The test was performed in the SGC by applying force to the RAP, without gyrating, at a constant loading rate at 0.05 mm/second until a stress of 700 kPa was reached. During the test, the applied force and specimen height were recorded every 0.1 seconds. The workability of the RAP was evaluated based on the slope of the Stress vs. Volumetric Strain curve. The DWT value is the test parameter, which is defined as the slope of the curve at 600 kPa (Figure 12) and is

calculated as the ratio of the change in stress between 650 kPa and 550 kPa pressure to the change in volumetric strain in the same pressure range (Equation 5). Existing studies have shown that DWT is able to discriminate different RAP sources (Dongre et al., 2020). In general, a higher DWT value indicates better overall RAP quality in terms of workability and compactability. The DWT test was conducted at 116°C (240°F) and 149°C (300°F) with two replicates at each temperature.

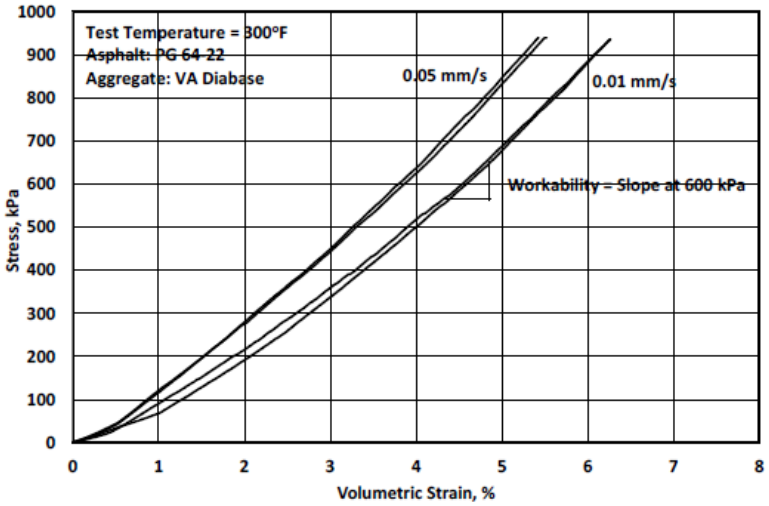


Figure 12. Determination of DWT Value based on the SGC Stress vs. Volumetric Strain Curve (Dongre et al., 2013)

$$DWT\ Value = \frac{\sigma_{650} - \sigma_{550}}{\varepsilon_{650} - \varepsilon_{550}} \qquad \text{Equation 5}$$

Where, σ_{650} and σ_{550} are the measured normal stress at the nearest index to 650 kPa and 550 kPa pressure, respectively; and ε_{650} and ε_{550} are the volumetric strain (%) at the nearest index to 650 kPa and 550 kPa pressure, respectively.

For grayscale-based image analysis, approximately 2,000g of the untreated or pretreated RAP sample were first scanned using a tabletop office scanner, as shown in Figure 13. A wood block frame was used to maintain a uniform thickness of the RAP sample for each scanning trial. A paper cover was placed on top of the block frame to prevent external light from interfering with the scan. Figure 14 presents the scanned image of a RAP sample for illustration purposes. The image was then processed for grayscale pixel analysis using MATLAB. The analysis was programed by assigning a grayscale value of zero (0) to pixels that were fully black and a value of 250 to pixels that were fully white. The distribution of the grayscale values for the scanned image were then computed and analyzed graphically, as shown in Figure 14. For RAP quality comparison purposes, a distribution curve with a small average grayscale value and a narrow distribution is desired as it indicates that the RAP sample has an overall darker color and thus, is expected to have more "activated" RAP binder and that it is more consistent. This grayscale-based image analysis effort was inspired by Swiertz et al. (2012) and Ling et al. (2014) on the coating and moisture susceptibility evaluations of cold-mix asphalt mixtures.

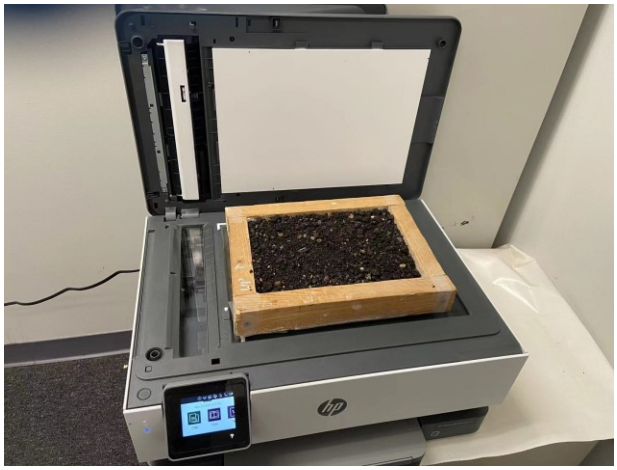


Figure 13. Apparatus used to Scan RAP for Grayscale-based Image Analysis

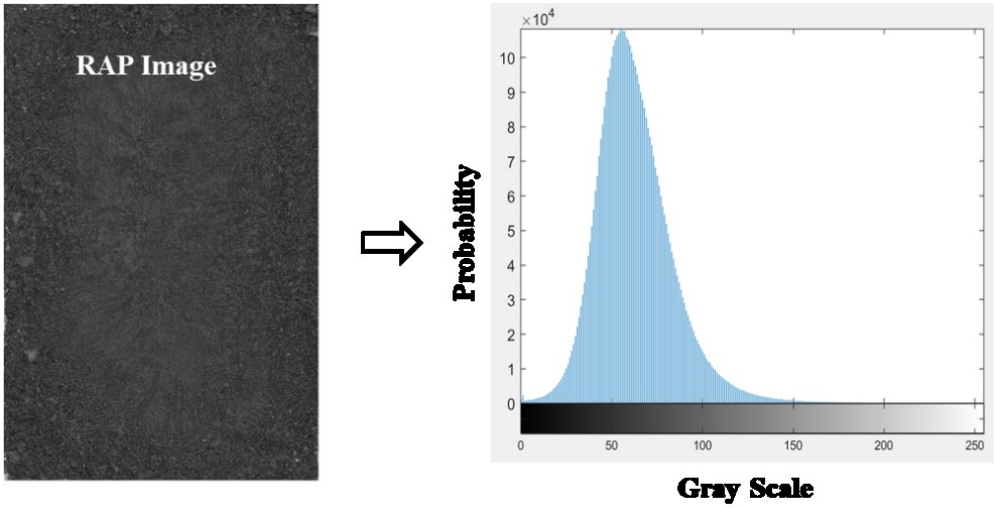


Figure 14. Conversion of Scanned RAP Image to Grayscale Distribution Curve

2.4.2 Marination of Pretreated RAP with Rejuvenator

From the results of the *RAP Pretreatment* sub-experiment, the best-performing method of pretreatment (i.e., emulsion pretreatment) was selected for further evaluation in the *RAP Marination* sub-experiment. The objective of the marination sub-experiment was to determine the impact of marination on the quality characteristics of the pretreated RAP with the rejuvenator. A total of four marination conditions were assessed. Two used an accelerated marination approach, which required marinating the pretreated RAP at 135°C (275°F) for 1.5 hours and 3 hours to simulate a scenario where the pretreated RAP would be marinated at an elevated temperature on the day of mixture production. The accelerated, elevated-temperature marination approach has been successfully used in batch plant operations by asphalt contractors in Japan (West and Copeland, 2015). The other two marination conditions used an extended, ambient-temperature marination approach. This required marinating the pretreated RAP in the laboratory without temperature control for 3 days and 7 days to simulate a scenario where the

pretreated RAP would be marinated at ambient temperatures for several days prior to mixture production.

After each marination condition, the pretreated RAP with the rejuvenator was tested with the DWT and grayscale-based image analysis for quality characterization using the same test and analysis procedures as the *RAP Pretreatment* sub-experiment. Asphalt binders were then extracted and recovered from the pretreated RAP samples with no marination, 3-hour marination at 135°C (275°F), and 7-day marination at room-temperature, and were then tested to characterize their rheological and chemical properties. The extracted binder testing plan for the *RAP Marination* sub-experiment is discussed as follows.

Superpave PG and ΔT_c Parameter: The performance grades of the extracted RAP binders with different marination conditions were determined following AASHTO T315 (M320) with an exception that the binders were tested as extracted and recovered without additional RTFO or PAV aging. Furthermore, the ΔT_c was determined based on the BBR results, where ΔT_c is defined as the numerical difference between the low continuous grade temperatures determined from the BBR stiffness criterion of 300 MPa and the m-value criterion of 0.3 (Anderson et al., 2011). The ΔT_c parameter has recently been used to assess the loss of stress relaxation properties of asphalt binders. Generally, a more positive (or less negative) ΔT_c value is desirable for asphalt binders with better ductility and block cracking resistance.

Multiple Stress Creep Recovery (MSCR): The MSCR test per AASHTO T350 (M332) was used to evaluate the elastic response and rutting resistance of the extracted RAP binders. The test was conducted at 64°C (147°F) on as-recovered binder samples without additional laboratory aging. The test applied 20 loading cycles at a low stress level of 0.1 kPa followed by 10 cycles at a high stress level of 3.2 kPa. Each loading cycle consisted of 1 second of creep and 9 seconds of recovery. For data analysis, the strain responses were utilized to calculate the percent recovery (%R) and non-recoverable creep compliance (J_{nr}) using Equation 6 and Equation 7, respectively. A higher %R value and a lower J_{nr} value indicate better binder elasticity and rutting resistance, respectively.

$$\%R = \frac{\varepsilon_r}{\varepsilon_r + \varepsilon_{nr}} * 100\% \qquad \text{Equation 6}$$

Where, ε_r = recoverable strain; and ε_{nr} = non-recoverable strain.

$$J_{nr} = \frac{\varepsilon_{nr}}{\sigma} \qquad \text{Equation 7}$$

Where, σ = creep stress.

Glover-Rowe (G-R) Parameter: The *G-R* parameter was utilized to evaluate the ductility and block cracking potential of the extracted RAP binders with different marination conditions. To determine the *G-R* parameter, the DSR frequency sweep test was conducted at multiple test temperatures over an angular frequency range of 0.1 to 10 rad/s. During the test, the peak-to-peak strain of the binder sample was controlled at one percent to ensure its behavior remained in the linear viscoelastic range. For data

analysis, the RHEA software was used to construct a limited DSR master curve by fitting the shear complex modulus (|G*|) and phase angle (δ) data to the discrete relaxation and retardation spectra (Baumgaertel and Winter, 1989). Then, the binder |G*| and δ at 15°C (59°F) and 0.005 rad/s were determined, from which the *G-R* parameter was calculated using Equation 8. In general, a high *G-R* parameter indicates low ductility and high susceptibility to block cracking.

$$G - R\ Parameter = \frac{|G^*|\ cos(\delta)^2}{sin(\delta)} \qquad \text{Equation 8}$$

Where, |G*| = binder shear complex modulus at 15°C (59°F) and 0.005 rad/s; and δ = binder phase angle at 15°C (59°F) and 0.005 rad/s.

SARA Fractions: For the extracted RAP binders with different marination conditions, the asphaltenes were determined as n-heptane insoluble following ASTM D3279. The maltenes fraction (i.e., saturates, aromatics, and resins) were then determined by thin layer chromatograph measured by means of flame ionization detector (TLC-FID) via Iatroscan. The colloidal stability of the extracted binders was determined based on the Gaestel "Colloidal Instability Index" (known as CII or I_c), as shown in Equation 9 (Gaestel et al., 1971). As the CII increases, the colloidal stability of the asphalt binder decreases.

$$CII = \frac{Asphaltenes+Saturates}{Aromatics+Resins} \qquad \text{Equation 9}$$

2.5 ASPHALT MIXTURE PERFORMANCE TESTING

This section discusses the experimental plan of the *Mixture Performance Testing* experiment for evaluating the workability and cracking resistance of high-RAP mixtures prepared with different rejuvenator application methods. Mixture workability evaluation was based on the DWT and the evaluation of cracking resistance was conducted using the IDEAL-CT and DCT. The DWT test followed the same procedure as used in the *RAP Pretreatment* and *RAP Marination* sub-experiments, except that the test was conducted on the loose asphalt mixture instead of the RAP. Prior to DWT testing, the loose mixture was conditioned for 2 hours at the compaction temperature following the short-term aging procedure for volumetric mix design per AASHTO R 30. The test was conducted at 116°C (240°F) and 149°C (300°F) with two replicates at each temperature. A 4,800g loose mixture sample was used for each replicate. The test parameter is the DWT value, where a higher value is desired for better mixture workability.

The IDEAL-CT per ASTM D8225 was used to determine the intermediate-temperature cracking resistance of asphalt mixtures. To consider the impact of mixture aging, the test was conducted on specimens that were short-term aged for 4 hours at 135°C (275°F) per AASHTO R 30 followed by long-term aging for 6 additional hours at 135°C (275°F) prior to compaction. During the test, a monotonic load was applied along a gyratory specimen with 62mm height and 7.0±0.5% air voids at a constant displacement rate of 50 mm/min. The test was performed at 25°C (77°F) with a minimum of four

replicates and all the replicates were conditioned in a temperature chamber at 25°C (77°F) for two hours prior to testing. Figure 15 shows the IDEAL-CT test device and specimen setup. For data analysis, the load-displacement curve was analyzed to determine the work of fracture and the slope of the curve at 25% reduction from the peak load, among other interim parameters. The final test parameter, cracking tolerance index (CT_{index}), was then calculated using Equation 10. A higher CT_{index} value is desired for better intermediate-temperature cracking resistance.

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

Where, t = specimen thickness; l_{75} = post-peak displacement at 75% of peak load; D = specimen diameter; G_f = fracture energy; and $|m_{75}|$ = absolute value of the post-peak slope at 75% of peak load.

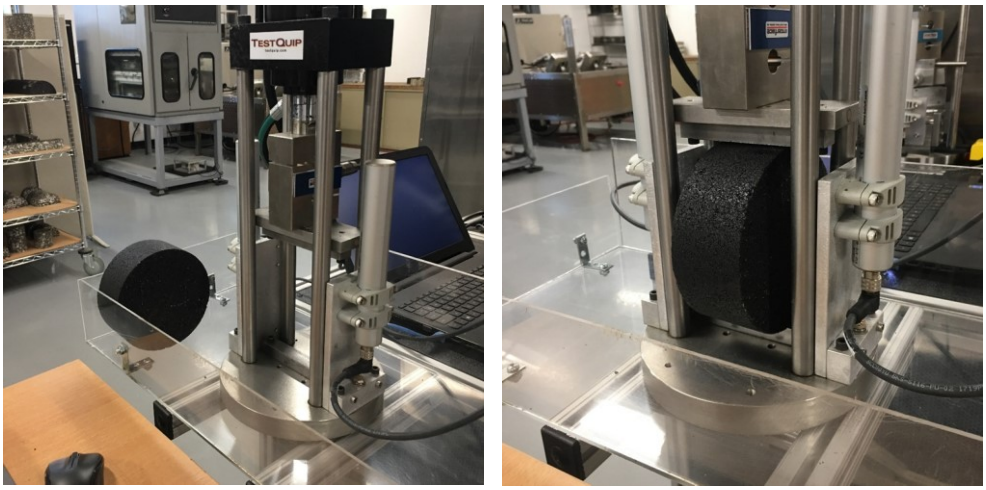


Figure 15. IDEAL-CT Test Device and Specimen Setup

The DCT test per ASTM D 7313 was used to assess the low-temperature cracking resistance of asphalt mixtures. Same with the IDEAL-CT, the DCT test was conducted on specimens that were short-term aged for 4 hours at 135°C (275°F) followed by long-term aging for 6 additional hours at 135°C (275°F) to consider the impact of mixture aging. The test was conducted at -18°C (0°F) with six replicates. The DCT specimen was cut from a 160 mm tall gyratory sample and then trimmed to possess a flat edge on one side of the specimen for instrumentation gage points, a 62.5 ± 5.0 mm notch down the center of the specimen from the flat edge, and two 1-inch diameter holes on each side of the notch. Prior to testing, the DCT specimen was loaded in tension by metal rods that were inserted through the specimen core holes. A clip gage was then installed over the crack mouth prior to the start of the test to control and record the crack mouth opening displacement (CMOD). The test was conducted in a CMOD control mode with the clip gage opening at a constant rate of 0.017 mm/sec. The test was terminated when the load dropped below 0.1 kN. Figure 16 shows the DCT test equipment and specimen setup. For data analysis, the fracture energy (G_f) was calculated using Equation 11, where the area under the load-CMOD curve was determined through numerical integration using the trapezoid rule. A higher G_f value is desired for better resistance to low-temperature cracking.

$$G_f = \frac{Area}{B*(W-a)}$$ Equation 11

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).

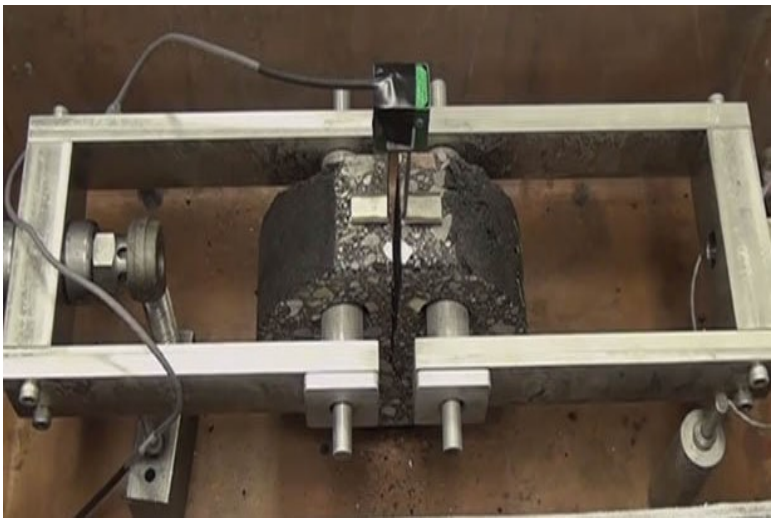


Figure 16. DCT Test Device and Specimen Setup

CHAPTER 3: CONTACT ANGLE AND VISCOSITY RESULTS OF REJUVENATORS

This chapter presents the test results and data analysis of the *Rejuvenator Characterization* experiment. The objective of the experiment was to characterize the wettability and viscosity of the two rejuvenators evaluated in this study (i.e., RA1 and RA2). For the wettability evaluation, the Sessile Drop test was conducted to measure the contact angle of RA1 and RA2, in both their original and emulsion forms, as well as water on two asphalt binder-coated solid substrates. The Rotational Viscosity test was conducted to measure the viscosity of RA1 and RA2 at various aging conditions.

3.1 SESSILE DROP TEST RESULTS

Figure 17 presents the contact angle results of RA1 and RA2 from the Sessile Drop test, where the error bars represent one standard deviation of the replicate measurements. For each asphalt-liquid interface (i.e., each combination of asphalt binder substrate and liquid of investigation), a minimum of three replicate measurements were collected. As shown, the two rejuvenators in both their original and emulsified forms showed good wetting properties (i.e., $\theta < 90°$) with both the PG 58S-28 and PG 67-22 binder. As expected, water showed poor wetting properties ($\theta > 90°$) with the asphalt binder substrates, since asphalt binders are hydrophobic (water-repellent) in nature. When comparing the two rejuvenators, RA1 showed slightly better wetting properties (as indicated by lower contact angles) than RA2 when tested with both binders. When comparing the emulsified rejuvenators, the emulsified RA1 showed better wettability with the PG 58S-28 binder than the emulsified RA2; while for the PG 67-22 binder, the two emulsified rejuvenators had similar wettability when considering the variability of the test. The difference in the wettability between the two rejuvenators could be one of the factors contributing to the different foaming characteristics observed in the *Foaming* experiment (Chapter 4).

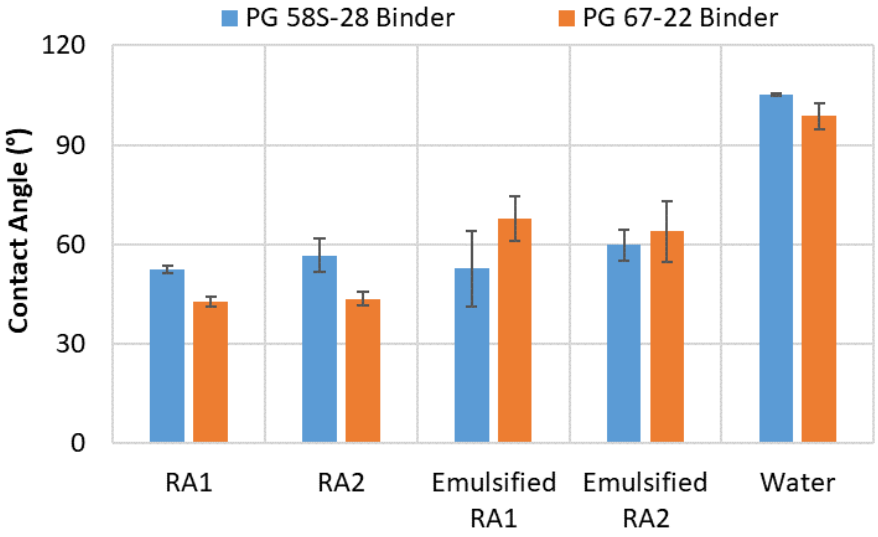


Figure 17. Sessile Drop Test Results for PG 58S-28 and PG 67-22 Asphalt Binders with Two Rejuvenators, Two Emulsified Rejuvenators, and Water

It is important to mention that the observed narrow range of variation among the contact angle results for the evaluated asphalt-liquid combinations (Figure 17) is not unique to this study. Other studies have reported similar observations (Bhasin, 2006; Little and Bhasin, 2006; Moraes, 2011). Despite this narrow range, the two rejuvenators had notably different contact angle values when tested with the PG 58S-28 binder versus the PG 67-22 binder, which highlighted the importance of binder source/composition on the rejuvenator's surface wetting characteristics.

3.2 ROTATIONAL VISCOSITY TEST RESULTS

Figure 18 presents the rotational viscosity results of RA1 and RA2 at 60°C (140°F), 110°C (230°F), and 130°C (266°F). All the viscosity measurements were performed at 100 RPM except that the 60°C (140°F) viscosity of RA2 after RTFO plus 40-hour PAV aging was measured at 50 RPM. As expected, the viscosity of the two rejuvenators decreased as temperature increased from 60°C (140°F) towards 130°C (266°F). At each temperature, an increase in viscosity was observed with aging (from RTFO towards RTFO plus 40-hour PAV aging) for both rejuvenators, but RA2 showed significantly higher viscosity values after RTFO plus 20-hour and 40-hour PAV aging. Furthermore, RA2 had a higher viscosity aging index at 60°C (140°F) than R1 (14.2 versus 1.6), which indicated it was relatively more susceptible to oxidative aging. This difference in the aging susceptibility of the two rejuvenators could be attributed to their different chemical compositions.

Figure 19 presents the visual observation of RA1 and RA2 at various aging conditions. For both rejuvenators, a change in color was observed as the oxidative aging progressed (from unaged towards RTFO plus 40-hour PAV aging), although a more pronounced color change was noted for RA2 from RTFO plus 20-hour PAV aging to RTFO plus 40-hour PAV aging. This greater change in appearance (i.e., color) after extended PAV aging could be an indication that RA2 was more susceptible to long-term oxidation than RA1, which agreed with the viscosity aging index results in Figure 19.

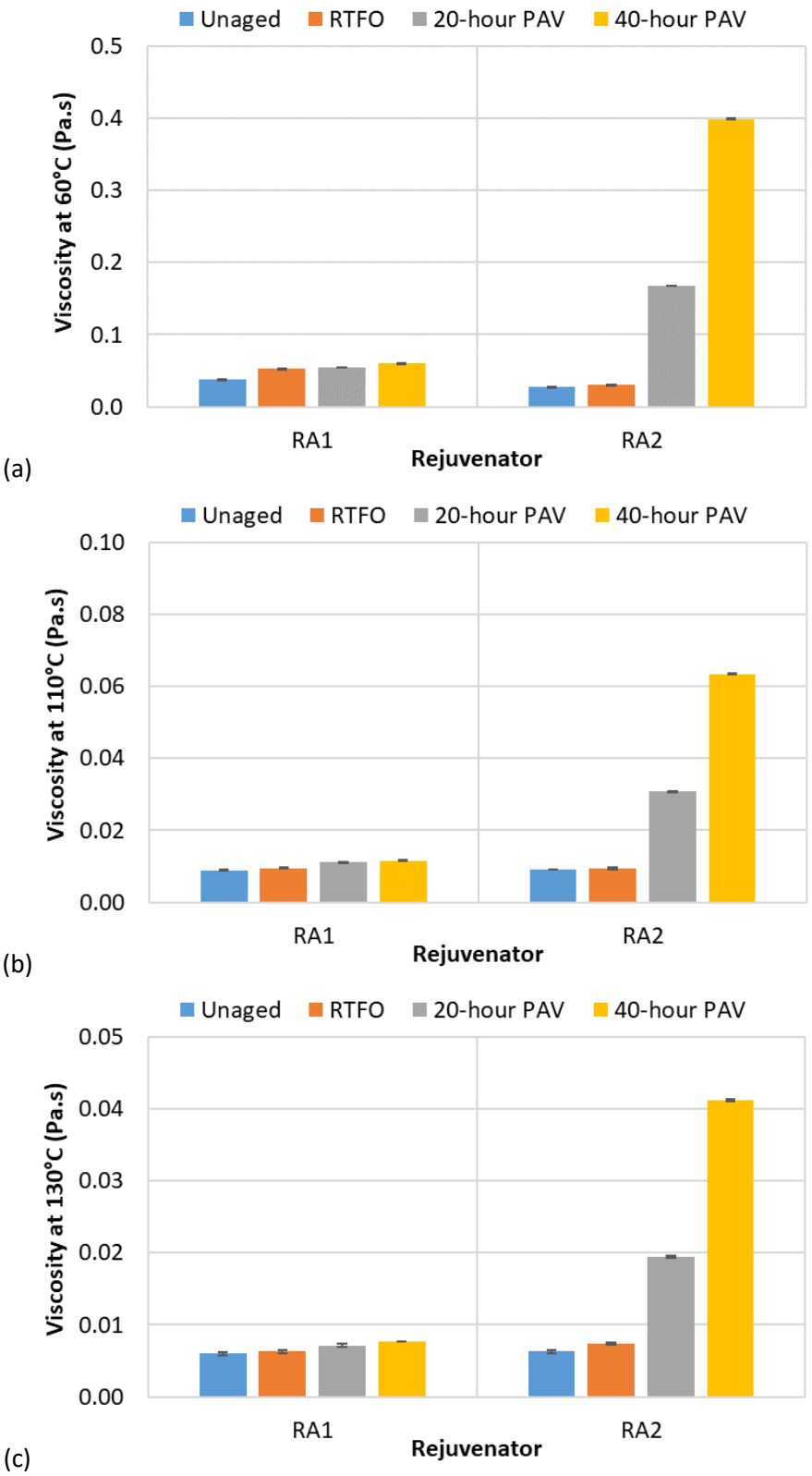


Figure 18. Rotational Viscosity Results of RA1 and RA2 at Various Aging Conditions; (a) 60°C (140°F), (b) 110°C (230°F), and (c) 130°C (266°F)

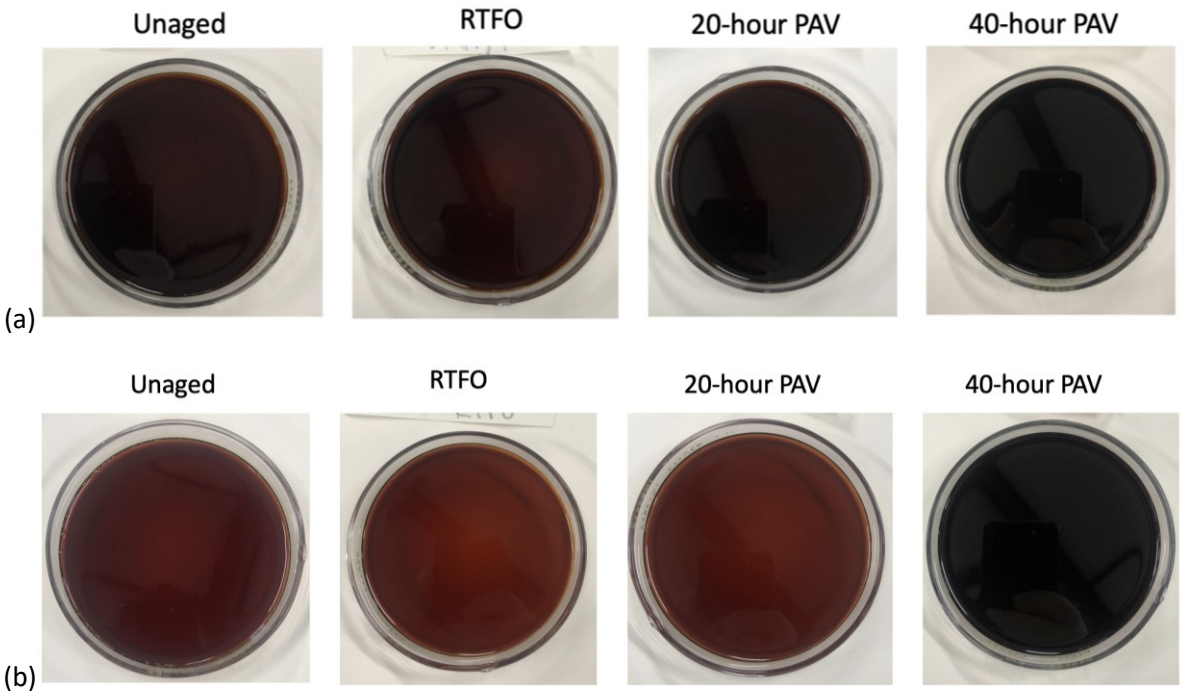


Figure 19. Visual Observation of Two Rejuvenators at Various Aging Conditions; (a) RA1, (b) RA2

CHAPTER 4: FOAMING CHARACTERISTICS OF REJUVENATORS AND REJUVENATED ASPHALT BINDERS

This chapter discusses the test results and data analysis of the *Foaming* experiment. The objective of the experiment was to determine the foaming characteristics and optimize the foaming conditions of the two rejuvenators (i.e., RA1 and RA2) and the two rejuvenated asphalt binders (i.e., rejuvenated PG 67-22 binder with RA1 and rejuvenated PG 58S-28 binder with RA2) evaluated in the study. The foaming measurements for the two rejuvenators were conducted at 110°C (230°F), 120°C (248°F), and 130°C (266°F), while those for the two rejuvenated asphalt binders were conducted at 130°C (266°F), 140°C (284°F), and 150°C (302°F). Each foaming temperature was evaluated with three water contents: 1%, 2%, and 3%. As discussed in Section 2.3, the assessment of foaming characteristics focused primarily on the FI parameter as it incorporated the concepts of ER and $t_{1/2}$. In general, a higher FI indicates higher volume expansion and better overall foam stability.

4.1 FI RESULTS OF REJUVENATORS

Figure 20 presents the average FI results of RA1 at various foaming conditions. For foaming at 120°C (248°F) and 130°C (266°F), the FI increased as the water content increased. However, the opposite trend was observed for the FI results at 110°C (230°F), which indicated that the overall foam quality decreased as the water content increased. When considering all the foaming conditions, RA1 exhibited the highest FI at 120°C (248°F) and 3% water content. Therefore, this temperature and water content combination was selected as the optimum foaming condition for RA1. At this condition, the rejuvenator had an average ER_{max} of 6.3 and an average $t_{1/2}$ of 8.8 seconds.

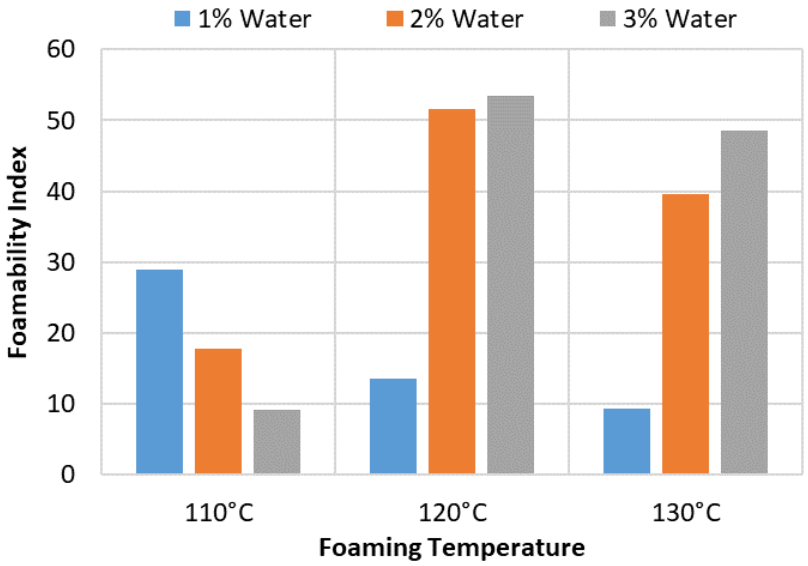


Figure 20. Foamability Index Results of RA1 at Various Foaming Conditions

Figure 21 presents the average FI results of RA2. This rejuvenator had no volume expansion when foamed at 110°C (230°F) and 1% water content; thus, the corresponding FI data was not available and is labeled as "N/A" in the figure. Overall, the FI of RA2 increased as the temperature and water content increased, which indicated improved foaming characteristics. Among all the foaming temperature and water content combinations, 130°C (266°F) and 3% water content yielded the highest FI value of 46.7, which was selected as the optimum foaming condition for RA2. At this condition, the rejuvenator had an average ER_{max} of 7.9 and an average $t_{1/2}$ of 5.7 seconds. Compared to RA1, RA2 had higher instantaneous volume expansion but lower foam stability when foamed at their corresponding optimum conditions. This difference could be partially attributed to the different wetting properties of the two rejuvenators discussed in Section 3.1.

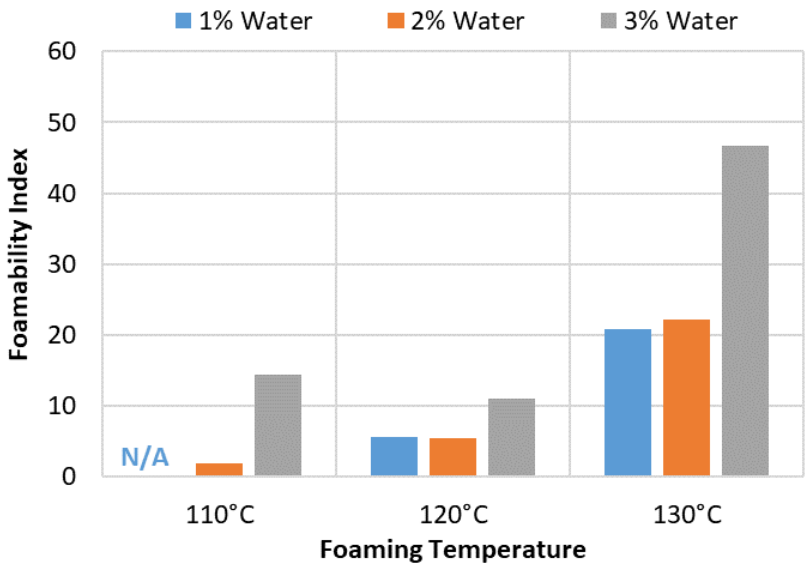


Figure 21. Foamability Index Results of RA2 at Various Foaming Conditions

4.2 FI RESULTS OF REJUVENATED ASPHALT BINDERS

Figure 22 presents the average FI results of the rejuvenated PG 67-22 binder with RA1 at various foaming conditions. This rejuvenated binder exhibited comparable foaming characteristics at different water contents when foamed at 130°C (260°F). However, its foaming characteristics at 140°C (284°F) and 150°C (302°F) varied greatly depending on the water content. At both temperatures, the FI increased significantly as the water content increased from 1% to 2%, which indicated better foaming characteristics. As the water content further increased to 3%, the FI for foaming at 140°C (284°F) reduced by approximately 45% while that for foaming at 150°C (302°F) only had a slight reduction. Ultimately, 150°C (302°F) and 2% water content was selected as the optimum foaming condition for the rejuvenated PG 67-22 binder with RA1 as it yielded the highest FI of 127.6 with an average ER_{max} of 11.1 and an average $t_{1/2}$ of 3.4 seconds.

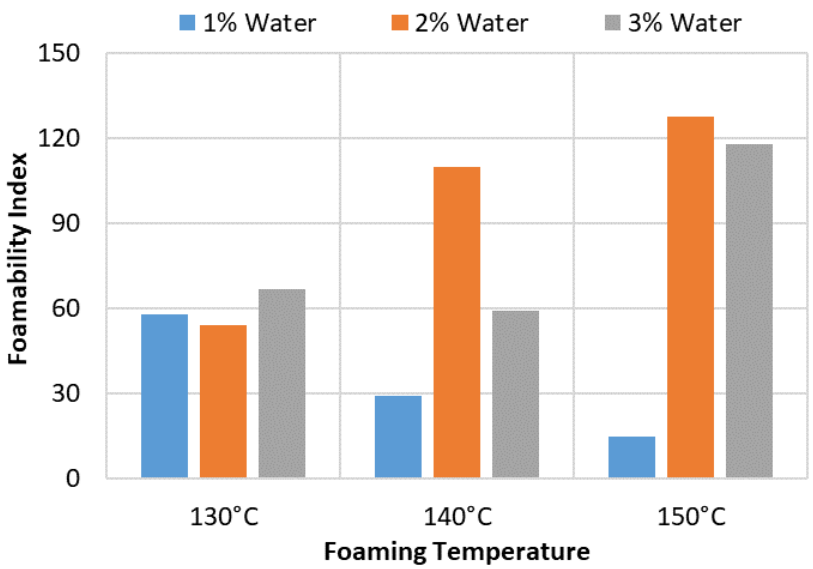


Figure 22. Foamability Index Results of Rejuvenated PG 67-22 Binder with RA1 at Various Foaming Conditions

Figure 23 presents the average FI results of the rejuvenated PG 58S-28 binder with RA2. Overall, this rejuvenated binder showed good foaming characteristics, with FI varying from approximately 90 to 120 among all the foaming conditions. Both temperature and water content did not appear to have a significant effect on the FI results. 150°C (302°F) and 2% water content yielded the highest FI of 117.5 and thus, was selected as the optimum foaming condition for the rejuvenated PG 58S-28 binder with RA2. The corresponding average ER_{max} and $t_{1/2}$ at this condition was 15.3 and 3.5 seconds, respectively.

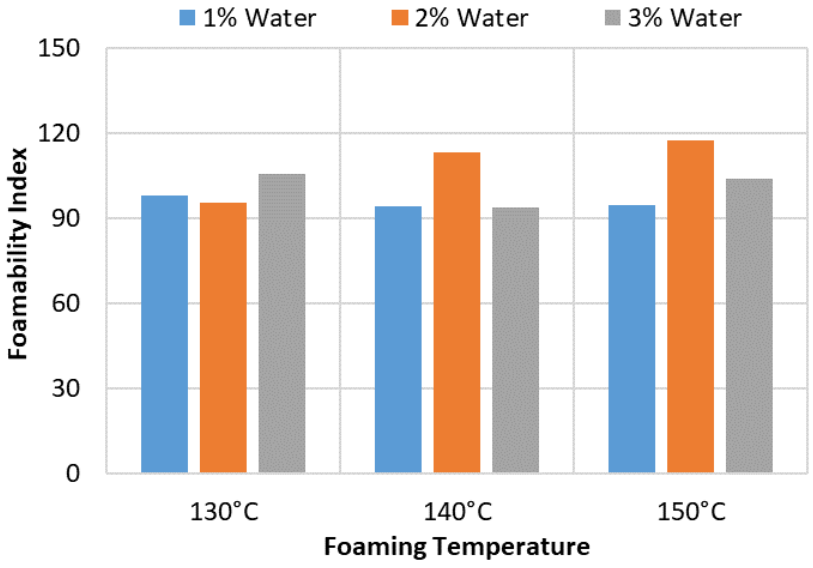


Figure 23. Foamability Index Results of Rejuvenated PG 58S-28 Binder with RA2 at Various Foaming Conditions

4.3 SUMMARY

Based on the FI results discussed in Figure 20 through Figure 23, the optimum foaming conditions for the two rejuvenators and the two rejuvenated asphalt binders were selected as follows: 120°C (248°F) and 3% water content for RA1; 130°C (266°F) and 3% water content for RA2; and 150°C (302°F) and 2% water content for both the rejuvenated PG 67-22 binder with RA1 and the rejuvenated PG 58S-28 binder with RA2. These foaming conditions were further evaluated in the *RAP Pretreatment*, *RAP Marination*, and *Mixture Performance Testing* experiments, which are discussed in Chapter 4, 5, and 6, respectively.

CHAPTER 5: QUALITY CHARACTERISTICS OF PRETREATED RAP WITH REJUVENATORS

This chapter discusses the test results and data analysis of the *RAP Pretreatment* sub-experiment. The objective of the experiment was to evaluate the quality characteristics of pretreated RAP [using the RAP from mix design A and RA1] with different rejuvenator application methods: spray-on, emulsion, and foaming. All the pretreatment methods used the same "effective" rejuvenator dosage of 16.1% by weight of the RAP binder, which was selected by the rejuvenator supplier to target 76°C as the high-temperature PG of the extracted RAP binder after adding the rejuvenator. The experimental plan for RAP quality characterization included 1) DWT testing to evaluate the workability of untreated and pretreated RAP with rejuvenators, 2) moisture content measurements to determine the impact of different rejuvenator application methods on the moisture content of the RAP, and 3) image analysis to quantify the grayscale-based color contrast distribution of untreated and pretreated RAP with rejuvenators.

5.1 DWT RESULTS OF UNTREATED AND PRETREATED RAP

Figure 24 presents the DWT results of the untreated and pretreated RAP with different rejuvenator application methods at 116°C (240°F) and 149°C (300°F). The error bars represent one plus and minus standard deviation of the replicate measurements. The DWT results at both test temperatures showed that the three pretreatment methods improved the workability of the RAP, as indicated by higher DWT values for the pretreated samples than the untreated (control) sample. Furthermore, the DWT values of all the RAP samples increased as the test temperature increased from 116°C (240°F) to 149°C (300°F), which indicated improved workability. It was hypothesized that this improvement in workability was due to the reduced viscosity of the RAP binder and the possibility that more RAP binder became "activated", which resulted in higher RAP binder availability at a higher temperature. Among the three pretreatment methods, the emulsion method had the highest DWT values at both test temperatures, followed by the spray-on and foaming methods, respectively.

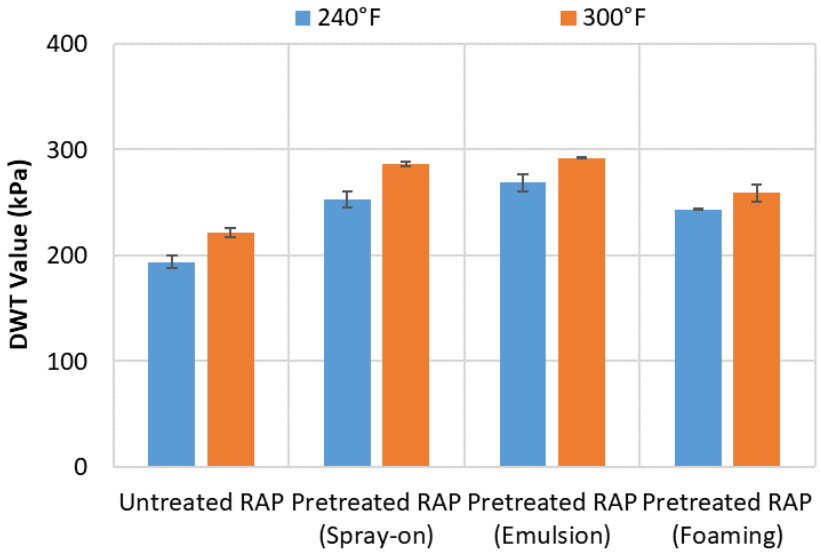


Figure 24. DWT Results of Untreated and Pretreated RAP with Different Rejuvenator Application Methods

5.2 MOISTURE CONTENT RESULTS OF UNTREATED AND PRETREATED RAP

Figure 25 presents the moisture contents of the untreated and pretreated RAP samples immediately after pretreatment. As shown, adding rejuvenators using the spray-on and foaming methods did not change the moisture content of the RAP, as the two pretreated samples had the same moisture content (i.e., 0.13%) as the untreated sample. Adding the emulsified rejuvenator, however, increased the moisture content of the RAP to 0.53%, which was due to the inclusion of water in the emulsified rejuvenator product (i.e., 40% by volume for RA1). For the pretreated RAP using the emulsion method, additional moisture content measurements were conducted for up to seven days of storage at room temperature when the sample was kept in large flat pans. As shown in Figure 26, the moisture content of the pretreated RAP with the emulsified rejuvenator dropped back to the pre-pretreatment level after one-day storage at room temperature.

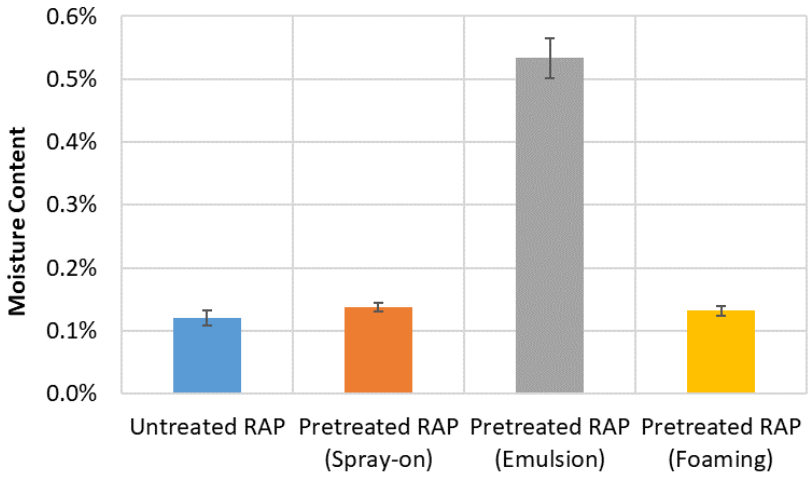


Figure 25. Moisture Contents of Untreated and Pretreated RAP with Different Rejuvenator Application Methods

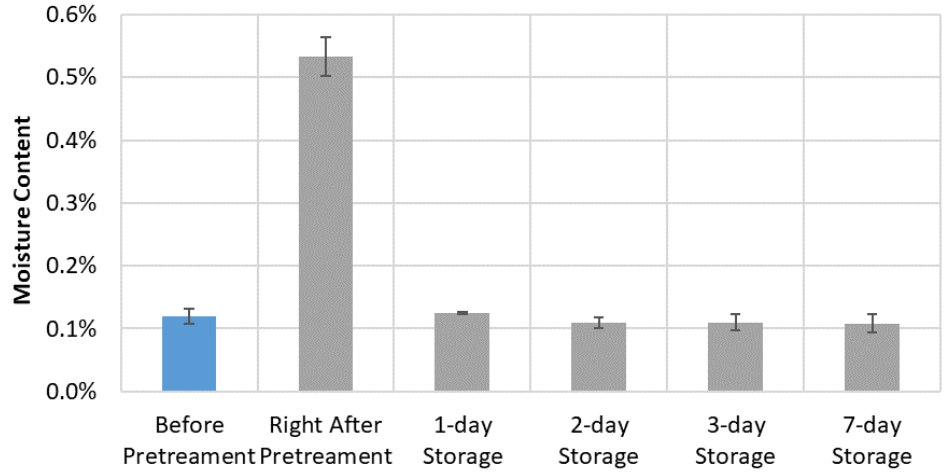


Figure 26. Moisture Contents of Pretreated RAP with Emulsified Rejuvenator at Various Storage Times

5.3 IMAGE ANALYSIS RESULTS OF UNTREATED AND PRETREATED RAP

Figure 27 presents the grayscale distribution plots of the untreated RAP and pretreated RAP with the spray-on, emulsion, and foaming methods. The grayscale values ranging from 0 to 250 represent the relative color of the RAP sample, where a small value indicates a dark black pixel and a large value indicates a light white pixel. As shown, all the three pretreated RAP samples had an overall darker appearance than the untreated sample as indicated by lower average grayscale values (i.e., 68.2 for the untreated RAP sample versus 40.9, 42.2, and 50.8 for the pretreated RAP samples). This indicated that adding rejuvenators as pretreatment improved the quality characteristics of the RAP, which agreed with the DWT results in Figure 24. Among the three RAP pretreatment methods, the emulsion and foaming methods seemed to be more effective than the spray-on method when evaluated based on the relative

change in color of the RAP after pretreatment (as indicated by a shift of the distribution plot towards the left) as well as the degree of color uniformity (as indicated by the width of the distribution plot).

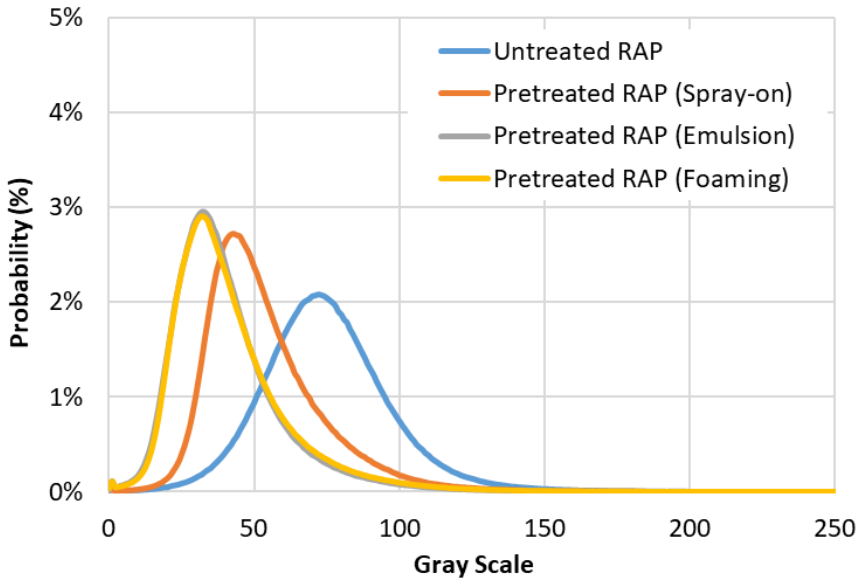


Figure 27. Grayscale Distribution Plots of Untreated and Treated RAP with Different Rejuvenator Application Methods

5.4 SUMMARY

The DWT and image analysis results discussed above indicated that the emulsion method was slightly more effective for RAP pretreatment than the spray-on and foaming methods. Therefore, the pretreated RAP with the emulsified rejuvenator was selected for further evaluation in the *RAP Marination* sub-experiment, which is discussed in Chapter 6.

CHAPTER 6: QUALITY CHARACTERISTICS OF MARINATED RAP WITH REJUVENATORS

This chapter discusses the test results and data analysis of the *RAP Marination* sub-experiment. The objective of the experiment was to determine the impact of marination on the quality characteristics of the pretreated RAP as well as the rheological and chemical properties of the extracted RAP binder. The RAP sample used for this experiment was the pretreated RAP A (i.e., the RAP from mix design A) with the emulsified RA1, as it showed the best quality characteristics in the *RAP Pretreatment* sub-experiment. Four marination conditions were evaluated: marination for 1.5 hours at 135°C (275°F), marination for 3 hours at 135°C (275°F), marination for 3 days at room temperature, and marination for 7 days at room temperature. At each marination condition, the pretreated RAP was characterized through DWT and grayscale-based image analysis. The pretreated RAP sample with no marination was also tested as control for comparison purposes. Furthermore, asphalt binders were extracted and recovered for three selected pretreated RAP samples and tested to determine their PG, MSCR, *G-R* results, and SARA fractions.

6.1 DWT RESULTS OF PRETREATED RAP AFTER MARINATION

Figure 28 presents the DWT results of the pretreated RAP at various marination conditions, where the error bars represent one standard deviation of the replicate measurements. At both test temperatures, the no-marination control RAP sample had the highest DWT values and thus, was expected to have the best workability. Marination time and temperature did not appear to significantly affect the workability of RAP as all the samples after marination had similar DWT values. These results indicated that marination had no effect on the rejuvenation of RAP from the workability perspective.

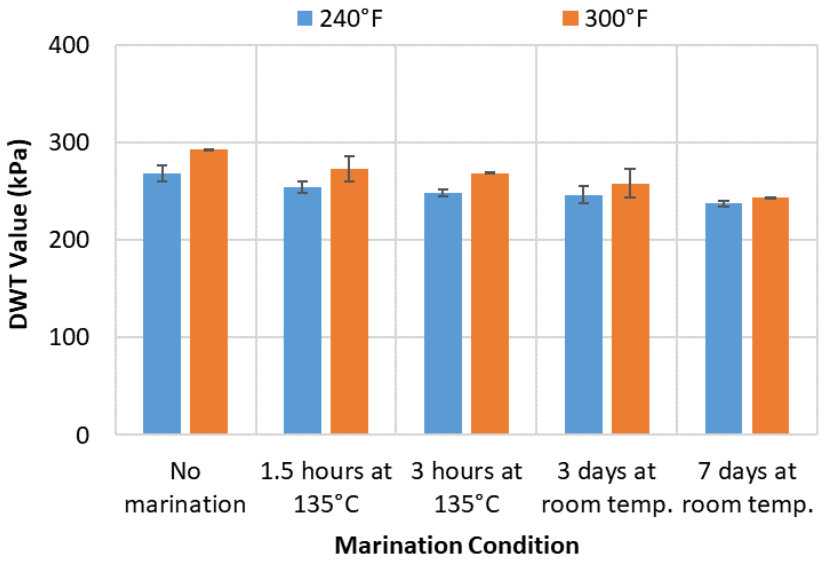


Figure 28. DWT Results of Pretreated RAP at Various Marination Conditions

6.2 IMAGE ANALYSIS RESULTS OF PRETREATED RAP AFTER MARINATION

Figure 29 presents the grayscale distribution curves of the pretreated RAP at various marination conditions. All the RAP samples had consistent color appearance and thus, exhibited reasonably overlapping grayscale distribution curves. The average grayscale value of the no-marination RAP sample was 40.9, which was slightly higher than the marinated RAP samples, which varied within a narrow range of 35.9 to 39.9. Therefore, the image analysis results indicated that marination did not improve RAP quality from the color appearance and consistency perspective, which agreed with the DWT results in Figure 28.

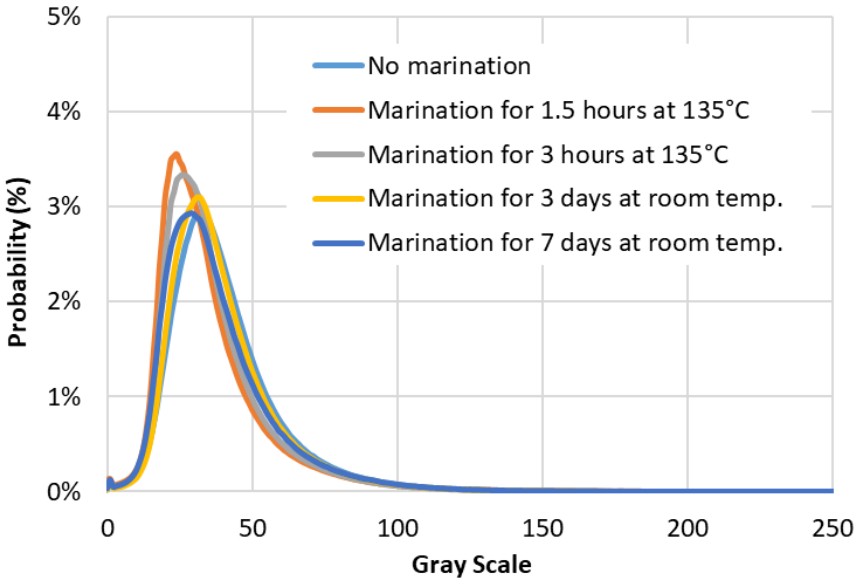


Figure 29. Grayscale Distribution Curves of Pretreated RAP at Various Marination Conditions

6.3 EXTRACTED RAP BINDER RESULTS

Table 3 presents the Superpave PG, ΔT_c, and MSCR results of extracted RAP binders with no marination, 3-hour marination at 135°C (275°F), and 7-day marination at room temperature. All binders were tested as recovered without additional RTFO or PAV aging. In comparison to the no marination condition (control), marination for 3 hours at 135°C (275°F) increased the high-temperature true grade of the extracted RAP binder by 5.3°C (82.5 to 87.8°C), while marination for 7 days at room temperature decreased the high-temperature true grade by 5.1°C (82.5 to 77.4°C). The extracted RAP binder after marination for 3 hours at 135°C (275°F) had a low-temperature true grade 1.1°C higher than the control (-30.1 to -29.0°C), while marination for 7 days at room temperature resulted in a low-temperature true grade -3.3°C lower than the control (-30.1 to -33.4°C). In terms of the impact on the stress relaxation property of the extracted RAP binder, marination for 7 days at room temperature was beneficial as the ΔT_c improved from 0.5 to 1.2°C, while marination for 3 hours at 135°C (275°F) resulted in a reduction of ΔT_c from 0.5 to -0.8°C.

Table 3. PG, ΔTc, and MSCR Results of Extracted RAP Binders with Different Marination Conditions

Marination Condition	T_{cont}, High (°C)	T_{cont}, Low S (°C)	T_{cont}, Low m-value (°C)	T_{cont} (°C)	ΔTc (°C)	Superpave PG	MSCR J_{nr} @ 3.2kPa (1/kPa)	MSCR %R @ 3.2kPa (%)
No marination	82.5	-30.1	-30.6	-30.1	0.5	82-28	0.94	3.2
3 hours at 135°C	87.8	-29.8	-29.0	-29.0	-0.8	82-28	0.40	12.9
7 days at Room Temp.	77.4	-33.4	-34.6	-33.4	1.2	76-28	2.03	0.7

The MSCR results indicated that, in comparison to the no marination condition, marination for 3 hours at 135°C (275°F) resulted in a decrease in J_{nr} (0.94 to 0.40 kPa^{-1}) indicating a binder stiffening impact, while marination for 7 days at room temperature resulted in an increase in J_{nr} (0.94 to 2.03 kPa^{-1}), indicating a binder softening impact. It was also observed that marination for 3 hours at 135°C (275°F) increased the %Recovery of the extracted RAP binder in comparison to the control (3.2 to 12.9%), while marination for 7 days at room temperature decreased the %Recovery from 3.2 to 0.7%. However, it should be noted that the increase in the %Recovery observed for the 3-hour, 135°C (275°F) marination was highly influenced by the J_{nr} of the extracted binder. Overall, the PG, ΔTc, and MSCR results in Table 3 indicated that the 7-day, room-temperature marination improved the rejuvenation of RAP in terms of decreasing binder stiffness and increasing relaxation properties, while the accelerated 3-hour, 135°C (275°F) marination had a stiffening and embrittlement impact on the RAP binder due to oxidative aging.

Table 4 summarizes the *G-R* parameter results (long with the |G*| and δ results at 15°C (59°F) and 0.005 rad/s) of the extracted RAP binders evaluated in the *RAP Marination* sub-experiment. In comparison to the no marination condition (control), marination for 3 hours at 135°C (275°F) significantly increased the *G-R* parameter of the extracted RAP binder from 29.0 to 65.1 kPa, while marination for 7 days at room temperature decreased the *G-R* parameter from 29.0 to 6.7 kPa. These results highlighted a stiffening effect of the extracted RAP binder from the 3-hour, 135°C (275°F) conditioning, which agreed with the binder results in Table 3.

Table 4. |G*| at 15°C and 0.005 rad/s and *G-R* Parameter Results of Extracted RAP Binders

Marination Condition	|G*| at 15°C and 0.005 rad/s (kPa)	δ at 15°C and 0.005 rad/s (°)	*G-R* Parameter (kPa)
No marination	161.6	66.1	29.0
3 hours at 135°C	236.1	60.6	65.1
7 days at Room Temp.	55.0	70.3	6.7

Figure 30 presents the *G-R* parameter results on a Black Space diagram, where the binder |G*| at 15°C (59°F) and 0.005 rad/s is plotted on the y-axis versus δ at the same condition on the x-axis. The dashed and bold 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, marination for 3 hours at 135°C (275°F) increased |G*| but decreased δ of the extracted RAP binder, indicating increased

stiffness and brittleness. As a result, the binder had a higher *G-R* parameter (65.1 kPa) than that with no marination (29.0 kPa) and thus, was located closer to the preliminary cracking damage zone on the Black Space diagram. Compared to the no marination condition (control), marination for 7 days at room temperature had lower |G*| and higher δ, which resulted in a lower *G-R* parameter (6.7 kPa *versus* 29.0 kPa for control) and thus, shifted the data point towards the bottom right corner of the diagram. These results supported the previous findings from the PG, ΔTc, and MSCR results in Table 3 that the 7-day, room-temperature marination was more beneficial for rejuvenation of RAP than the accelerated 3-hour, 135°C (275°F) marination.

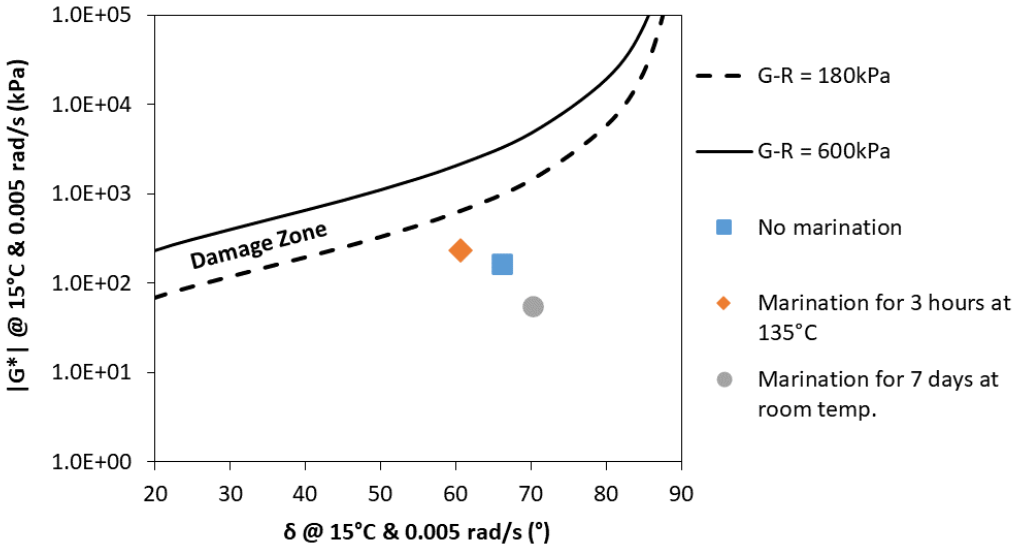


Figure 30. *G-R* Parameter Results of Extracted RAP Binders on a Black Space Diagram

The SARA analysis was performed to separate each extracted RAP binder into four chemical fractions based on differences in solubility and polarity. The results are summarized in Figure 31. The numerical differences in each of the SARA fractions among the extracted RAP binders with different marination conditions are summarized as follows:

- Saturates: marination for 7 days at room temperature (3.5%) > no marination (2.8%) > marination for 3 hours at 135°C (275°F) (2.5%)
- Aromatics: marination for 7 days at room temperature (29.5%) > no marination (29.3%) > marination for 3 hours at 135°C (275°F) (27.3%)
- Resins: no marination (39.4%) > marination for 3 hours at 135°C (275°F) (39.2%) > marination for 7 days at room temperature (38.4%)
- Asphaltenes: marination for 3 hours at 135°C (275°F) (31.0%) > marination for 7 days at room temperature (28.6%) > no marination (28.5%)

Using the asphaltenes content as an indicator for asphalt aging, marination for 3 hours at 135°C (275°F) caused further aging of the pretreated RAP while marination for 7 days at room temperature did not significantly affect the chemical fractions of the extracted RAP binder.

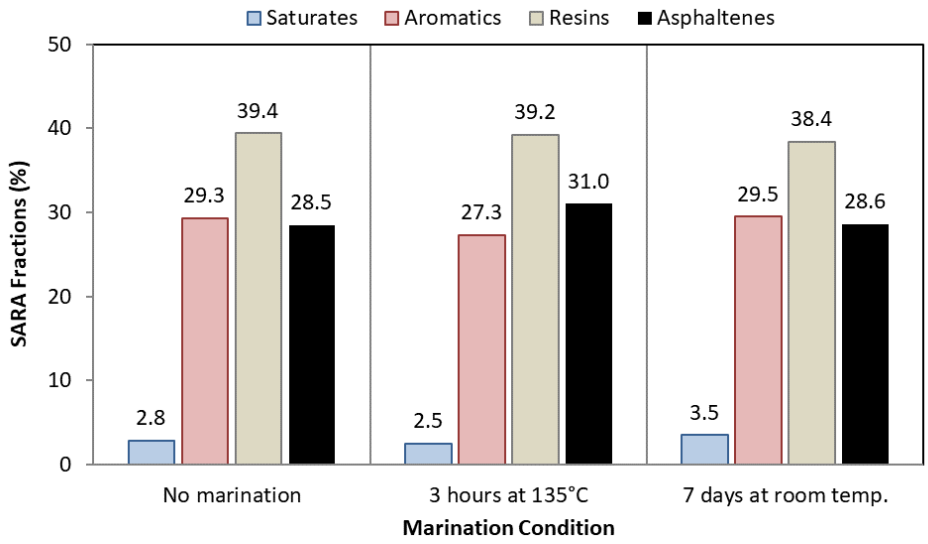


Figure 31. SARA Fractions of Extracted RAP Binders with Different Marination Conditions

Figure 32 presents the CII results of the extracted RAP binders, where CII is related to the aggregation or agglomeration of the asphalt fractions. As shown, the extracted RAP binder with marination for 3 days at 135°C (275°F) had a higher CII and thus, lower colloidal stability than those with no marination and marination for 7 days at room temperature.

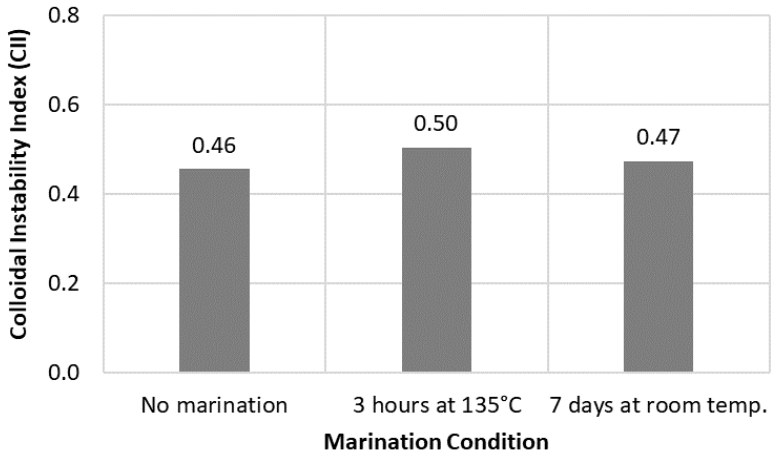


Figure 32. CII of Extracted RAP Binders with Different Marination Conditions

6.4 SUMMARY

The rheological evaluation and SARA analysis results of the extracted RAP binders indicated that marinating the pretreated RAP for 7 days at room temperature slightly enhanced the effectiveness of rejuvenation, while the 3-hour, 135°C (275°F) marination further aged the rejuvenated RAP binder, which resulted in increased stiffness, brittleness, and oxidation. These differences in the binder properties, however, were not observed when characterizing the quality characteristics of the pretreated RAP samples at various marination conditions. The DWT and image analysis results indicated

that in general, marination did not improve the workability, color appearance, or consistency of the pretreated RAP.

CHAPTER 7: MIXTURE PERFORMANCE TEST RESULTS

Chapter 7 presents the test results and data analysis of the *Mixture Performance Testing* experiment. The objective of the experiment was to evaluate the workability and cracking resistance of high-RAP mixtures prepared with different rejuvenator application methods and those with no rejuvenator. Mixture workability evaluation was based on DWT results and the mixture cracking resistance was evaluated using the IDEAL-CT and DCT. The DWT was conducted on short-term aged specimens (i.e., 2 hours at the compaction temperature per AASHTO R30), while the IDEAL-CT and DCT were conducted on long-term aged specimens [i.e., 4 hours at 135°C (275°F) per AASHTO R 30 followed by 6 additional hours at 135°C (275°F) for critical aging)]. The experimental plan included two high-RAP mix designs, two virgin binders, and two rejuvenators. Mix design A was a 9.5mm NMAS Superpave mixture with 45% RAP and was evaluated with the PG 67-22 binder and RA1. Mix design B was a 12.5mm NMAS Superpave mixture with 50% RAP and was evaluated with the PG 58S-28 binder with RA2. For each mix design, a control mixture without rejuvenator and four rejuvenated mixtures (with the same "effective" rejuvenator dosage, but using different rejuvenator application methods) were prepared and tested, described as follows:

- Mix X-1 is the control mixture containing no rejuvenator
- Mix X-2 is a rejuvenated mixture prepared by pre-blending the rejuvenator with the virgin binder
- Mix X-3 is a rejuvenated mixture prepared by pre-treating the RAP with the emulsified rejuvenator
- Mix X-4 is a rejuvenated mixture prepared by pre-treating the RAP with the foamed rejuvenator
- Mix X-5 is a rejuvenated mixture prepared by foaming the rejuvenated virgin binder (after pre-blending the rejuvenator with the virgin binder)

(Note that "X" refers to the mix design denotation, with "A" for mix design A and "B" for mix design B.)

In this chapter, all the test results are presented using column charts, where the columns represent the average DWT, IDEAL-CT, and DCT index parameter results and the error bars represent one plus and minus standard deviation. For data analysis, both the mean value analysis and the Games-Howell post-hoc group analysis at a significance level of 0.05 (for the IDEAL-CT and DCT only) were conducted to compare the test results of the control and rejuvenated mixtures for each mix design. The capital letters located inside the columns represent the group analysis results, where mixtures sharing a same letter had no statistically significant difference among their test results.

7.1 DWT RESULTS

Figure 33 and Figure 34 presents the DWT results of mix design A and B, respectively, at 116°C (240°F) and 149°C (300°F). For mix design A, all four of the rejuvenated mixtures had higher average DWT values than the control mixture at both test temperatures (Figure 33), which indicated that adding rejuvenators in general, regardless of the application method used, was able to improve the mixture workability. Among the four rejuvenated mixtures, Mix A-5 (prepared by foaming the rejuvenated virgin binder) had

a slightly higher average DWT value at 149°C (300°F) than Mix A-2 through Mix A-4, but these differences were not significant when considering the variability of the test results. Overall, the DWT results in Figure 33 indicated that the different rejuvenator application methods did not have a significant effect on the workability of high-RAP mixtures prepared with mix design A.

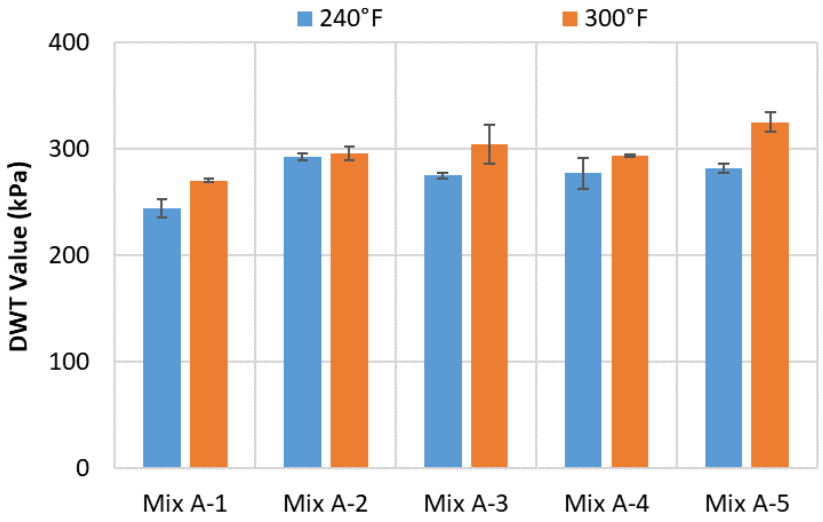


Figure 33. DWT Results of Mix Design A

For the mix design B results in Figure 34, all the rejuvenated mixtures had higher average DWT values than the control mixture, which was consistent with the results of mix design A. The differences in DWT results between the control and rejuvenated mixtures were more pronounced at the higher test temperature. At 149°C (300°F), the two mixtures prepared with the pretreated RAP (i.e., Mix B-3 and Mix B-4) had the highest average DWT values, which indicated that the RAP pretreatment method provided better mixture workability at 149°C (300°F) than the other rejuvenator application methods for mix design B. However, at 116°C (240°F), the four rejuvenated mixtures had very similar average DWT values indicating equivalent workability. Overall, the DWT results in Figure 34 indicated that the different rejuvenator application methods did not significantly affect the workability of high-RAP mixtures prepared with mix design B.

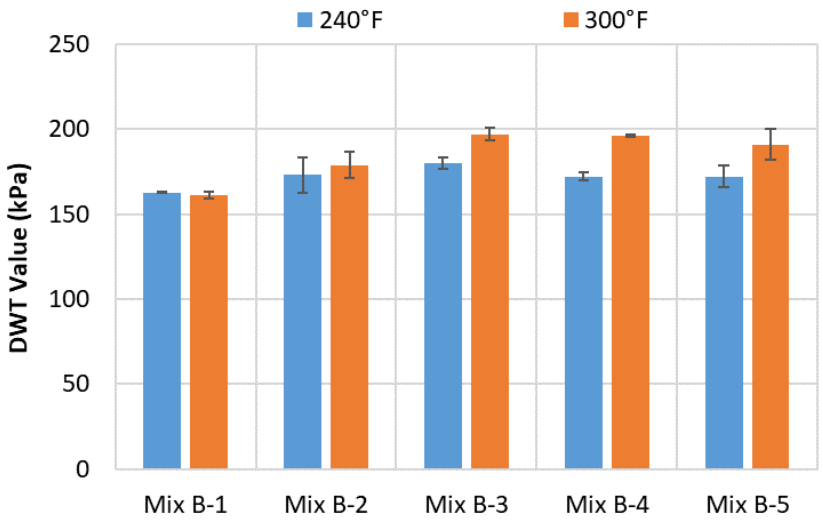


Figure 34. DWT Results of Mix Design B

When comparing the DWT results in Figure 33 and Figure 34, it can be observed that the high-RAP mixtures prepared with mix design A had consistently higher DWT values (in the range of 250 to 300 kPa) than those with mix design B (in the range of 150 to 200 kPa), indicating better workability. This difference was likely because mix design A had a finer gradation, higher binder content, and higher rejuvenator dosage than mix design B, all of which was expected to contribute to improved mixture workability. Therefore, these results demonstrated the feasibility of using the DWT test to evaluate mixture workability.

7.2 IDEAL-CT RESULTS

Figure 35 presents the CT_{index} results, along with the corresponding group analysis results, of high-RAP mixtures prepared with mix design A. As shown, all four of the rejuvenated mixtures had higher average CT_{index} values than the control mixture, implying improved intermediate-temperature cracking resistance due to the addition of the rejuvenator. Among the rejuvenated mixtures, Mix A-2 and Mix A-5 had higher average CT_{index} than Mix A-3 and Mix A-4, which indicated that pre-blending the rejuvenator into the virgin binder provided better rejuvenating effectiveness than adding the rejuvenator by pretreating the RAP. The statistical group analysis confirmed that all the rejuvenated mixtures had significantly higher CT_{index} results than the control mixture. However, there was no statistical difference in the CT_{index} among the four rejuvenated mixtures except that Mix A-5 had a significantly higher CT_{index} than Mix A-4. Overall, the CT_{index} results in Figure 35 indicated that the different rejuvenator application methods did not significantly affect the intermediate-temperature cracking resistance of high-RAP mixtures prepared with mix design A when tested at a long-term aging condition.

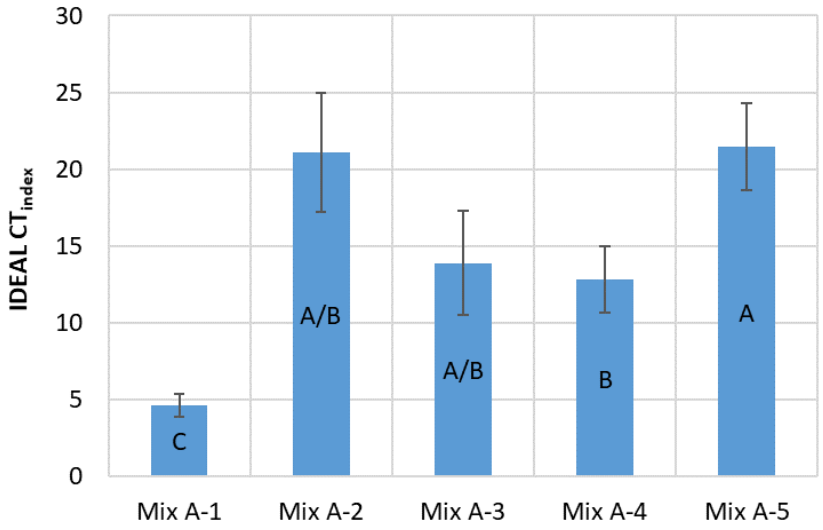


Figure 35. IDEAL-CT CT_{index} Results of Mix Design A

In addition to the CT_{index} parameter, two interim index parameters derived from the IDEAL-CT load-displacement curve were evaluated: G_f and $|m_{75}|/l_{75}$, where G_f indicates mixture toughness and $|m_{75}|/l_{75}$ indicates the mixture's relative ductile-brittle behavior. As expressed in Equation 10, a higher G_f and a lower $|m_{75}|/l_{75}$ yields a higher CT_{index}. As shown in Figure 36(a), all the rejuvenated mixtures had almost identical average G_f values, which were slightly higher than that of the control mixture. This difference, however, was found to be insignificant according to the statistical group analysis. The $|m_{75}|/l_{75}$ results in Figure 36(b) showed that pre-blending the rejuvenator into the virgin binder provided the RAP mixtures with more ductile behavior than adding the rejuvenator by pretreating the RAP, as indicated by lower average $|m_{75}|/l_{75}$ values for Mix A-2 and A-5 compared to Mix A-3 and A-4. Furthermore, the control mixture had a significantly higher $|m_{75}|/l_{75}$ value and thus, more brittle behavior than the rejuvenated mixtures, which resulted in a lower CT_{index} indicating reduced intermediate-temperature cracking resistance. The results in Figure 36 showed that the $|m_{75}|/l_{75}$ parameter was able to better discriminate the different rejuvenator application methods than the G_f parameter.

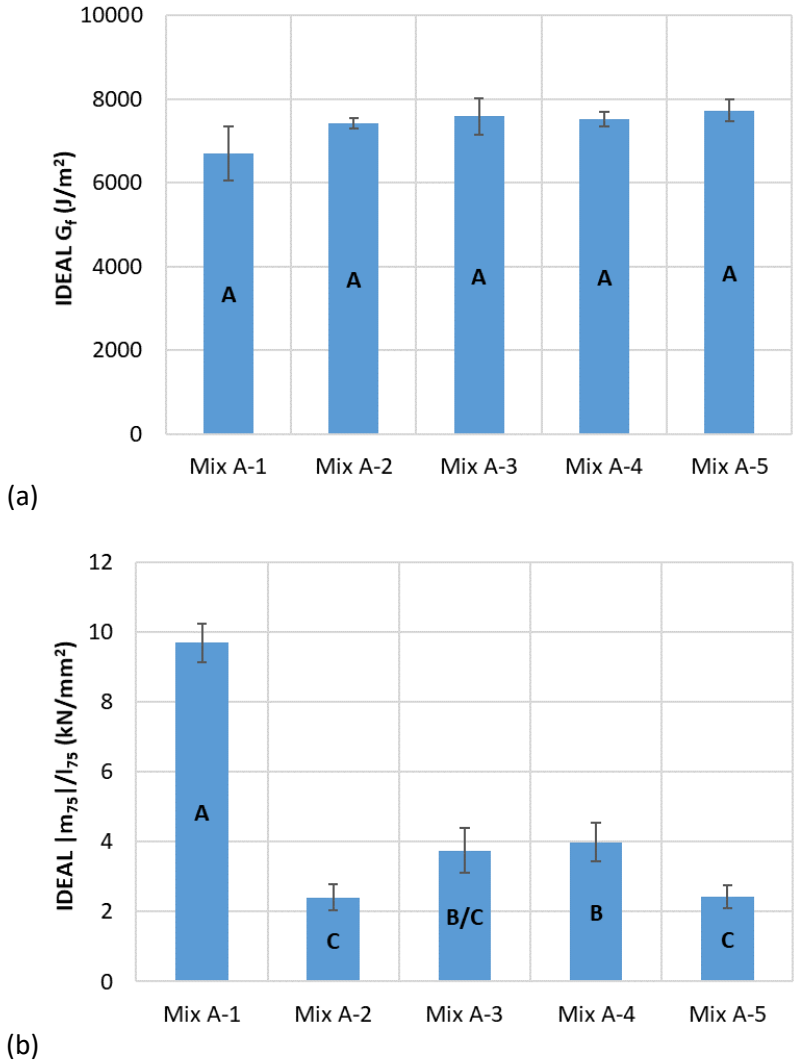


Figure 36. IDEAL-CT Interim Index Parameter Results of Mix Design A; (a) G_f, (b) $|m_{75}|/l_{75}$

Figure 37 presents the CT_{index} results of high-RAP mixtures prepared with mix design B, along with the corresponding statistical group analysis results. As shown, all four of the rejuvenated mixtures had higher average CT_{index} than the control mixture, indicating improved intermediate-temperature cracking resistance due to adding rejuvenators. The rejuvenated mixtures prepared with the pre-blending method (i.e., Mix B-2 and B-5) had slightly higher average CT_{index} values (by approximately five CT_{index} units) than those prepared with the RAP pretreatment method (i.e., Mix B-3 and B-4), This indicated that pre-blending the rejuvenator into the virgin binder provided better rejuvenating effectiveness than adding the rejuvenator through RAP pretreatment. However, the group analysis results indicated that there was no significant difference in the CT_{index} of all the mixtures when considering the variability of the test results.

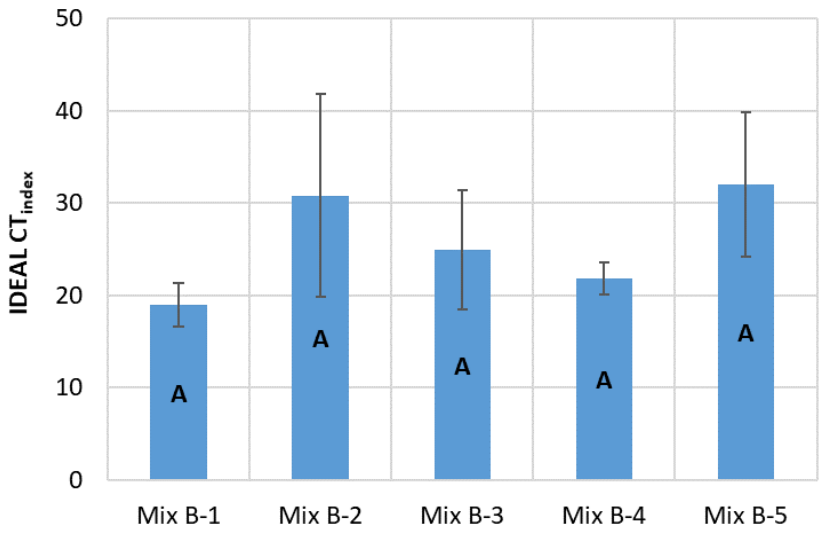
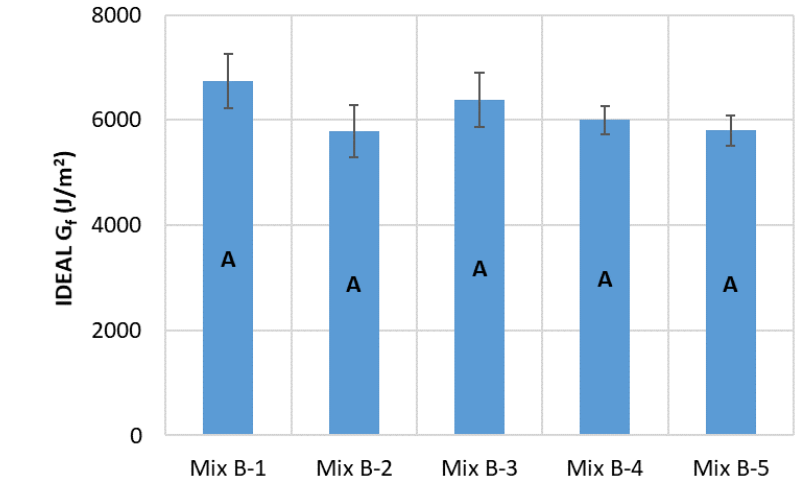
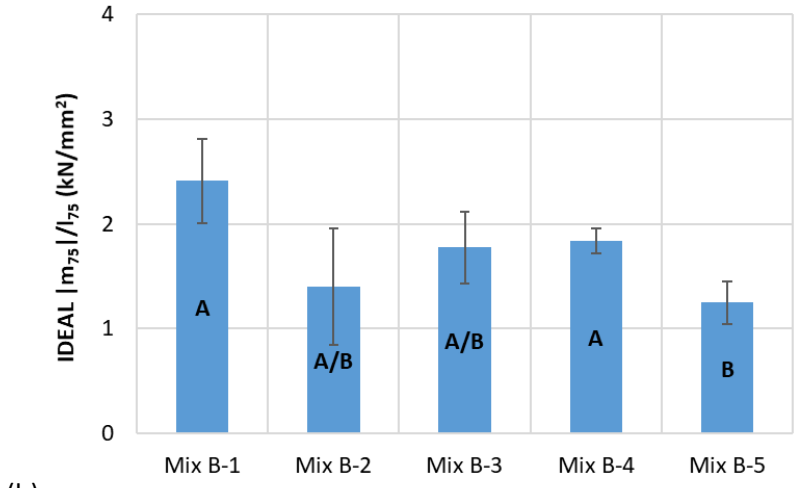


Figure 37. IDEAL-CT CT_{index} Results of Mix Design B

Figure 38 shows the G_f and $|m_{75}|/l_{75}$ results of mix design B. As shown in Figure 38(a), the control mixture had a consistently higher average G_f value than the rejuvenated mixtures, although these differences were not found to be significant according to the statistical group analysis. Compared to G_f, $|m_{75}|/l_{75}$ appeared to better discriminate the control mixture and the rejuvenated mixtures with different rejuvenator application methods, as shown in Figure 38(b). The control mixture had a higher $|m_{75}|/l_{75}$ value, indicating more brittle behavior from the indirect tensile testing in the IDEAL-CT than the rejuvenated mixtures. Among the rejuvenated mixtures, Mix B-2 and B-5 had lower $|m_{75}|/l_{75}$ values than Mix B-3 and B-4, which indicated that the pre-blending method of adding the rejuvenator yielded RAP mixtures with more ductile behavior in the IDEAL-CT than the RAP pretreatment method. The statistical group analysis results showed that only the difference in $|m_{75}|/l_{75}$ between Mix B-1 or B-4 and Mix B-5 was significant, while the differences among all the other mixtures were not.



(a)



(b)

Figure 38. IDEAL-CT Interim Index Parameter Results of Mix Design B; (a) G_f, (b) $|m_{75}|/l_{75}$

In summary, for both mix designs, pre-blending the rejuvenator into the virgin binder appeared to provide slightly better rejuvenating effectiveness than adding the rejuvenator by pretreating the RAP, although the differences in the CT_{index} results were not statistically significant in most cases. Furthermore, the different rejuvenator application methods affected the relative ductile-brittle behavior of high-RAP mixtures as indicated by $|m_{75}|/l_{75}$, while they had no significant impact on mixture toughness as indicated by the G_f parameter.

7.3 DCT RESULTS

Figure 39 and Figure 40 present the DCT G_f results of mix design A and B, respectively, which also include the statistical group analysis results. As shown in Figure 39, all the rejuvenated mixtures had higher average G_f values than the control mixture, which indicated that in general, adding rejuvenators improved the thermal cracking resistance of high-RAP mixtures prepared with mix design A. The four rejuvenated mixtures had very similar G_f values, implying that the thermal cracking resistance of mix

design A was not affected by the different rejuvenator application methods. The statistical group analysis results showed that there was no significant difference in the G_f results among all the mixtures, including the control mixture versus the four rejuvenated mixtures. This lack of discrimination was mainly attributed to the abnormally high variability of Mix A-1 with a COV 33.3%. Overall, the results in Figure 39 indicated the different rejuvenator application methods evaluated in the study did not appear to affect the thermal cracking resistance of high-RAP mixtures prepared with mix design A.

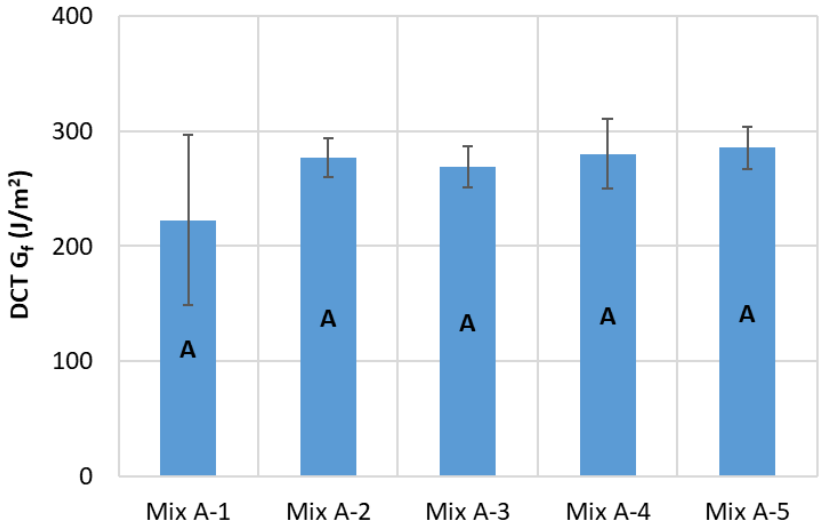


Figure 39. DCT G_f Results of Mix Design A

As shown in Figure 40, for mix design B, three out of the four rejuvenated mixtures (i.e., Mix B-2, B-3, and B-5) had higher average DCT G_f values than the control mixture, while Mix B-4 had a slightly lower average G_f value than the control mixture, which was unexpected. The group analysis results showed that no significant difference in terms of DCT G_f existed between the control mixture and the rejuvenated mixtures except Mix B-2, which was prepared by pre-blending the rejuvenator into the virgin binder without foaming. Among the rejuvenated mixtures, only Mix B-2 and Mix B-4 had significantly different G_f results when considering the variability of the test, while the differences among all the other mixtures were found to be insignificant. Overall, the results in Figure 40 indicated that the different rejuvenator application methods did not have a significant impact on mixture thermal cracking resistance in terms of DCT G_f, and that adding the rejuvenator by pretreating the RAP did not provide better rejuvenating effectiveness than pre-blending the rejuvenator into the virgin binder for high-RAP mixtures prepared with mix design B.

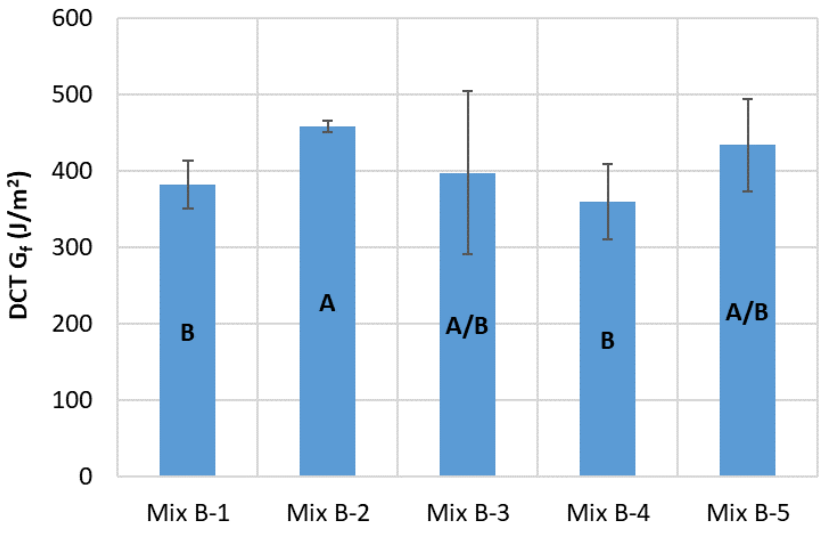


Figure 40. DCT G_f Results of Mix Design B

7.4 SUMMARY

The DWT, IDEAL-CT, and DCT results from this experiment indicated that adding rejuvenators, in general, improved the workability, intermediate-temperature cracking resistance, and thermal cracking resistance of high-RAP mixtures, although in some cases, the improvement was not significant according to the statistical group analysis. Among the different rejuvenator application methods evaluated, pre-blending the rejuvenator into the virgin binder, with and without foaming, provided slightly better or equivalent rejuvenating effectiveness and resultant mixture performance properties than adding the rejuvenator through RAP pretreatment. In cases where there was an improvement in the IDEAL-CT results, the rejuvenated mixtures prepared with the pre-blending method had a more ductile post-peak behavior than those prepared with the pretreatment method.

CHAPTER 8: CONCLUSIONS AND RECOMMENDATIONS

The objective of this study was to evaluate different methods of adding rejuvenators for improving the workability and cracking resistance of two high-RAP asphalt mixtures with 45% or 50% RAP. To that end, a comprehensive experimental plan was executed, which consisted of four supplementary experiments focusing on rejuvenator characterization, foaming measurements of rejuvenators and rejuvenated asphalt binders, RAP pretreatment and marination evaluations, and mixture performance testing, respectively. Major findings and conclusions of the study are summarized below:

- The two rejuvenators used in the study (i.e., RA1 and RA2), in both their original and emulsion forms, exhibited good wetting properties with the PG 67-22 and PG 58S-28 virgin binders when measured using the Sessile Drop test. Furthermore, they had similar rotational viscosity results at unaged and RTFO aged conditions but had significantly different results (higher viscosity for RA2 than RA1) after RTFO plus 20-hour and 40-hour PAV aging, which highlighted the different aging susceptibility of the two rejuvenator products.
- The two rejuvenators and the two rejuvenated asphalt binders (i.e., PG 67-22 with RA1, and PG 58S-28 with RA2) showed good foaming characteristics at most of the foaming conditions evaluated in the study. Based on the FI parameter, the optimum foaming conditions were selected as follows: 120°C (248°F) and 3% water content for RA1; 130°C (266°F) and 3% water content for RA2; and 150°C (302°F) and 2% water content for the two rejuvenated asphalt binders.
- Adding the rejuvenator for RAP pretreatment, in general, significantly improved the quality characteristics of the RAP from mix design A. This improvement was manifested by higher DWT values as well as darker appearance and better color consistency from the grayscale-based image analysis for the pretreated RAP samples versus the untreated sample. Among the different rejuvenator application methods for RAP pretreatment, the emulsion method was slightly more effective than the spray-on and foaming methods.
- The spray-on and foaming methods of adding rejuvenators for RAP pretreatment did not affect the moisture content of the RAP. The emulsion method, on the other hand, significantly increased the moisture content of the RAP due to the inclusion of water in the emulsified rejuvenator product. The moisture content of the pretreated RAP dropped back to the pretreatment level after one day of room-temperature storage in the laboratory.
- Based on the rheological evaluation and SARA fraction results of the extracted RAP binders, marinating the pretreated RAP for 7 days at room temperature slightly improved the rejuvenating effectiveness, but the 3-hour, 135°C (275°F) marination caused further aging of the pretreated RAP, resulting in increased binder stiffness, brittleness, and oxidation. However, marination did not significantly affect the workability, appearance, or color consistency of the pretreated RAP samples, as they had similar DWT results and grayscale distribution curves at various marination conditions.
- Adding rejuvenators, in general, improved the workability and cracking resistance of high-RAP mixtures when evaluated using the DWT, IDEAL-CT, and DCT tests, although in some cases, the improvement was not significant when considering the variability of the test results. Among the

different rejuvenator application methods, pre-blending the rejuvenator into the virgin binder, with or without foaming, provided slightly better or equivalent rejuvenating effectiveness and resultant mixture performance properties than adding the rejuvenator through RAP pretreatment.

Based on the findings of this study, it is recommended that asphalt contractors continue to use the pre-blending method of adding rejuvenators to design and produce high-RAP mixtures due to performance and ease of operation considerations. Furthermore, asphalt contractors equipped with a plant foaming unit are suggested to use the foaming-enhanced pre-blending method of adding rejuvenators for improved mixture workability and compactability. The foaming-enhanced pre-blending method may also allow asphalt contractors to produce high-RAP mixtures at reduced temperatures as WMA, which provides significant economic and environmental benefits. Given the promising results obtained in the *RAP Pretreatment and Marination* experiment of the study, future research is recommended to further evaluate the use of the DWT as a quick tool to evaluate the overall quality and consistency of RAP stockpiles for asphalt mixture design and production.

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