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

by

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Abstract

Most asphalt mixtures being placed in the United States currently use RAP as not only a sustainable method of building pavement surfaces but also an economical one. The stiffness and brittleness of the aged RAP binder, which negatively affects mixture performance in high quantities, can be mitigated using rejuvenators. The current method of rejuvenator incorporation does not consider RAP pretreatment for potentially enhancing RAP binder activation. In this study, the degree of dispersion of rejuvenator for RAP pretreatment was evaluated along with the effect of marination. The experimental plan included two bio-based rejuvenators, two high RAP mix designs, and three RAP pretreatment methods. The performance properties of high RAP mixtures prepared with different rejuvenator addition methods were assessed based on the DWT, Cantabro, IDEAL-CT, I-FIT, and DCT. Results suggested that asphalt contractors should continue to preblend rejuvenators into the virgin binder for best rejuvenating effectiveness and mixture performance.

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List of Abbreviations

AASHTO American Association of Highway and Transportation Officials

ASTM American Society for Testing and Materials

CT_{Index} Cracking Tolerance Index

CMOD Crack Mouth Opening Displacement

DCT Disc-Shaped Compact Tension Test

DOT Department of Transportation

DSR Dynamic Shear Rheometer

DWT Dongré Workability Test

ER Expansion Ratio

ER_{max} Maximum Expansion Ratio

FI Foamability Index

FTIR Fourier Transform Infrared Spectroscopy

G_f Fracture Energy

HMA Hot Mix Asphalt

I₇₅ Post-Peak Displacement at 75% of Peak Load

IDEAL-CT Indirect Tensile Asphalt Cracking Test

I-FIT Illinois Flexibility Index Test

ITS Indirect Tensile Strength

LAA Los Angeles Abrasion

LTOA Long-Term Oxidative Aging

|m₇₅| Post-Peak Slope at 75% of Peak Load

NCAT National Center for Asphalt Technologies

NMAS Nominal Maximum Aggregate Size

NRRA National Road Research Alliance

OT Overlay Test

PAV Pressure Aging Vessel

PG Performance Grade

RA Recycling Agent

RAP Reclaimed Asphalt Pavement

RBR Recycled Binder Ratio

RTFO Rolling Thin Film Oven

SCB Semi-Circular Bending

SGC Superpave Gyratory Compactor

STOA Short-Term Oxidative Aging

 $t_{1/2}$ Expansion Half-Life

TCE Trichloroethylene

 $\Delta T_C \qquad \qquad Delta \; T_C$

VMA Voids in the Mineral Aggregate

W_f Work of Fracture

Chapter 1: Introduction

1.1 Background

Due to significant economic and environmental benefits, reclaimed asphalt pavement (RAP) has been utilized in earnest in new asphalt pavements. However, the heavily aged RAP binder increases asphalt pavement stiffness and brittleness, with high RAP content mixtures potentially susceptible to cracking and durability issues. One method of mitigating these performance issues is the incorporation of recycling agents (RA), also known as rejuvenators, to increase the blending of the RAP binder and virgin binder, along with the revitalization of the rheological properties of the RAP binder. RAs are defined by the Asphalt Institute in 1986 as organic materials with chemical and physical characteristics chosen to retore the rheological properties of aged asphalt to achieve target specifications (*Asphalt Hot-Mix Recycling*, 1986).

Along with the physical and chemical interactions with the RAP, the degree of dispersion and application (e.g., foaming and emulsion) of the RA is vital for optimal rejuvenation of the RAP material for overall mixture performance.

The foaming-enhanced RAP pretreatment method is one of the production practices through which successful high RAP mixtures (up to 75%) have been produced in Japan (Koshi et al., 2017). Previous laboratory investigations at the National Center for Asphalt Technologies (NCAT) found that the foaming of rejuvenators can achieve similar volume expansion to that of asphalt binders. Like foaming-enhanced RAP pretreatment, the emulsion-enhanced pretreatment method also utilizes water for volume expansion of the rejuvenator, but to a larger extent and with surfactants to maintain the emulsion. Emulsified rejuvenators are popular with asphalt pavement preservation methods, especially in rejuvenating seal applications to preserve functional and structural integrity (Moraes, 2019). Due to the successes of this technique, emulsion-enhanced

RAP pretreatment is expected to increase the dispersion of the rejuvenator and "activate" the RAP binder.

1.2 Research Hypothesis

Rejuvenators can improve the workability, durability, and cracking resistance of asphalt mixtures containing RAP by rejuvenating the rheological and chemical properties of the recycled binder. It is hypothesized that the RAP rejuvenating effectiveness of a rejuvenator can be further improved when its application is performed using either foaming or emulsion methods.

1.3 Research Objectives

The overall objective of this study was to explore three alternative methods of incorporating rejuvenators and determine their effects on the performance properties of high RAP asphalt mixtures. The rejuvenator incorporation methods evaluated in the study include:

- Traditional pre-blending method: the rejuvenator is pre-blended into the virgin binder, then combined with the virgin aggregate and RAP.
- Foaming-enhanced pre-blending method: the rejuvenator is pre-blended into the virgin binder, then foamed with water to mix with the virgin aggregate and RAP.
- Foaming-enhanced pretreatment method: the rejuvenator is foamed with water to pretreat the RAP, then mixed with the virgin binder and virgin aggregates.
- Emulsion-enhanced pretreatment method: the emulsified rejuvenator is applied to the RAP as pretreatment, then mixed with the virgin binder and virgin aggregates.

The study also sought to 1) optimize the foaming conditions of the rejuvenators and rejuvenated asphalt binders; 2) determine the effects of RAP pretreatment on the quality of the

material; 3) evaluate the impact of marination on RAP pretreated with rejuvenator on the quality characteristics of the RAP material; and 4) determine the workability, durability, intermediate-temperature cracking resistance, and thermal cracking resistance performance due to rejuvenator incorporation method for high RAP asphalt mixtures.

Chapter 2: Literature Review

2.1 State of the Practice: Current Usage of Rejuvenators in High RAP Mixtures

Utilizing rejuvenators when designing asphalt mixtures is becoming more common in the United States as a method of building more economical and environmental pavements without sacrificing mixture performance. A National Road Research Alliance (NRRA) survey indicated that of the seven states that responded, only three state Department of Transportations (DOT) had experience in rejuvenators in asphalt mixtures utilizing RAP material (Blanchette et al., 2020). Among those three states, five different products had been utilized, with the majority being biobased oils. Among the states that responded to the survey, the maximum RAP usage allowed in new asphalt pavements without rejuvenators varies by state regulation. Many states limit RAP content by placing a cap on maximum allowable binder replacement ratio. Other states limit even further by adding RAP limits by mixture type (surface versus base layers), asphalt grade (binder grade and aggregate gradation specifications). With the use of rejuvenators, three of the seven states reported that RAP content limits do not change. In comparison, the other four states reported that there is no specification for rejuvenator usage with RAP. One state did not specifically limit the RAP content in unrejuvenated mixtures but blending charts for higher amounts (greater than 25%, as per American Association of Highway and Transportation Officials (AASHTO) M323) are required along with passing all volumetric mixture design requirements. State usage of rejuvenators in high RAP mixtures is increasing, with this survey reporting a need for guidance from research, rejuvenator manufacturers, and DOTs on rejuvenator usage, especially in terms of rejuvenator dosage, RAP content limits with rejuvenator utilization, and rejuvenator incorporation methodology.

While the state DOTs and asphalt pavement contractors have limited experience with rejuvenator usage in high RAP mixtures, it remains a field of further study, as synthesized by Kaseer, Epps Martin, and Arámbula-Mercado (2019). The researchers found that the main reasons for the lack of rejuvenator incorporation in high RAP mixtures were: 1) the deficit in rejuvenator type selection and dosage, 2) the lack of test methodology for characterizing rejuvenator products, 3) the lack of expertise in rejuvenator blending methodology into asphalt mixtures, 4) expertise deficiency in evaluating rejuvenator effectiveness, 5) an incomplete understanding of long-term aging impacts of rejuvenator products, and 6) a lack of knowledge on economic benefits of rejuvenators. A 2015 web survey of state DOTs, paving contractors, and rejuvenator suppliers by Epps Martin (2015) found that 80% of the state DOTs that responded did not allow recycling agents, including rejuvenators and softening agents. On the contractor side, 64% of respondents reported that recycling agents were not used mainly due to a lack of product experience and knowledge in dosage optimization. In general, 60% of respondents indicated that tall oils and biobased oils were the most common recycling agents, and more than 80% of respondents reported that test methods for characterizing recycling agent effectiveness were based on binder testing such as penetration grading, kinematic viscosity, or performance grading (PG).

The synthesis report (Kaseer et al., 2019) also reported on the methods of rejuvenator incorporation, stating that the mechanism of rejuvenation is three-fold: uniform dispersion of the virgin binder, recycled binder, and rejuvenator throughout the mixture; diffusion of the rejuvenator into the recycled binder; and compatibility between virgin binder, recycled binder, and the rejuvenator. The dispersion and diffusion of the rejuvenator into the asphalt mixture are impacted by the incorporation method, with an expectation of better diffusion when the applying rejuvenator directly to recycled asphalt materials before mixing with virgin materials. However, the synthesis found that Tran, Taylor, and Willis (2012) reported that most asphalt plants in the U.S. blend the rejuvenator into the virgin binder before mixing. Contrary to the U.S., Japan utilizes rejuvenators

often by mixing and conditioning the recycled asphalt material with the RA hours prior to mixing, as surveyed by West and Copeland (2015). While these methods could increase rejuvenator effectiveness, application in the U.S. requires significant asphalt plant modifications with alternative equipment, which has implementation challenges from the practicality perspective.

The synthesis report (Kaseer et al., 2019) found that recycling agent dosage also impacted the mixture performance of high RAP mixtures, as dosage must improve cracking resistance without detriment to rutting resistance. Typically, the dosage is selected based on experience or at the recommendation of the recycling agent manufacturer. Within those parameters, methods vary further, with some researchers dosing based on blending charts, while others set maximum dosages by targeting a specific high temperature PG and minimum dosages by targeting a particular low temperature PG. The effectivity of the recycling agent and the dosage are affected by the source of the recycled asphalt material, as less aged RAP material tends to require a lower recycling agent dosage. Mixture performance of rejuvenated high RAP mixtures tend to have high rutting resistance but develop cracking stress more quickly than virgin mixtures. Again, dosage and application method become vital to properly revitalize recycled binder properties, along with studying the long-term impacts of the recycling agents on mixture performance.

2.2 Rejuvenator Application Methods

Currently, the most common incorporation method for liquid RAs is to pre-blend into the virgin binder prior to mixing with the virgin aggregate and RAP. However, this may limit full interaction between the rejuvenator and the RAP. Therefore, methods of pretreating the RAP before hot mix asphalt (HMA) production should be considered if agent addition aims to rejuvenate the aged RAP binder.

Though not extensively, multiple studies have considered this, with various mixing methods to simulate potential pretreatment in an asphalt production plant. Researchers (Xie et al.,

2019) found that the rejuvenator incorporation method significantly impacted the air voids of a 19.0mm NMAS Superpave mixture containing 50% RAP. This study evaluated three different rejuvenators, with dosage determined by manufacturer experience, with 12% by weight of RAP binder for all rejuvenators. NCAT (2014) separates rejuvenators into five groups: paraffinic oils, aromatic extracts, tall oils, naphthenic oils, and triglycerides and fatty acids (derived from vegetable oils). This study by Xie used a rejuvenator from the paraffinic group, the tall oil group, and the triglyceride and fatty acid group. Each rejuvenator was evaluated using three mixing methods: the first to simulate addition to the heated RAP; the second to simulate spraying application to RAP prior to mixing; and the third to simulate the rejuvenator pre-blending into virgin binder incorporation method. This study focused on the rejuvenator incorporation method's impacts on volumetric properties. Samples were aged for 2 hours at 150 °C. From this study, the incorporation method impacted the voids in the mineral aggregate (VMA) only for the triglycerides and fatty acids group rejuvenator. The other rejuvenators assessed showed no impact. While the volumetric property impacts are vital for high RAP mix designs, this study did not consider performance aspects of the mixture or long-term aging impacts.

Another study by Xie et al., 2020 assessed the performance of RAP mixtures, focusing on resistance to raveling and cracking. Four different methods of rejuvenator incorporation were considered using two mixtures, one with 40% RAP and the other with 25% RAP + 5% RAS. The first method simulated the traditional pre-blend into virgin binders, the second simulated spray application on the belt line of an asphalt plant, and the last two methods simulated marination pretreatment, with spray application and then 48-hour or 14-day marination in closed bags at ambient temperature. As with the previous study, pretreatment methods only consider spray pretreatment without a dispersion agent. Results of this study assessed the durability of the mix

with the Cantabro mass loss test, and cracking resistance with the Texas overlay test (OT), the Illinois flexibility index test (I-FIT), and the disc-shaped compact tension test (DCT). The study also evaluated five different rejuvenators for rejuvenating effectiveness with Dynamic Shear Rheometry (DSR) testing on blended binder samples, which are composite virgin binder, extracted RAP binder and rejuvenator blends, with the best performing one selected for mixture analysis. Using a tall oil, the 40% RAP mixture utilized a 5% by weight of total recycled binder dosage, while the other mixture utilized a 13.4% by weight of total recycled binder dosage. The results of the 40% RAP mixture testing found that rejuvenator usage in general improved the durability of the mixture, with short-term oxidative aging (STOA) showing no difference in performance between incorporation methods. In contrast, the long-term oxidative aging (LTOA) results displayed that the marinated conditions were statistically significant from the control, pre-blend method, and belt spray method. Regarding intermediate-temperature cracking resistance for the 40% RAP mixture, rejuvenator application improves the I-FIT flexibility index; however, there was no significant difference between methods in either aging condition. All application methods improved the number of cycles to failure with reflective cracking resistance assessed through the OT with the 40% RAP mixture, though there was no statistical significance in either aging condition. Thermal cracking resistance was assessed using the DCT, the application of rejuvenator for the 40% RAP mixture slightly improved fracture energy, but there was no statistical difference between the control and experimental mixtures under both aging conditions. This study was thorough in assessing multiple mixture performance parameters, but the methods of application were limited as no dispersal agent was used, such as emulsified rejuvenator or foamed rejuvenator.

In a study by Ruthore and Zaumanis (2020), a tall oil-based rejuvenator was assessed with a 60% RAP mixture with multiple mixing methodologies to assess the indirect tensile strength

(ITS) and stiffness modulus, along with aging due to laboratory mixing. This study considered three rejuvenator incorporation methods, the first being spray application, with a 2-hour and a 24hour rest period, and the last being the traditional pre-blend into the virgin binder. Additionally, the effects of mixing time and temperature were assessed, with 2, 4, and 7 minutes of mixing time and 130 °C, 155 °C, and 180 °C for mixing temperatures evaluated. This study also evaluated the effect of a small mixer versus a larger mixer to understand the differences in laboratory-produced mixes and asphalt plant mixers. In addition to the ITS testing and the stiffness modulus testing (EN 12607-26), the asphalt binder was extracted and recovered from the produced mixes to evaluate via DSR and Fourier transform infrared spectroscopy (FTIR). From the testing results, none of the rejuvenation methods showed a statistically significant improvement in stiffness modulus, though all methods did reduce mixture stiffness. The mixing temperature increased the stiffness of the mixtures while mixing time seemed to have no impact. Additionally, the stiffness results found that the degree of blending between the RAP binder and virgin binder changed based on mixing equipment. While this study successfully characterized aspects of the mixing process that impact stiffness and degree of blending, it only considered spray application and room temperature marination regarding the incorporation method. This study also focused on laboratory mixing methods and did not extend to actual asphalt plant practices.

The effect of different rejuvenator incorporation methods in a batch asphalt plant was assessed by Zaumanis (2019), considering ten separate addition sites to optimize the best performing locations in terms of mixture performance and plant efficiency. Mixture performance testing was evaluated on only two of the ten locations, spraying rejuvenator on the RAP conveyor belt ahead of the dryer and applied in the mixer itself to contrast the control specimens without rejuvenator, using stiffness modulus (EN 12697-26) testing, fatigue testing (EN 12697-24), and

Semi-Circular Bending (SCB) (AASHTO TP 124-16) testing. Rejuvenator dosage was 5% by total binder mass, though accurate rejuvenator dosage varied slightly, as asphalt plants allow binder content to change by 0.5%, meaning the rejuvenator content varies with the binder content. With that in mind, the actual binder contents and rejuvenator dosages were back-calculated postproduction for comparison, and both rejuvenator and binder contents were within variation limits. From the stiffness testing, the addition of the rejuvenator reduced the complex modulus across the frequency spectrum; however, there was no significant difference between the incorporation methods tested. Fatigue testing displayed the spray treatment onto the cold RAP belt having increased resistance over the mixer incorporation method; however, the control specimens were not assessed, so the degree of increased fatigue resistance was unknown. Finally, the intermediatetemperature cracking resistance of the mixture increased with the addition of the rejuvenator, with no significant difference between the addition methods. From this study, it is seen that in a batch asphalt plant, alternative rejuvenator incorporation methods can improve mixture performance. However, this study is limited, as it only considered alternative methods in the performance testing but not the traditional pre-blending method of rejuvenator incorporation. Additionally, this study did not consider the impacts of long-term aging on the mixture cracking performance evaluation.

Overall, these previous studies did not consider the rejuvenator incorporation method's impact on RAP quality, foaming application methods, and marination effect on high RAP content mixtures. While the mixture performance has been assessed for multiple mixing methods, assessing RAP quality and subsequent improvement from pretreatment has not. RAP quality improvement can enhance overall mixture workability and blending of RAP binder with the virgin binder. Foaming application for RAP pretreatment and the rejuvenated binder offers better dispersion of the "wet" material components, allowing for a better coating of the "dry" materials.

The marination effect of pretreating RAP only considered a 40% RAP mixture with room temperature marination. Considering these points, a more comprehensive understanding of the RAP pretreatment effect can be gained.

2.3 RAP Quality Assessment Methods

RAP quality assessment and understanding are vital for mix design and tend to focus on the gradation of the material and the extracted and recovered binder grade. The material's gradation can be controlled with fractionating the material into the different sieve sizes. Many state DOTs require extracted binder gradation to characterize the quality and consistency of RAP stockpiles. However, the recovered binder grading process is time-consuming and can be costly for contractors who do not have the proper equipment for binder performance grading. Additionally, the solvents used for extraction can be expensive and environmentally hazardous if not disposed of properly. Yet, this quality characterization of the recovered binder is vital for rejuvenator dosage determination, as discussed in subchapter 1.2.1.

The Dongré Workability Test (DWT) offers an alternative method for determining RAP quality by considering the coupled impacts of the RAP binder content, RAP binder quality, and the RAP aggregate material properties and gradation. Dongre, Li, and Youtcheff (2021) found that the DWT parameter could be easily determined using existing equipment, a Superpave Gyratory Compactor (SGC), with repeatability and reproducibility at ±5% and ±10%, respectively. Additionally, the DWT was able to differentiate between different RAP sources over multiple test temperatures, as seen in Figure 1. Overall, the preliminary results from the test development found that 1) the DWT can characterize loose RAP with low variability, 2) the DWT displays RAP behavior as a function of temperature and interfacial coating, and 3) the DWT shows differences between RAP sources and ages. Although this test is still in development, it holds great potential

as a ranking tool between rejuvenator pretreatment methods for the same RAP source. The test methodology and testing parameters are discussed further in Chapter 2.

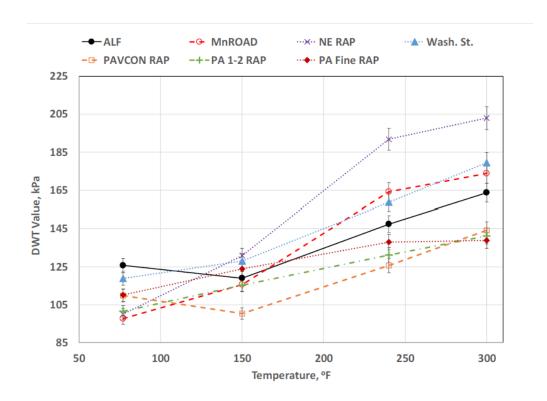


Figure 1. Behavior of DWT Value over Temperature for Different RAP Sources (Dongre et al., 2021)

Chapter 3: Research Methodology

The objectives of this study were achieved by separating the overall experimental plan into four phases, as summarized below.

The first phase of the experimental plan focused on assessing and then optimizing the foaming characteristics of the rejuvenators and rejuvenated virgin asphalt binders. Volume expansion was measured using a laser distance meter, and those results were utilized to calculate foaming index parameters. From the calculations, the optimum foaming condition of each material was chosen for further assessment in RAP pretreatment and in creating high RAP content mixtures for performance testing. This *Foaming Optimization* phase is discussed further in section 2.2.

The second phase of the experimental plan assessed the impact of RAP pretreatment with a rejuvenator on RAP quality. In contrast with the non-pretreated (control) RAP, three pretreatment methods were evaluated: spray-on, emulsion, and foaming applications. The application methods were assessed using the DWT, the Cantabro test, and grayscale image analysis. Additionally, the moisture content of the control and pretreated RAP samples was monitored. This *RAP Pretreatment* phase is further discussed in section 2.3

Using the application method that showed the most promising RAP quality improvement from the *RAP Pretreatment* phase, the impact of marination on RAP quality was assessed in the third phase of the experimental plan. Short-term and long-term marination conditions were assessed, with the former occurring at elevated temperatures and the latter at ambient temperatures for four marination conditions. Once the conditions were achieved, the marination effect was evaluated using DWT, Cantabro testing, and grayscale image analysis. This *RAP Marination* phase is further discussed in section 2.4.

The last phase of the experimental plan focused on the impact of the rejuvenator application method on high RAP content HMA performance. A total of five mixes per mix design were assessed, with four alternative mixing methods analyzed: pre-blending rejuvenator into the virgin binder, pretreating RAP with emulsified rejuvenator, pretreating RAP via foamed rejuvenator, and foaming the pre-blended virgin binder with rejuvenator. These alternative methods were compared with the control mix, which contained no rejuvenator. The DWT and Cantabro testing were used for workability and durability evaluation, respectively. To evaluate intermediate-cracking temperature resistance, the IDEAL-CT and I-FIT tests were used. Finally, low temperature cracking resistance was assessed by the DCT test. This *Mixture Performance* phase is discussed further in section 2.5.

3.1 Material Selection and Mix Design

For the high RAP mixtures to be evaluated, three aspects are required: the overall mixture design, the RAP sourcing, and the rejuvenator type and dosage. Two mix designs were assessed, with mix design A being a 9.5mm NMAS Superpave mixture with 45%RAP and mix design B being a 12.5mm NMAS Superpave mixture with 50% RAP. The recycled binder ratios (RBR) of these mixtures were 0.40 and 0.51, respectively. Additionally, mix design A utilized a PG 67-22 virgin binder to emulate mix designs in southern states, while mix design B used a PG 58S-28 virgin binder to emulate mix designs in northern states. The summary of these mix designs can be seen in Table 1.

Table 1. High RAP Asphalt Mix Design Summary

		MIX DESIGN A	MIX DESIGN B
N	MAS (MM)	9.5	12.5
VIRGIN BINDER		PG 67-22	PG 58S-28
RBR		0.40	0.51
MIXTURE ASPHALT CONTENT (%)		5.9	5.6
VMA (%)		16.9	15.5
EFFECTIVE A	SPHALT CONTENT (%)	5.62	4.92
DUST :	BINDER RATIO	1.12	1.68
BULK SPECIFIC	GRAVITY OF AGGREGATE	2.659	2.657
	GRADATION		
SIEVE (MM)	SIEVE #	PERCENT P	ASSING (%)
19	3/4"	100.0	100.0
12.5	1/2"	100.0	97.8
9.5	3/8"	97.0	90.8
4.75	#4	76.0	61.1
2.36	#8	53.4	44.9
1.18	#16	40.9	37.2
0.6	#30	30.6	30.9
0.3	#50	16.5	23.1
0.15	#100	9.7	15.0
0.075	#200	6.3	8.3

Along with the two mix designs, two RAP sources were considered, subsequently referred to as RAP A and RAP B, corresponding to mix design A and mix design B, respectively. RAP A was sourced from Alabama with the extracted and recovered binder graded PG 100+2, with a true grade of PG 105.0+2.1. RAP B was sourced from Georgia, with the extracted and recovered binder graded PG 94-4, with a true grade of PG 98.4-6.0. RAP binder contents were determined using the ignition method. All extracted binders were recovered according to AASHTO T164 and American Society for Testing and Materials (ASTM) D5404 using trichloroethylene (TCE). Additionally, Delta T_C (ΔT_C) was evaluated on each extracted and recovered RAP binder. This parameter provides insight into the relaxation properties of an asphalt binder which impacts non-load-related

cracking, and other age-induced embrittlement distresses (Asphalt Institute Technical Advisory Committee, 2019). The full summary of these two RAP sources can be seen below in Table 2.

Table 2. RAP Source Properties Summary

		RAP A	RAP B
NMAS	(MM)	9.5	9.5
RAP BINDER CONTENT (%)		5.30	5.72
RAP BINDER		PG 100-(+2)	PG 94-4
ΔT _C AFTER RTFO+20HR PAV AGING		-7.3 °C	-13.4 °C
	BLACK RO	OCK GRADATION	
SIEVE (MM)	SIEVE #	PERCENT PASSING (%)	
19	3/4"	100.0	100.0
12.5	1/2"	100.0	99.0
9.5	3/8"	98.0	97.0
4.75	#4	82.0	83.0
2.36	#8	65.0	69.0
1.18	#16	54.0	58.0
0.6	#30	42.0	48.0
0.3	#50	25.0	37.0
0.15	#100	13.0	24.0
0.075	#200	8.1	13.5

The third aspect of this study was the rejuvenators used to evaluate of mixing methods. Two bio-based liquid rejuvenators were chosen for this study and are referred to as RA1 and RA2, corresponding to mix design A and mix design B, respectively. Emulsified versions of each rejuvenator were utilized in the study's RAP pretreatment and mixture performance assessment portions, with RA1 containing 40% water by weight and RA2 containing 30% water by weight. Emulsified rejuvenator products were produced by the individual manufacturer. Dosage determination methodology varied for each rejuvenator and subsequent mix design. For mix design A and RA1, the manufacturer suggested a dosage of 16.1% by weight of RAP binder to target 76

°C as the high temperature PG of the rejuvenated RAP binder. This dosage translates to 10.8% by weight of virgin binder and 0.85% by weight of RAP. For mix design B and RA2, the manufacturer suggested a dosage of 6.0% by weight of RAP binder to target the low temperature true grade of a corresponding 20% RAP binder blend based on theoretical blending chart evaluation. This dosage translates to 6.3% by weight of virgin binder and 0.34% by weight of RAP. The rejuvenators and the subsequent emulsions can be seen in Figure 2.





Figure 2. Rejuvenators and Emulsified Rejuvenators

3.2 Foaming Optimization

This portion of the *Research Methodology* focuses on the foaming characteristic assessment of the rejuvenators and rejuvenated asphalt binders. For RAP pretreatment and the mixture performance assessment phases of the study, the optimum foaming condition at a specific

temperature and water content must be determined. In this study, three temperatures were assessed for the rejuvenators and rejuvenated asphalt binders: 110 °C, 120 °C, 130 °C, and 130 °C, 140 °C, and 150 °C, respectively. Across all temperatures assessed, three water contents were considered: 1%, 2%, and 3%.

The Wirtgen WLB 10s laboratory foaming unit was used for the foaming optimization phase, targeting 200 g for each foaming shot. For each foaming condition assessed, two repeatable and high-quality shots were performed, but tester discretion was utilized for total number of foaming shots. Expansion measurements were conducted using a laser distance meter connected to the Wirtgen WLB 10s laboratory foaming unit, as seen in Figure 3. The laser recorded the real-time distance and time data and instantly transferred the points to a computer spreadsheet via Bluetooth, as seen in Figure 4. From this data, an expansion curve over time could be generated to determine the critical foaming parameters: maximum expansion ratio (ER_{max}), expansion half-life ($t_{1/2}$), and foamability index (FI).

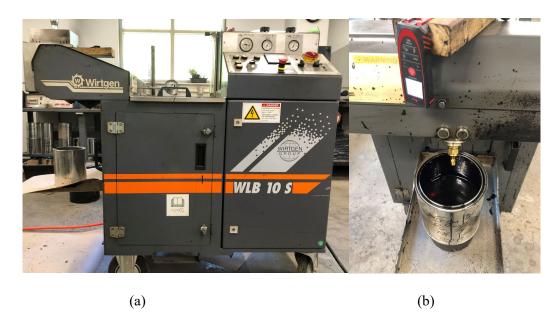
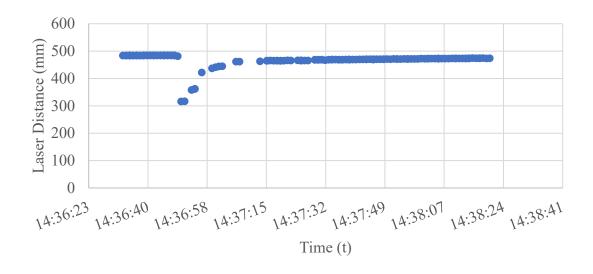


Figure 3. Foaming Optimization Setup: (a) Wirtgen WLB 10s Laboratory Foaming Unit and (b) Laser

Distance Meter



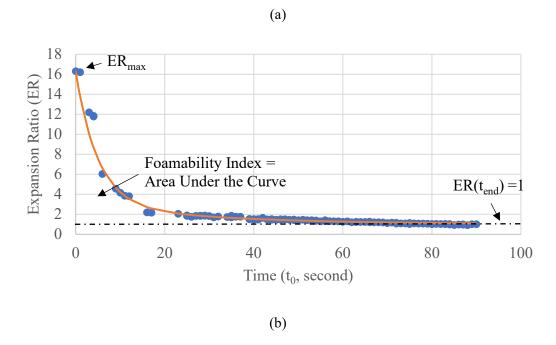


Figure 4. Foaming Optimization Analysis: (a) Measured Laser Distance Data and (b) Expansion Ratio

Data Processing

Expansion ratio (ER) is a key foaming parameter to express the degree of volume expansion of the material at a specific foaming condition, and the maximum value is referred to as

the ER_{max}. The half-life of the volumetric expansion ($t_{1/2}$) indicates the relative stability of the expansion. It is defined at the time required for the expansion to collapse to half of the peak expansion. Combining the ER and the $t_{1/2}$, the foamability index (FI) evaluates the entire expansion and collapse of the foamed material over time. As seen in Figure 4b, it is defined as the area under the fitted ER curve. As a trend, a higher ER, $t_{1/2}$, and FI represent better foaming characteristics. However, ER and $t_{1/2}$ are typically in an inverse relationship, with higher ER values being less stable and collapsing faster, i.e., a smaller $t_{1/2}$. Thus, FI became the optimizing parameter for this study, as it considers the expansion of the foam and the stability of the foam.

Data processing of the collected laser distance meter utilized the following method. First, for each laser distance data point, as seen in Figure 4a, an equivalent ER could be calculated according to Equation 1.

$$ER(t) = \frac{h_{initial} - h(t)}{h_{initial} - h_{final}}$$
 [Equation 1]

Wherein, ER(t) is the ER at time t, $h_{initial}$ is the initial height of the foam, h(t) is the height of the foam at time t, and h_{final} is the final height of the foam.

The highest value given by iterating this equation through the data generated in each foaming application shot is the ER_{max} and is treated as the peak of the expansion. Post the peak expansion, an exponential curve can be generated to fit the data, according to Equation 2 and as seen in Figure 3b.

$$ER(t_0) = ae^{-bt_0} + ce^{-dt_0}$$
 [Equation 2]

Wherein, t_0 is the time in seconds post-ER_{max}, and a, b, c, and d are fitting coefficients.

After the fitting coefficients are calculated, the $t_{1/2}$ could be found by setting the right side of Equation 2 equal to half of ER_{max}. Lastly, the FI could be found by integrating Equation 2 over

time, from the peak (t_0) to the end time of the foaming application shot (t_e) , with a baseline of ER = 1. This integration can be seen in Equation 3.

$$FI = \int_{t_0}^{t_e} ER(t)dt = \int_{t_0}^{t_e} (ae^{-bt} + ce^{-dt} - 1)dt$$
 [Equation 3]

The foaming condition that gives the highest FI is treated as the optimum foaming condition for that material.

3.3 RAP Pretreatment Testing

The goal of the *RAP Pretreatment* phase was to assess the impact of pretreating RAP versus untreated RAP using three methods of rejuvenator application: spray-on, emulsion, and foaming. For RAP A, all pretreatment applications used the same RA1 dosage of 0.85% by RAP weight. For RAP B, the same dosage of RA2 was used for all pretreatment methods: 0.34% by RAP weight. Prior to pretreatment with a rejuvenator, the RAP was dried and split to maintain homogeneity. RAP pretreatment occurred using the Wirtgen pugmill, as seen in Figure 5. Approximately 20,000g to 30,000g of RAP were used for each pretreatment trial.



Figure 5. Wirtgen Pugmill Utilized in Pretreatment

A spray bottle was used to apply the rejuvenating product for the spray-on pretreatment method. While the pugmill was running, the product was sprayed through the opening on the cover

of the pugmill. The pugmill was run for a total of 120 seconds. For the emulsified rejuvenator application, the product was poured on top of the RAP in the pugmill in a "zig-zag" motion. After the application, mixing inside the pugmill was stopped after 120 seconds. Foaming pretreatment application was performed at the optimized foaming condition for RA1 and RA2 for RAP A and RAP B, respectively. Using the foaming nozzle for the foaming unit, the application began at approximately 10 seconds of pugmill mixing, with a total of 120 seconds of pugmill mixing to keep consistency with other application methods.

After pretreatment, both untreated and pretreated RAP for both sources was assessed for quality characterization by the DWT, Cantabro testing, and grayscale image analysis. Additionally, moisture content was monitored as the emulsified rejuvenator products contained a considerable amount of water, 40% by weight for RA1 and 30% by weight for RA2.

The DWT conducted for the RAP quality assessment focused on the impact on workability due to pretreatment. For each test, 4200g of material was tested in a Superpave Gyratory Compactor (SGC) at target test temperatures of 240 °F and 300 °F, with two samples at each temperature. Utilizing consistent compaction force in the SGC, without gyrating, a constant loading rate of 0.05 mm/second was applied to the sample until 700 kPa of stress was reached. Throughout the test, pressure and height data were collected every 0.1 seconds. The RAP workability can then be analyzed from the slope of the stress versus volumetric strain curve, with the DWT value measurement seen in Figure 6. The slope at 600 kPa is the test parameter from the DWT, and it is calculated by the ratio of the change in stress between 650 kPa and 550 kPa to the change in volumetric strain through the same pressure change, as seen in Equation 4.

$$DWT_T = \frac{\sigma_{650} - \sigma_{550}}{\varepsilon_{650} - \varepsilon_{550}}$$
 [Equation 4]

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_{650} are the volumetric strain (%) at the nearest index to 650 kPa and 550 kPa pressure, respectively.

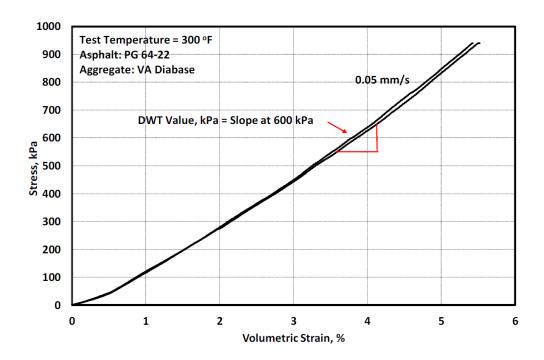


Figure 6. DWT Value Calculation from SGC Stress Versus Volumetric Strain Curve (Dongre et al., 2021)

In general, a higher DWT value implies better workability, and thus a better RAP quality. Using samples compacted for the DWT test, the Cantabro test was run on all samples compacted at 240 °F in a Los Angeles abrasion (LAA) drum, as seen in Figure 7. After Corelok air void measurement, samples were conditioned overnight for a minimum of 4 hours at 25 °C. Air voids trended between 14% and 17%. Sample mass was weighed prior to the test and after the 10-minute test. From there, the mass loss could be calculated according to Equation 5.

$$CML$$
 (%) = $\frac{Mass\ Initial - Mass\ Final}{Mass\ Initial} * 100$ [Equation 5]



Figure 7. Cantabro Testing Apparatus

The objective of running the Cantabro test was to evaluate the impact of the rejuvenator addition method for both RAP sources on the durability of the untreated and pretreated RAP.

Additionally, the degree of rejuvenator distribution was assessed using grayscale image analysis of the resulting pretreated RAP. Approximately 2,000g of untreated or pretreated RAP sample was first scanned using an office scanner, as seen in Figure 8. Sample thickness uniformity was controlled by using a wooden frame for each scan. External light was blocked from interfering with scanning trials by placing a paper cover over the wooden frame. Scans were then processed through MATLAB for grayscale pixel analysis.



Figure 8. Grayscale Image Analysis Apparatus

Analysis was programmed to assign a value of zero (0) to fully black pixels and a value of 250 to fully white pixels. The distribution of the grayscale values for the scanned RAP image were then computed and analyzed graphically, as shown in Figure 9. Improved RAP quality is seen through less variability in pixel value (i.e., a narrower curve) and a lower average grayscale value. This indicates that the RAP color is darker overall, implying possibly increased RAP binder activation.

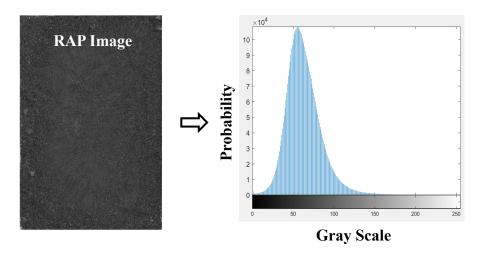


Figure 9. Grayscale Image Analysis

Moisture content was monitored for each pretreatment method, as both the emulsion and foaming pretreatment methods utilize water. RAP material was stored at room temperature and measured up to 7-days of storage after pretreatment.

3.4 RAP Marination Testing

From the results of the pretreatment study, the best performing method of pretreatment (i.e., emulsion) will be selected for the *RAP Marination* phase on RAP A only. The goal of this phase was to assess the impact of marination with rejuvenator on RAP quality. Four marination conditions in total were evaluated. Two conditions assessed accelerated, high temperature conditions, marinating at 135 °C for 1.5 hours and 3 hours. The other two conditions considered a longer term, ambient temperature marination, at room temperature (~25°C) for 3 days and 7 days. Once the marination condition was achieved, the pretreated RAP was evaluated for quality characteristics using the DWT, the Cantabro test, and grayscale image analysis, utilizing the same procedures and analysis methodology from the *RAP Pretreatment* phase in subchapter 2.3.

3.5 Mixture Performance Testing Plan

This phase evaluated the high RAP content mixture performance by rejuvenator addition method from three fronts: workability, durability, intermediate temperature cracking resistance, and low temperature cracking resistance. Mixture workability assessment utilized the DWT, while mixture durability focused on the Cantabro testing. The IDEAL-CT and the I-FIT assessed intermediate-temperature cracking resistance. Low temperature cracking resistance was assessed through DCT testing.

DWT mixture testing followed the same methodology as the RAP quality assessment as discussed in subchapter 2.3, except that 4800g of the loose mixture is used. Before DWT testing,

the loose mixture was conditioned for two hours at compaction temperature as described for the short-term aging procedure in AASHTO R 30. Again, testing temperatures were 240 °F and 300 °F, with two replicates for each temperature. Similar to RAP quality DWT testing, a higher DWT value indicates better mixture workability.

Cantabro testing evaluated mixture durability and utilized the 240 °F compacted samples from the DWT evaluation. The testing procedure followed the same methodology as the RAP quality assessment in subchapter 2.3.

Intermediate-temperature cracking resistance was evaluated through the IDEAL-CT and I-FIT tests. IDEAL-CT samples were short-term aged for 4 hours at 275 °F, according to AASHTO R 30, and then long-term aged for 6 hours at 275 °F. Following ASTM D8225, samples were gyratory compacted to 62 mm heights and 7.0±0.5% air voids, then tested with a monotonic load at a constant displacement rate of 50 mm/minute. Mixtures were assessed after 2 hours of 25 °C conditioning, with a minimum of four replicates tested. Data analysis and performance assessment focused on the load-displacement curve, with attention to the fracture energy, the slope of the curve at various loading points, the displacement over the test, and other interim parameters. A sample testing apparatus can be seen in Figure 10. The test parameter developed from this analysis is the cracking tolerance index (CT_{Index}), calculated using Equation 6.

$$CT_{Index} = \frac{t}{62} * \frac{I_{75}}{D} * \frac{G_f}{|m_{75}|} * 10^6$$
 [Equation 6]

Wherein, t is the sample thickness, I_{75} is the post-peak displacement at 75% of the peak load, D is the sample diameter, G_f is the fracture energy, and $|m_{75}|$ is the post peak slope at 75% of the peak load.

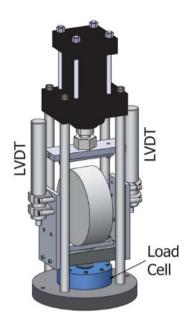


Figure 10. Sample IDEAL-CT Apparatus (ASTM D8225 -19)

The I-FIT test was conducted per a modified AASHTO T 393 procedure due to utilizing IDEAL-CT samples, at the same aging condition as the IDEAL-CT testing, with short-term aging at 275 °F for 4 hours, followed by long-term aging at 275 °F for 6 hours. Samples were cut to have a radius of 70 to 75 mm, a notch of 15.0±1.0 mm deep and 1.5±0.5mm wide, and a thickness of 62 mm. To account for the specification requirement of 50 mm thickness, the work of fracture is backcalculated from the reported fracture energy, as seen in Equation 7, and then applied to the flexibility index equation (Equation 8) with the actual measurements. Additionally, a correction factor was applied for specimens that did not meet the specification of 7.0±0.5% air voids, as seen in Equation 8. Data analysis focused again on the load-displacement curve, utilizing the work of fracture and post-peak slope of the curve. A higher flexibility index indicates a better intermediate-temperature cracking resistance. The testing apparatus can be seen in Figure 11.

$$W_f = G_f * 50 * 60$$
 [Equation 7]

Flexibility Index =
$$\frac{\frac{W_f}{(r-d_n)*t}}{|m|} * A * \frac{0.0651}{V_Q - V_q^2}$$
 [Equation 8]

Wherein, W_f is the work of fracture, G_f is the fracture energy, r is the radius of the specimen, d_n is the notch depth, |m| is the absolute value of the post-peak slope, A is a scaling factor equal to 0.01, and V_a is the air voids as a decimal.

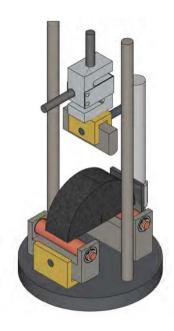


Figure 11. Sample I-FIT Apparatus (AASHTO T 393 - 21)

The DCT test, utilizing ASTM D7313, characterized the low temperature cracking resistance for asphalt mixtures. Similar to the IDEAL-CT and I-FIT testing, samples were short-term aged for 4 hours at 275 °F, then long-term aged for 6 hours at 275 °F. Testing occurred at -18 °C with six replicates. From 160 mm gyratory samples, two specimens were cut with 50±3.5 mm thickness, then trimmed to give a flat edge in which a 62.5±5.0 mm notch was created. Then, two 25±1.0 mm diameter holes were drilled on each side of the notch. Once conditioned to temperature, the specimen is loaded with tension by inserting metal rods through the drilled holes. A clip gage was attached to the crack mouth to control and record the crack mouth opening displacement (CMOD). The clip gage controlled the opening at a rate of 0.017 mm/second, and the test ended when the load fell below 0.1 kN. The test parameter developed from this test is the fracture energy,

with Equation 9 showing the calculation. Figure 12 shows a sample testing apparatus. For better low temperature cracking resistance, a higher G_f is preferred.

$$G_f = \frac{AREA}{B*(W-a)}$$
 [Equation 9]

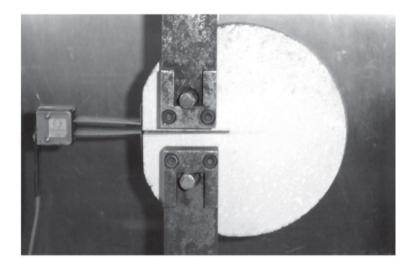


Figure 12. Sample DCT Apparatus (ASTM D7313 – 20)

Wherein, G_f is the fracture energy, AREA is the numerically integrated area under the load-CMOD curve, B is the specimen thickness, and W-a is the initial ligament length.

All performance tests were assessed with statistical analysis, such as mean and standard deviation. All the mixture cracking tests utilized mean value analysis and Games-Howell post-hoc statistically grouping analysis with a 0.05 significance level.

Chapter 4: Foaming Optimization Results

This chapter covers the results of the *Foaming Optimization* phase of the testing plan, as discussed in section 2.2. The objective of the section was to determine the optimum foaming conditions for the two rejuvenators (RA1 and RA2) and the two rejuvenated asphalt binders (PG 67-22 + RA1 and PG 58S-28 + RA2). The foaming experiment assessed multiple temperatures, with the rejuvenators at 110 °C, 120 °C, and 130 °C, and the rejuvenated asphalt binders at 130 °C, 140 °C, and 150 °C. Each temperature was evaluated at multiple water contents: 1%, 2%, and 3%. As previously discussed, the data analysis focused on the FI results, as it balanced the volume expansion and collapse behavior. A higher FI indicates increased volume expansion while also maintaining good foam stability.

4.1 Foamed Rejuvenator Optimization

The results for the FI of RA1 at the tested foaming conditions can be seen in Figure 13. For foaming conditions at 120 °C and 130 °C, the foamability index increased with higher water content, indicating that at these temperatures, foam quality and stability improved with higher water content. At the lower temperature (110 °C), the trend is the opposite, with FI (i.e., foam quality) decreasing with higher water content. The optimum condition was determined at 120 °C with 3% water content, as this condition had the highest FI. At this condition, the average ER_{max} was 6.3 and the average t_{1/2} was 8.8 seconds.

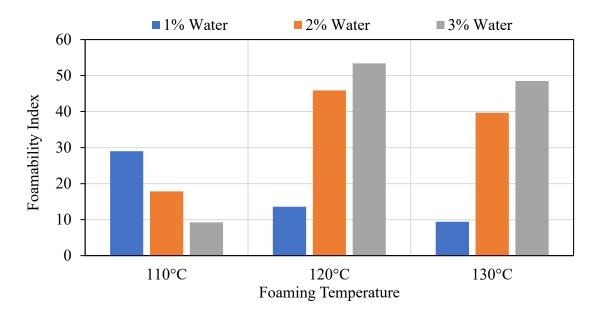


Figure 13. RA1 Foaming Results

Figure 14 shows the FI results of RA2 at the tested foaming conditions. All temperature conditions showed the same trend of increased foam quality with water content. Most notably, the 110 $^{\circ}$ C and 1% foaming condition gave no volume expansion, resulting in a "N/A" label in the figure. The optimum foaming condition for RA2 occurred at 130 $^{\circ}$ C with 3% water content. This condition gave the highest FI, with an average ER_{max} of 7.9 and an average $t_{1/2}$ of 5.7 seconds.

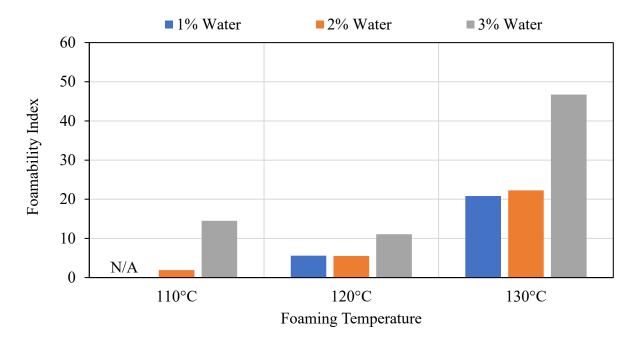


Figure 14. RA2 Foaming Results

Comparing the two rejuvenators, the optimum condition for RA2 gave better volume expansion than RA1 but had a shorter half-life, indicating less stability. Overall, RA2 had a lower foaming quality than RA1, as seen by the FI results. Both rejuvenators are modified bio-based oils, so these differences in results could be due to the different chemical composition and manufacturing processes of the two products. Water content affects these rejuvenators as the higher water content creates more steam in the foaming process, leading to increased expansion. Due to the dosage being 1% to 3%, the total amount of water added for each of these shots was between 2 and 6 grams. As a result, there is no concern for liquid water interacting with the RA, as the steam escapes due to the thin film produced.

4.2 Foamed Rejuvenated Asphalt Binder Results

The FI results for the PG 67-22 binder dosed with RA1 can be seen in Figure 15. At the lowest temperature assessed, 130 °C, the results at the water contents considered did not vary and showed no overall trend. However, at the higher temperatures assessed, 140 °C and 150 °C, the

impact of water content on foam quality was very noticeable. At 140 °C, the FI peaked at 2% water content, with a significant drop in FI at 3% water content. For the 150 °C, the FI peaked at 2% water content. However, this temperature saw a lower decrease in the index at the 3% water content. The optimum foaming condition was selected at 150 °C with 2% water content, as it gave the overall highest FI, with an average ER_{max} of 11.1 and an average $t_{1/2}$ of 3.4 seconds.

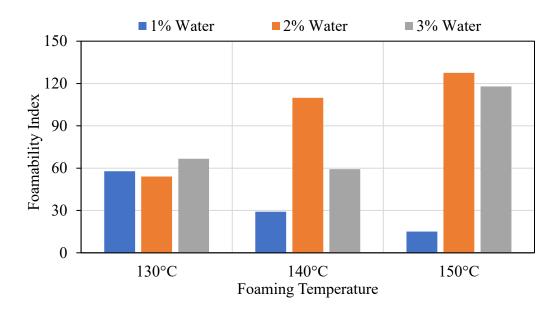


Figure 15. PG 67-22 Dosed with RA1 Foaming Results

The FI results of the PG 58S-28 binder dosed with RA2 across the tested conditions can be seen in Figure 16. Foam quality was consistent across all conditions, with temperature and water content not impacting FI significantly. The highest FI was seen at 150 $^{\circ}$ C and 2% water content, thus this condition was selected as the optimum. At this condition, an average ER_{max} of 15.3 and an average $t_{1/2}$ of 3.5 seconds were recorded.

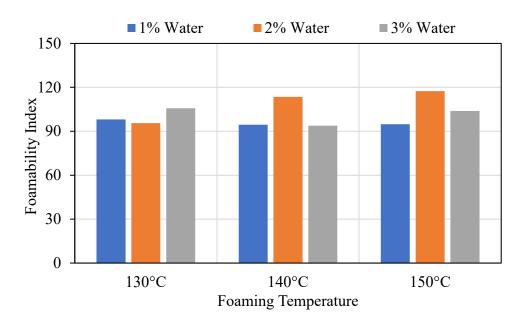


Figure 16. PG 58S-28 Dosed with RA2 Foaming Results

Comparing the two rejuvenated binders, the PG 58S-28 + RA2 had better and more consistent foaming characteristics at all temperatures and water contents. Both rejuvenated asphalt binders were optimized at the same condition, with comparable $t_{1/2}$, PG 58S-28 + RA2 saw better volume expansion.

4.3 Foaming Optimization Summary

The summarized foaming optimizations can be seen in Table 3. These results were utilized for RAP Pretreatment and Marination in Chapters 4 and 5, respectively, and for Mixture Performance testing in Chapter 6.

Table 3. Foaming Optimization Result Summary

OPTIMUM TEMPERATURE OPTIMUM WATER CONTENT

	(°C)	(%)
RA1	120	3
RA2	130	3
PG 67-22 + RA1	150	2
PG 58S-28 +RA2	150	2

Chapter 5: RAP Pretreatment Study

This chapter covers the results of the *RAP Pretreatment* phase of the experimental plan, as previously discussed in section 2.3. The two RAP sources evaluated were RAP A and RAP B, as described in section 2.1. These sources were treated with rejuvenators RA1 and RA2, respectively. Three methods of rejuvenator application were assessed: spray-on, emulsified product, and foamed product. RAP quality evaluation utilized the DWT, Cantabro testing, and grayscale analysis for physical testing. These tests assess the workability and compactability of the RAP, the durability of the RAP, and the degree of dispersion of the rejuvenator application method.

5.1 DWT Results of Untreated and Pretreated RAP

Figure 17 shows the results of the DWT at 240 °F and 300 °F compaction temperatures for untreated and pretreated RAP A. The error bars in the figure represent one plus and minus standard deviation. The pretreatment methods all utilized a rejuvenator dosage 0.85% by RAP weight, as described in section 2.1. All the pretreatment methods yielded higher DWT values than the untreated RAP, indicating improved RAP workability from pretreatment. Additionally, increasing the compaction temperature increased the workability of the RAP across untreated and pretreated specimens. This is potentially due to a decrease in the viscosity of the RAP binder; thus, more RAP binder becomes activated and contributes to compaction. Among the pretreatment methods, the emulsified RA1 pretreatment gave the highest DWT values at both temperatures, followed by the spray-on pretreatment and the foamed RA1 pretreatment.

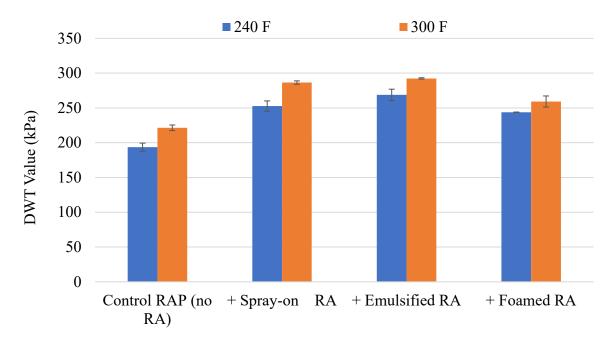


Figure 17. RAP Source A DWT Results

Figure 18 shows the RAP B DWT results for the control specimens (no RA2) and the pretreated specimens compacted at 240 °F and 300 °F. Again, the error bars represent one plus and minus standard deviation of the replicate measurement. At the lower compaction temperature (240 °F), all pretreatment methods displayed slightly increased workability from the control specimens. Of the pretreatment methods at this temperature, foamed RA2 pretreatment showed the best workability, followed by the spray-on pretreatment then the emulsion pretreatment. At the higher temperature (300 °F), this overall trend differed, with the spray-on and emulsified RA2 pretreatments performing comparably to the control specimens. Foamed RA2 pretreatment still displayed the highest DWT values, slightly greater than the control. This is potentially because more RAP binder is activated and contributing to compaction at the higher temperature. Additionally, the RA2 dosage used for all pretreatments was 0.34% by RAP weight. This dosage is below half the dosage used for RA1 with RAP A, confirming that a higher dosage produces a greater change in DWT value.

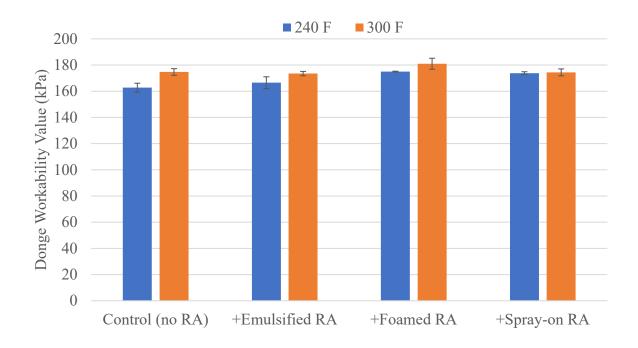


Figure 18. RAP Source B DWT Results

Comparing the two RAP sources, RAP A has higher DWT values in general and a larger impact on DWT values due to pretreatment. This is due to higher rejuvenator dosage for RAP A, at 0.85% by RAP weight versus 0.34% by RAP B weight for RA2. Overall, for RAP B, the pretreatment experimental methods did not significantly improve the DWT value from the control RAP at either temperature assessed.

5.2 Cantabro Mass Loss Results

Figure 19 displays the results of the Cantabro testing performed on the 240 °F compacted RAP A DWT samples. Error bars in the graph represent one plus and minus standard deviation of the replicate measurements. The control specimens broke down with 100% mass loss of the compacted samples. All the pretreatment methods produced improved mass loss percentages, with none of the application method averages making values greater than 20% loss. Of the pretreatment methods, the spray-on application performed the best, followed by foamed application, and

emulsified RA1 application, respectively. However, though these differences are not practically significant when considering the variability of the test. This indicates that pretreatment for RAP A at the dosage specified for RA1 greatly improved the durability of the RAP.

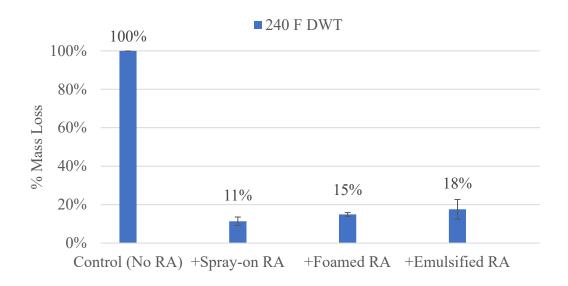


Figure 19. RAP Source A Cantabro Results

Figure 20 shows the results of Cantabro testing for the 240 °F compacted RAP B DWT samples. Again, error bars represent one plus and minus standard deviation of the replicate measurements. The control specimens saw almost complete breakdown from the test, with an average of 99% mass loss. All experimental pretreatment methods showed a reduced mass loss, though at a reduced degree, compared to the RA1 + RAP A pretreatments. Of the RAP B pretreatment methods, the spray-on and foamed RA2 applications showed the most significant reduction in mass loss, followed by the emulsified RA2 pretreatment, though again, the differences between rejuvenator applications are not practically significant due to test variability. For this RAP source and dosage, pretreatment improved RAP durability. However, the degree of durability improvement is not as large as RAP A pretreatments.

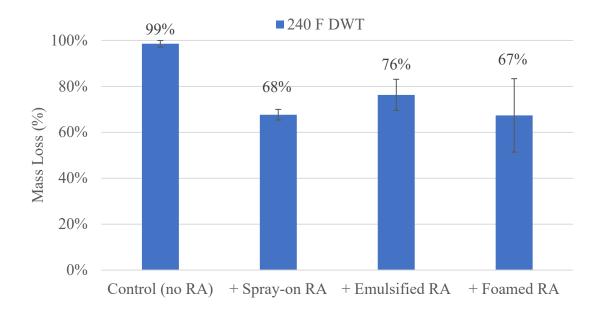


Figure 20. RAP Source B Cantabro Results

Comparing the two RAP source durability results, RAP A pretreatment had significantly better Cantabro results. This is due primarily to the 0.85% by RAP weight RA1 dosage, which is higher than the RA2 dosage of 0.34% by RAP B weight.

5.3 Image Analysis Results

Figure 21 displays the image analysis results of the untreated and pretreated RAP A samples. The curves represent the distribution of pixels with each grayscale value. All the pretreatment methods decreased the mean grayscale value and narrowed the distribution curve, indicating an overall darkening of the RAP material and a more consistent color. Overall, this can be interpreted as potentially increased RAP binder activation due to rejuvenator dispersion. Of the pretreatment methods, the emulsified RA1 and foamed RA1 performed the best with the lowest mean grayscale value, followed by the spray-on pretreatment. All three methods of pretreatment had similar widths of distribution curves, indicating similar RAP color consistency.

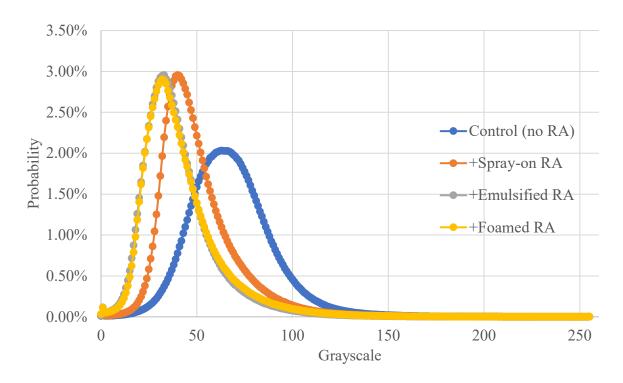


Figure 21. RAP Source A Grayscale Analysis Results

Figure 22 shows the grayscale distribution curves of untreated and pretreated RAP B samples. All the RA2 pretreatment methods decreased the mean grayscale value and narrowed the distribution curve, indicating a darker RAP material with a more consistent overall coloring. From this image analysis, the distribution curve shift indicates potentially increased RAP binder activation due to pretreatment. Among the pretreatment methods, all applications had similar mean grayscale values. Still, the the emulsified RA2 and foamed RA2 pretreatment methods produced a higher probability of the mean value and a narrower distribution than the spray-on pretreatment, indicating better consistency in coloring. This displays that using a dispersing agent, like water, with rejuvenators improves the application of the product and the activation of the RAP binder.

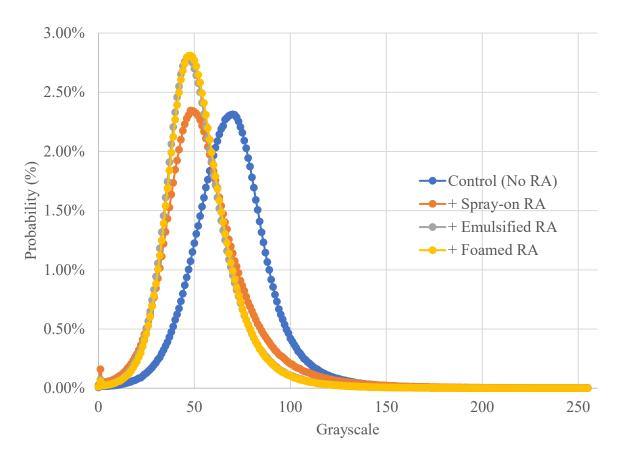


Figure 22. RAP Source B Grayscale Analysis Results

Comparing RAP A and RAP B image analysis results, RAP A saw a more dramatic shift in the distribution curves from the control to the pretreatment methods than RAP B. This is due to the dosage of RA1 for RAP A being much higher than that of RA2 for RAP B. Both sources saw the best grayscale analysis results using the emulsified and foamed rejuvenators, indicating that these methods provide the best degree of dispersion of the application methods assessed.

5.4 Moisture Content Results

Moisture content results after pretreatment of RA1 on RAP A can be seen in Figure 23. Error bars represent one plus and minus standard deviation of the replicate measurements. The spray-on pretreatment and the foamed RA1 pretreatment had moisture contents comparable to the control specimens. After mixing, the emulsified pretreatment had the highest moisture content, as

the emulsified RA1 contains 40% water by weight. The other experimental treatments had much lower water contents, as the spray-on treatment did not utilize water, and the foamed treatment used 3% water content by weight of the rejuvenator, which corresponds to 0.025% by weight of the RAP.

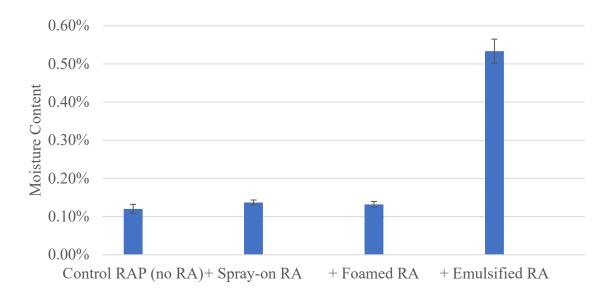


Figure 23. RAP Source A Moisture Content Results After Treatment

As a result of the high moisture content after treatment, the emulsified pretreatment was monitored for moisture content changes over multiple days of storage at ambient room temperature (~77 °F), as seen in Figure 24. Immediately after pretreatment, the emulsion pretreated RAP sample exhibited notably higher moisture content, which dropped to untreated RAP levels after two days of storage. Moisture content monitoring was necessary in the mixture performance phase for this pretreatment method to ensure proper RAP samples were measured without water weight impacting measurement.

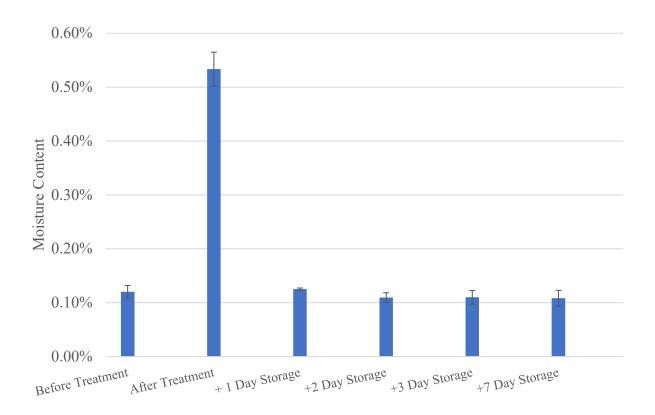


Figure 24. RAP Source A Emulsion Pretreatment Moisture Content Results

Figure 25 displays the moisture content results for the RA2 pretreatment of RAP B after pugmill mixing. Emulsified application showed the greatest increase in moisture content of the experimental pretreatments, followed by the foamed pretreatment method. The spray-on application was comparable to the control RAP sample, owing to the application method not utilizing water.

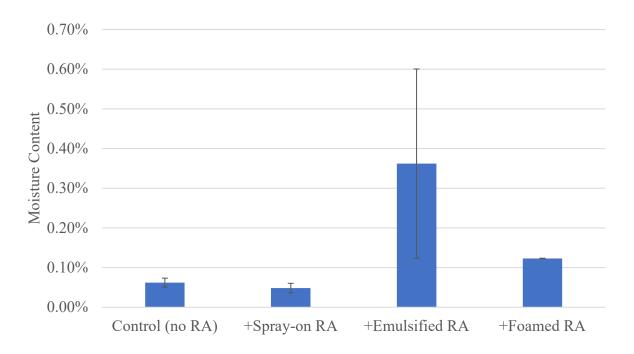


Figure 25. Moisture Content Results for RAP Source B Pretreatment

5.5 RAP Pretreatment Results Summary

For RAP A, the emulsified pretreatment gave the best DWT performance and grayscale distribution. The spray-on pretreatment gave the best Cantabro results, but emulsified RA1 application was the following best performing. From this, the best RA1 pretreatment method for RAP A is the emulsified application. This method will be utilized for the RAP marination pretreatment phase of the experimental plan.

For RAP B, the foamed RA2 pretreatment produced the best DWT and Cantabro performance, along with one of the best grayscale distributions. These results indicate that the foaming application of RA2 had the best degree of dispersion and the best "activation" of the RAP binder.

Overall, RAP pretreatment improved material workability and durability. Emulsion and foaming pretreatment produced better dispersion through grayscale image analysis than the spray-on pretreatment, indicating that a good pretreatment dispersion necessitates the use of an expansion

agent, such as water. These methods also increased water contents to levels well above the control RAP, depending on dosage, which became a factor in the mixing process. Additional water weight can alter mixture batching weights, so the pretreated RAP material must monitor the water content for application methods utilizing water to accurately weigh out RAP samples or be appropriately dried prior to batching.

Chapter 6: RAP Marination Study

This chapter covers the results of the *RAP Marination* phase of the experimental plan. The impact of marination was assessed using physical tests to evaluate workability, durability, and degree of dispersion. The RAP source used for this phase was RAP A and was dosed with 0.85% by weigh of RAP with emulsified RA1. Four total marination conditions were evaluated: two at ambient temperatures and two at high temperatures. At the ambient temperature (77 °F), a short-term, 3-day marination and a long-term, 7-day marination were observed. For the high temperature (275 °F) assessment, the short-term condition was 1.5 hours, and the long-term was 3 hours of marination. The physical tests utilized were the DWT, Cantabro, and grayscale image analysis.

6.1 DWT Marination Results

Figure 26 displays the results of the DWT testing at 240 °F and 300 °F for the unmarinated and the marinated emulsion pretreated RAP A. None of the experimental marination conditions increased the DWT value more significantly than the unmarinated samples at either temperature. The ambient temperature marination generated DWT values slightly more significant than the high temperature marination conditions of the experimental marination conditions.

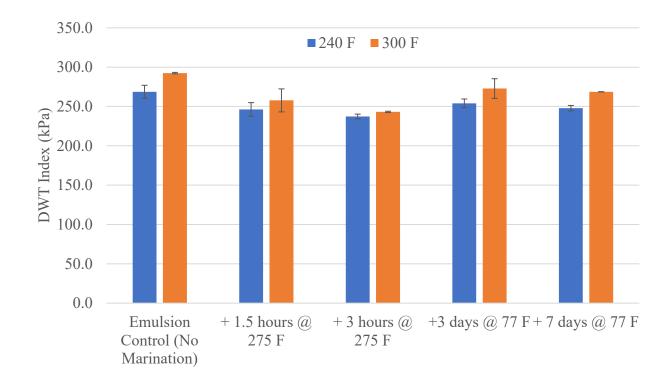


Figure 26. RAP Source A + Emulsified RA1 DWT Marination Results

These results indicate that marination does not significantly improve RAP workability and compatibility. At higher marination temperatures, the overall DWT value reduced the most, indicating that the high temperature further aged the RAP binder. Though the emulsified RA1 is expected to "activate" the RAP binder, the accelerated, high temperature marination may stiffen the RAP binder due to oxidation.

6.2 RAP Marination Cantabro Results

Figure 27 displays the results of the Cantabro testing performed on the 240 °F compacted DWT specimens. Assessing the average mass loss percentages of experimental marination methods, all marination methods produced reduced mass loss from the control emulsion samples, indicating increased durability due to marination. However, there was no practical difference between experimental marination methods and the control emulsion samples due to test variability.

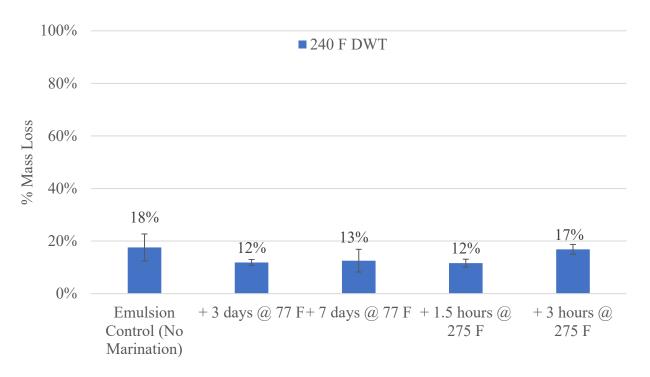


Figure 27. RAP Source A + Emulsified RA1 Cantabro Results

Overall, these results suggest that marination slightly improves RAP durability but not significantly.

6.3 RAP Marination Image Analysis Results

Figure 28 shows the grayscale image analysis distribution results of the emulsion pretreated RAP A samples at the unmarinated and various marination conditions. All the experimental methods produced very similar distributions to the unmarinated sample and each other, in terms of the width of the distribution and mean grayscale value. There is a slightly darker color, as seen through the mean value for the distribution, for the high temperature conditions. However, this indicates the potential oxidative aging of the RAP binder at 275 °F marination.

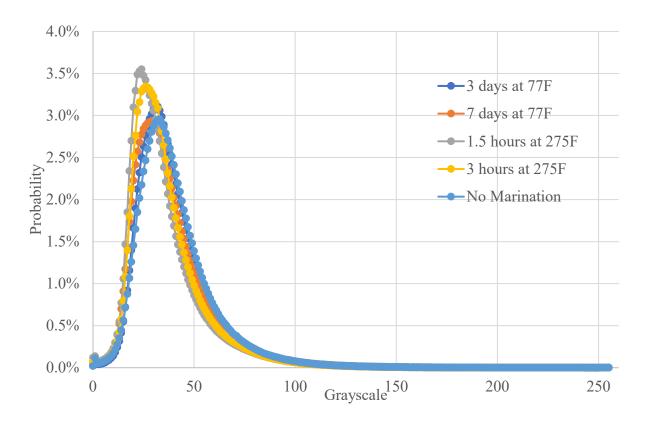


Figure 28. RAP Source A + Emulsified RA1 Marination Grayscale Analysis Results

From these results, marination has no impact on the degree of distribution of the rejuvenator or further "activation" of the RAP binder. Additionally, high temperature marination can shift the overall color of the RAP darker, but this can correlate to increased aging of the RAP binder rather than RAP binder "activation".

6.4 RAP Marination Summary

From the results of the RAP marination phase, it is seen that there is no improvement in RAP workability or rejuvenator degree of dispersion for RAP binder "activation". However, there is a slight improvement in RAP durability for low temperature marination and short, high-temperature marination conditions. Overall, for the marination conditions assessed, marination did not significantly improve the quality of RAP A with the selected rejuvenator dosage.

Chapter 7: Mixture Performance Results

This chapter covers the *Mixture Performance* phase of the experimental plan, focusing on mixture workability, durability, intermediate temperature cracking resistance, and low temperature cracking resistance evaluations. DWT and Cantabro samples were short-term aged for 2 hours at compaction temperature, per AASHTO R30, to evaluate mixture workability and durability. Cracking resistance samples (IDEAL-CT, I-FIT, and DCT) were evaluated using long-term aged samples, 4 hours at 275 °F, followed by critical aging for additional 6 hours at 275 °F. Mixture testing considered two high RAP mix designs with two bio-based rejuvenators and two virgin binders. As discussed in subchapter 2.1, mix design A was a 9.5mm NMAS Superpave mixture with 45% RAP utilizing a PG 67-22 binder and RA1, while mix design B was a 12.5mm NMAS Superpave mixture with 50% RAP utilizing a PG 58S-28 binder and RA2. For each mixture design, a control mixture without a rejuvenator and four experimental rejuvenated mixtures with the same effective dosage were prepared as seen in Table 4.

Table 4. Mixture ID Description

MIXTURE ID	DESIGNATION	RA INCORPORATION METHOD
MIX X-1	Control	No RA
MIX X-2	Experimental	Pre-blended into Virgin Binder
MIX X-3	Experimental	Emulsified Pretreatment
MIX X-4	Experimental	Foamed Pretreatment
MIX X-4	Experimental	Foamed Rejuvenated Binder

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

The results from the tests discussed were assessed using column charts, where the columns represent the average values, and the error bars are one plus and minus the standard deviation of the replicate measurements. For the IDEAL-CT, I-FIT, and DCT testing, both mean value analysis and Games-Howell post-hoc group analysis were performed at a significance level of 0.05. The capital letters above the columns represent this group analysis, where mixtures that share the same letter exhibit no significant statistical difference in test results.

7.1 DWT Results

Figure 29 and Figure 30 presents the DWT results of mix design A and B, respectively, at 240 °F and 300 °F, respectively. For mix design A, all four experimental rejuvenated mixtures showed higher average DWT values than the control mixtures at both evaluation temperatures, which indicated that the addition of the rejuvenator in general improved the mixture's workability. Among four experimental mixtures, Mix A-5, prepared with the foaming rejuvenated PG 67-22 binder, showed the highest average DWT value at 300 °F than other experimental mixtures.

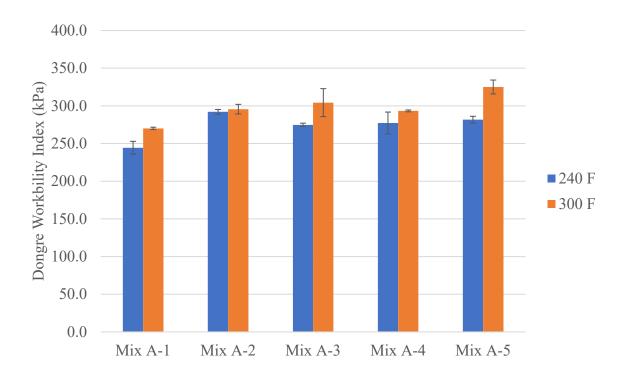


Figure 29. Mix Design A DWT Results

For the mix design B results in Figure 30, the average DWT values of all the experimental mixtures were higher than those of the control mixture at both test temperatures, which was consistent with mix design A. The differences in DWT values between the experimental and control mixtures were more pronounced at the higher test temperature. At 300 °F, the highest average DWT values were the experimental pretreatment mixtures (Mix B-3 and Mix B-4), indicating that the RAP pretreatment methods provide better workability at 300 °F than other rejuvenator incorporation methods for mix design B. However, at the lower test temperature (240 °F), all four experimental, rejuvenated mixtures performed similarly. Overall, the impact of the rejuvenator incorporation method on mixture workability was negligible for this mix design.

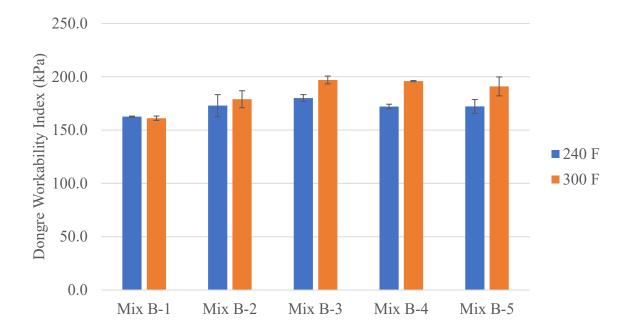


Figure 30. Mix Design B DWT Results

Comparing the DWT results of the two mix designs, mix design A consistently had higher values (250 – 300 kPa) than mix design B (150 – 200 kPa), indicating better workability. Mix design A had a finer gradation, a higher binder content, and a higher rejuvenator dosage than mix design B, leading to these higher DWT values. Additionally, these results depict the ability of the DWT test to differentiate mixtures with different workability.

7.2 Cantabro Mass Loss Results

Figure 31 presents the Cantabro testing results for mix design A, tested with the 240 °F compacted DWT samples. All rejuvenated mixtures produced average mass loss percentages lower than 10% and significantly lower than the control mixture. This indicates that the addition of a rejuvenator, regardless of the method used, improves the durability of this high RAP mix design. Among the rejuvenated mixtures, Mix A-5 resulted in the lowest mass loss, with Mix A-2 being a close second. The two pretreatment rejuvenated mixtures (Mix A-3 and Mix A-4) performed similarly, but not as well as the pre-blended rejuvenated mixtures. These results indicate that the

rejuvenator incorporation method does not significantly impact mixture durability for this high RAP mix design.

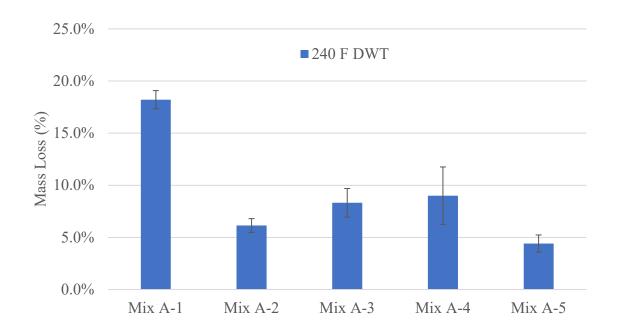


Figure 31. Mix Design A CML Results

Figure 32 displays the Cantabro testing results for mix design B, tested on the 240 °F compacted DWT samples. As seen in the results, all rejuvenated mixtures had lower mass loss percentages than the control mixture, indicating that rejuvenator addition improved mixture durability for this high RAP mix design. Among the rejuvenated combinations, all incorporation methods performed similarly, meaning that for this mix design incorporation method did not significantly impact mixture durability.

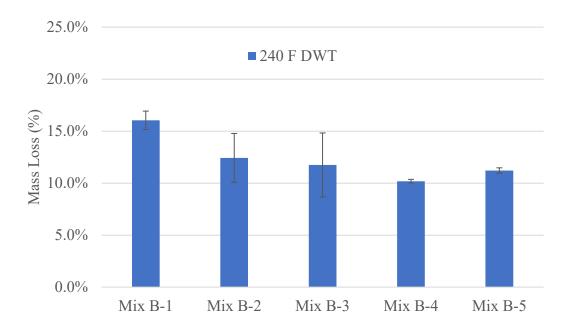


Figure 32. Mix Design B CML Results

Comparing the two mix designs, the control mixtures for both performed similarly. Still, the rejuvenated mixtures with mix design A had better improvement in durability than those of mix design B. This is due to a lower RAP content (45% versus 50%) in mix design A, a higher binder content and rejuvenator dosage, and a finer gradation. Rejuvenator addition improves mixture durability for both high RAP mix designs, but the incorporation method showed no significant impact.

7.3 IDEAL-CT Results

Figure 33 displays the results of the IDEAL-CT testing for mix design A, along with the group analysis results. From these results, all four experimental mixtures had higher average CT_{Index} values than the control mixture, implying increased resistance to intermediate temperature cracking due to the addition of the rejuvenator. Within the rejuvenated mixtures, Mix A-2 and Mix A-5 had higher mean 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 pretreating the

RAP with the rejuvenator. The statistical group analysis results confirmed that all four rejuvenated mixtures showed significantly higher CT_{Index} values than the control mixture. However, there was no statistical difference among the four experimental rejuvenated mixtures except that Mix A-5 yielded significantly higher CT_{Index} than Mix A-4. Overall, the CT_{Index} results for mix design A indicate that the addition method of rejuvenator did not significantly impact the intermediate-temperature cracking resistance of this high RAP mix design at a long-term aging condition.

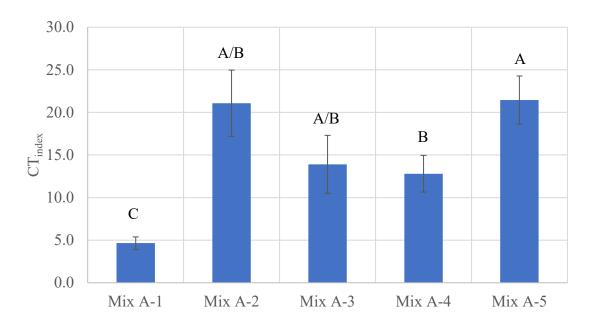


Figure 33. Mix Design A IDEAL-CT Results

In addition to the CT_{Index} test parameter, two other interim parameters from the IDEAL-CT load-displacement curve were assessed, G_f and $|m_{75}|/I_{75}$, wherein G_f describes mixture toughness and $|m_{75}|/I_{75}$ describes a mixture's relative ductile-brittle behavior. The G_f and $|m_{75}|/I_{75}$ results for mix design A can be seen in Figure 34 and Figure 35, respectively. All the experimental rejuvenated mixes had slightly higher average G_f than the control, yet statistical group analysis found no significant difference between the control and the rejuvenated mixtures. The $|m_{75}|/I_{75}$ results in Figure 35 showed that the pre-blending incorporation methods (Mix A-2 and Mix A-5) had lower $|m_{75}|/I_{75}$ than the other mixtures, implying less brittle behavior than the control and

pretreating incorporation method. Additionally, all rejuvenated mixtures were statistically different from the control, which had the highest $|m_{75}|/I_{75}$ result, indicating the most brittle behavior. From these results, the $|m_{75}|/I_{75}$ parameter could better discern the differences in the rejuvenator incorporation method for this high RAP mix design than the G_f parameter.

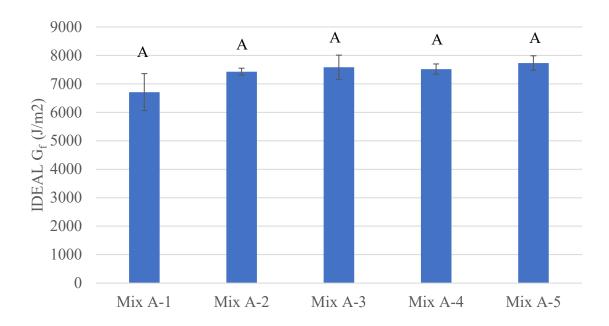


Figure 34. Mix Design A IDEAL-CT Fracture Energy Results

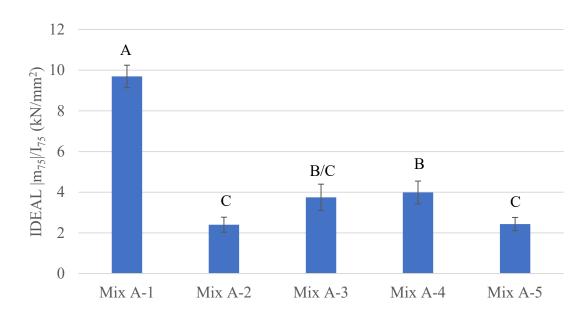


Figure 35. Mix Design A IDEAL-CT | m₇₅|/I₇₅ Results

Figure 36 presents the average CT_{Index} results of mix design B with the corresponding statistical group analysis results. All four experimental rejuvenated mixtures had higher average CT_{Index} values than the control mixture, indicating improved intermediate-temperature cracking resistance with the addition of a rejuvenator. The mixtures prepared with pre-blending rejuvenator showed slightly higher average CT_{Index} results than those prepared with pretreated RAP, indicating that pre-blending the rejuvenator into virgin binder provided better rejuvenating effectiveness than incorporating the rejuvenator through RAP pretreatment. However, the group analysis results indicated that none of the mixtures were statistically different from each other due to variability in test results.

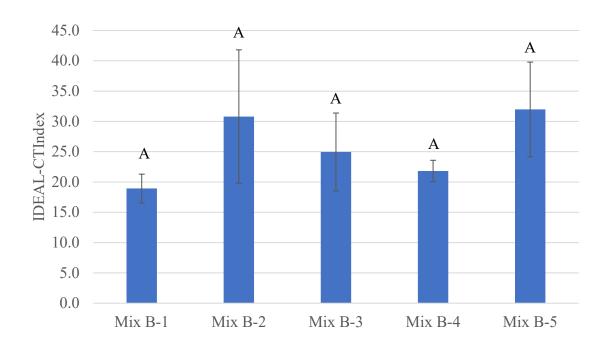


Figure 36. Mix Design B IDEAL-CT Results

The interim parameters G_f and $|m_{75}|/I_{75}$ from the IDEAL-CT load-displacement curves for mix design B were assessed, as seen in Figure 37 and Figure 38, respectively. As seen in Figure 37, all experimental rejuvenated mixtures performed similarly, with average G_f lower than the

control mixture. However, statistical grouping showed that G_f did not differ between the control and experimental mixtures. Comparatively, the $|m_{75}|/I_{75}$ parameter displayed better discrimination between the control and rejuvenated mixtures, as seen in Figure 38. The control mixture exhibited the highest $|m_{75}|/I_{75}$ value, indicating more brittle behavior than the rejuvenated mixtures. Among the experimental rejuvenated mixtures, Mix B-2 and Mix B-5 had lower ratios than the two rejuvenated mixtures prepared with the RAP pretreatment methods, indicating the pre-blending incorporation method causes more ductile behavior. Statistical grouping results showed that only Mix B-1 or Mix B-4 and Mix B-5 were significantly different, while the other mixture differences were not.

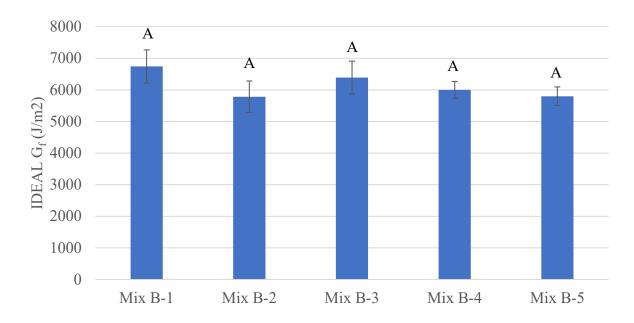


Figure 37. Mix Design B IDEAL-CT G_f Results

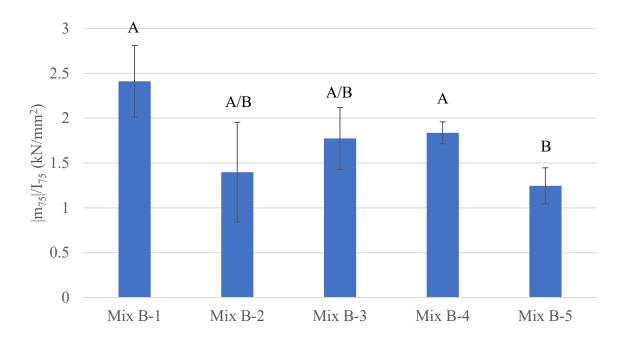


Figure 38. Mix Design B IDEAL-CT | m₇₅|/I₇₅ Results

Comparing both mixture's IDEAL-CT results showed that pre-blending rejuvenator with virgin binder produced slightly better rejuvenating effectiveness than the two RAP pretreatment methods assessed; however, statistical grouping showed that the differences were not significant in most cases. Furthermore, the differences in rejuvenator incorporation methods were visible when assessing the ductile-brittle behavior ($|m_{75}|/I_{75}$) of the mixture. However, they did not impact the mixture toughness as indicated by the G_f .

7.4 I-FIT Results

Figure 39 displays the results of the I-FIT testing for mix design A, with the corresponding statistical grouping analysis. All the experimental rejuvenated mixtures produced higher average flexibility indices than the control. Among the rejuvenated mixtures, the pre-blending methods gave the highest average flexibility index; however, due to the testing variability, this was not statistically significant from the other rejuvenated mixtures. Only Mix A-5 and Mix A-1 had statistically different flexibility index results.

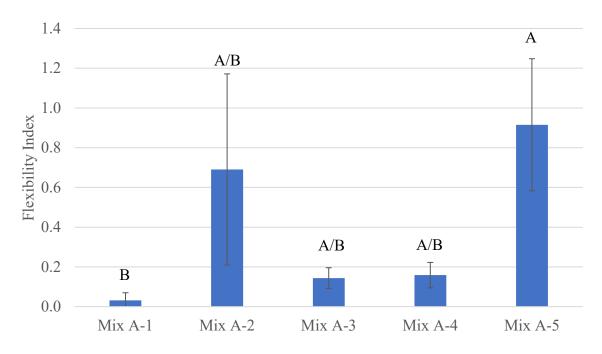


Figure 39. Mix Design A I-FIT Results

Figure 40 displays the results of the I-FIT testing for mix design B with the corresponding statistical grouping analysis. All the experimental rejuvenated mixtures produced higher average flexibility index values than the control mixture, with Mix B-2, Mix B-3, and Mix B-5 being statistically different from the control. Mix B-2 produced the highest average flexibility index among the rejuvenated mixtures, while the other three experimental rejuvenated mixtures producing comparable results. Due to test variability, Mix B-4 is not statistically different from the control, though the average index is comparable to the other experimental mixes.

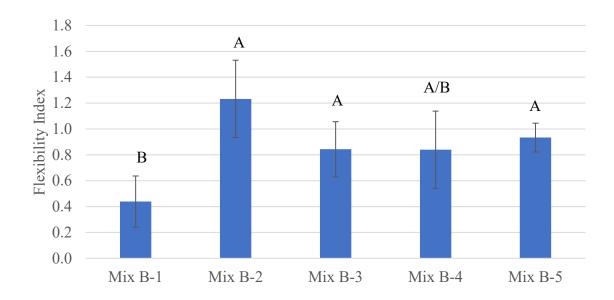


Figure 40. Mix Design B I-FIT Results

Comparing mix design results, rejuvenator incorporation improves the I-FIT flexibility index, indicating better intermediate temperature cracking resistance. The incorporation method is statistically significant in most cases, especially in mix design B. Overall, the pre-blending methods produced the highest flexibility index values, indicating that this incorporation method provides the best rejuvenating effect.

7.5 DCT Results

The DCT G_f results of mix design A are presented in Figure 41, with corresponding statistical grouping results. All experimental rejuvenated mixtures had slightly higher average G_f than the control mixture, which indicated that rejuvenator addition improved the thermal cracking resistance of the high RAP mixture. Among the four experimental rejuvenated mixtures, the average G_f results appeared equivalent, which was confirmed by the group analysis results, which indicated no significant difference between the rejuvenated mixtures. Additionally, group analysis indicated no statistical difference between the control and rejuvenated mixtures. Overall, the

thermal cracking resistance of this mix design was not affected by the different rejuvenator incorporation methods.

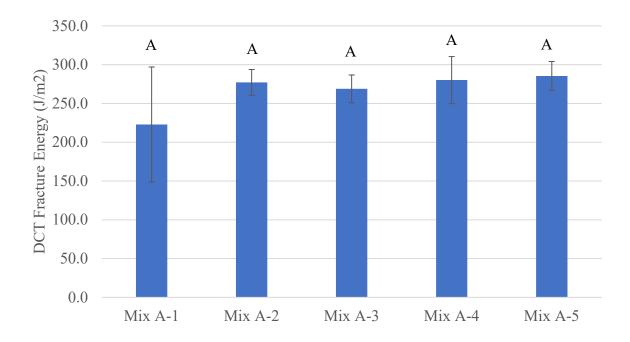


Figure 41. Mix Design A DCT Results

Figure 42 displays the DCT G_f results of mix design B with the corresponding statistical grouping analysis results. All rejuvenated mixtures, except Mix B-4, had G_f values higher than the control; however, only Mix B-2 was statistically different from the control. The other pre-blended mixture was not significantly different from the control due to test variability. These results indicate that rejuvenator incorporation is significant for the thermal cracking resistance of this high RAP mix design. The pre-blending incorporation method provided statistically significant improvement in thermal cracking resistance, as indicated by DCT G_f, while the foaming pretreatment method gave similar results to the mixture without a rejuvenator. This indicates that for this mix design, the traditional pre-blending incorporation method provides the best rejuvenating effectiveness among the methods studied.

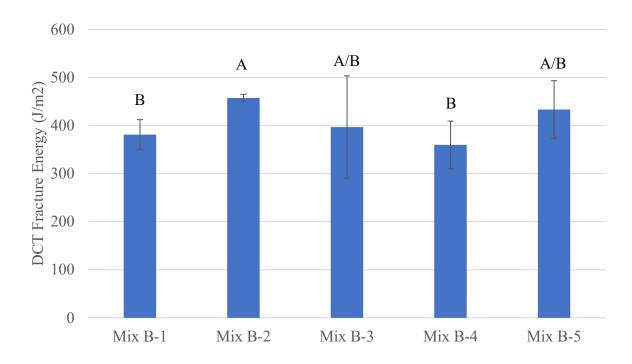


Figure 42. Mix Design B DCT Results

Comparing the results of the two high RAP mix designs, mix design B statistical groups showed more sensitivity to the rejuvenator incorporation method.

7.6 Mixture Testing Summary

From the myriad of tests assessed in the chapter, the addition of rejuvenators in general improves the workability, durability, intermediate-temperature cracking resistance, and thermal cracking resistance of high RAP mixtures. In in some cases, that improvement was not statistically significant. Overall, the pre-blending method (either the traditional method or the foaming-enhanced method) produced better rejuvenating effectiveness and therefore better resultant mixture performance properties than the two rejuvenator incorporation methods through RAP pretreatment.

Chapter 8: Conclusions

This study sought to explore alternative rejuvenator incorporation methods and determine their potential to improve the workability, durability, and cracking resistance of high RAP asphalt mixtures. The experimental plan to assess the impacts was divided into four phases, focusing on foaming optimization of rejuvenators and rejuvenated binders, RAP pretreatment, RAP marination, and mixture performance testing, respectively. The major findings from this study are summarized as follows:

- Both the rejuvenators and the rejuvenated asphalt binders displayed good foaming characteristics at most foaming conditions assessed in the study. RA1 had an optimum condition at 120 °C and 3% water content, while RA2 optimized at 130 °C and 3% water content. Both rejuvenated asphalt binders optimized at the same foaming condition: 150 °C and 2% water content.
- The addition of RA1 to RAP A significantly improved the quality characteristics with the emulsified RA1 being the most effective application method. This was seen through higher DWT values, and a darker and more consistent color appearance from grayscale-based image analysis. The Cantabro mass loss was comparable among the different rejuvenator application methods for RAP A and RA1. Due to the high dosage of RA1 for RAP A, moisture control was of concern for the emulsion pretreatment; however, the moisture content of the emulsion pretreated RAP A dropped to the untreated level after two days of ambient temperature storage. The RA2 application did not significantly improve the RAP B quality as compared to RA1 + RAP A. The RAP B DWT results did not improve significantly, confirmed by grayscale-based image analysis showing only a slightly darker appearance and color consistency. This slight quality improvement was confirmed with Cantabro mass loss results that were only 30% lower than the untreated RAP B. The most

effective application method for RAP B + RA2 appeared to be the foaming method. Moisture content was also assessed for RAP B + RA2 pretreatment methods, with the moisture content level increasing the most after emulsion pretreatment.

- The marination impact on RAP quality was evaluated with RAP A pretreated with emulsified RA1. DWT values indicated no improvement in RAP quality at any marination conditions, as confirmed by grayscale-based image analysis showing no difference in mean pixel value or pixel distribution. Furthermore, Cantabro mass loss results displayed no difference between the marinated and non-marinated RAP samples in terms of durability.
- For both high RAP mix designs, the addition of rejuvenator, in general, improved mixture workability, durability, intermediate-temperature cracking resistance, and thermal cracking resistance, though not statistically significant in some cases. From the results of the multiple performance tests, the pre-blending method (either the traditional method or foaming-enhanced method) produced better rejuvenating effectiveness and therefore, better resultant mixture performance properties than the two rejuvenator incorporation methods through RAP pretreatment.

The findings of this study suggest that asphalt contractors should continue to use the preblending method for rejuvenator incorporation for the design and production of high RAP mixtures due to mixture performance and ease of plant operation considerations. From the results of subchapter 4.1, further studies are recommended to evaluate the efficacy of the DWT test to potentially evaluate the overall quality and consistency of RAP stockpiles as a quick yet robust tool for asphalt contractors and state highway agencies.

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Appendices

Appendix A – Mix Design A: IDEAL-CT Statistical Analysis

CT_{Index} Vs. Mixture ID

Method

Null hypothesis All means are equal
Alternative hypothesis Not all means are equal

Significance level $\alpha = 0.05$

Equal variances were not assumed for the analysis.

Factor Information

Factor	Levels Values
ID	5 Mix A-1, Mix A-2, Mix A-3, Mix A-4, Mix A-5

Welch's Test

Source	DF Num	DF Den	F-Value	P-Value
ID	4	6.59587	46.73	0.000

Model Summary

R-sq	R-sq(adj)	R-sq(pred)
86.49%	82.89%	75.98%

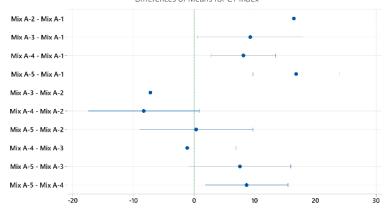
Means

ID	N	Mean	StDev	95% CI
Mix A-1	4	4.650	0.733 (3.484, 5.816)
Mix A-2	4	21.07	3.89 (14.89, 27.26)
Mix A-3	4	13.90	3.40	(8.48, 19.32)
Mix A-4	4	12.80	2.16	(9.37, 16.23)
Mix A-5	4	21.45	2.82 (16.96, 25.94)

Grouping Information Using the Games-Howell Method and 95% Confidence

ID	N	Mean	Grouping	
Mix A-5	4	21.45 A		
Mix A-2	4	21.07 A	В	
Mix A-3	4	13.90 A	В	
Mix A-4	4	12.80	В	
Mix A-1	4	4.650	C	

Games-Howell Simultaneous 95% Cls Differences of Means for CT Index



If an interval does not contain zero, the corresponding means are significantly different.

Interval Plot of CT Index vs ID 95% CI for the Mean 25 20 20 Mix A-1 Mix A-2 Mix A-3 Mix A-4 Mix A-5

Individual standard deviations are used to calculate the intervals.

<u>IDEAL-CT Fracture Energy Vs. Mixture ID</u> Method

Null hypothesis All means are equal
Alternative hypothesis Not all means are equal

Significance level $\alpha = 0.05$

Equal variances were not assumed for the analysis.

Factor Information

Factor	Levels Values
ID	5 Mix A-1, Mix A-2, Mix A-3, Mix A-4, Mix A-5

Welch's Test

Source	DF Num	DF Den	F-Value	P-Value
ID	4	7.07101	2.08	0.187

Model Summary

R-sq	R-sq(adj)	R-sq(pred)
54.16%	41.93%	18.50%

Means

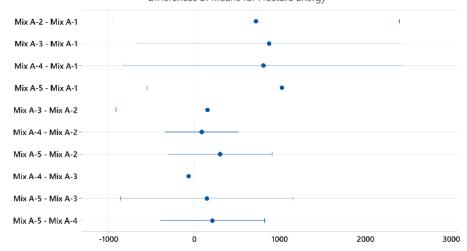
ID	N	Mean	StDev	95% CI
Mix A-1	4	6710	650	(5676, 7744)
Mix A-2	4	7429.4	121.0	(7237.0, 7621.9)
Mix A-3	4	7584	427	(6905, 8263)
Mix A-4	4	7518.5	181.6	(7229.6, 7807.4)
Mix A-5	4	7732	254	(7328, 8137)

Grouping Information Using the Games-Howell Method and 95% Confidence

ID	N	Mean Grouping	
Mix A-5	4	7732 A	
Mix A-3	4	7584 A	
Mix A-4	4	7518.5 A	
Mix A-2	4	7429.4 A	
Mix A-1	4	6710 A	

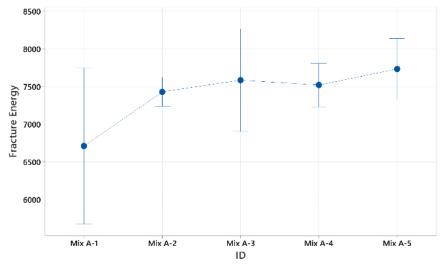
Means that do not share a letter are significantly different.





If an interval does not contain zero, the corresponding means are significantly different.

Interval Plot of Fracture Energy vs ID 95% CI for the Mean



Individual standard deviations are used to calculate the intervals.

IDEAL-CT | m₇₅|/I₇₅ Vs. Mixture ID

Method

Null hypothesis All means are equal Alternative hypothesis Not all means are equal

Significance level $\alpha = 0.05$

Equal variances were not assumed for the analysis.

Factor Information

Factor	Levels Values
ID	5 Mix A-1, Mix A-2, Mix A-3, Mix A-4, Mix A-5

Welch's Test

Source	DF Num	DF Den	F-Value	P-Value
ID	4	7.36643	114.63	0.000

Model Summary

 R-sq	R-sq(adj)	R-sq(pred)
97.44%	96.76%	95.45%

Means

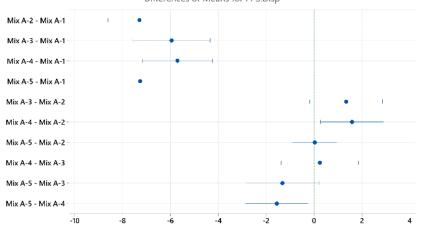
ID	N	Mean	StDev	95% CI
Mix A-1	4	9.693	0.552	(8.815, 10.571)
Mix A-2	4	2.401	0.370	(1.812, 2.989)
Mix A-3	4	3.748	0.647	(2.719, 4.777)
Mix A-4	4	3.987	0.559	(3.098, 4.876)
Mix A-5	4	2.433	0.325	(1.916, 2.951)

Grouping Information Using the Games-Howell Method and 95% Confidence

ID	N	Mean	Group	ping	
Mix A-1	4	9.693 A			
Mix A-4	4	3.987	В		
Mix A-3	4	3.748	В	\mathbf{C}	
Mix A-5	4	2.433		\mathbf{C}	
Mix A-2	4	2.401		\mathbf{C}	

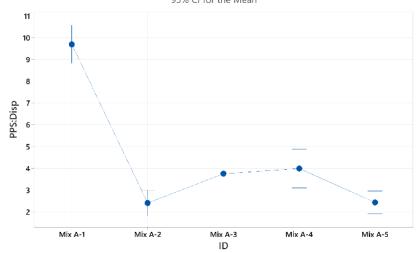
Means that do not share a letter are significantly different.

Games-Howell Simultaneous 95% Cls Differences of Means for PPS:Disp



If an interval does not contain zero, the corresponding means are significantly different.

Interval Plot of PPS:Disp vs ID 95% CI for the Mean



Appendix B – Mix Design A: I-FIT Statistical Analysis Flexibility Index Vs. Mixture ID

Method

Null hypothesis All means are equal
Alternative hypothesis Not all means are equal

Significance level $\alpha = 0.05$

Equal variances were not assumed for the analysis.

Factor Information

Factor	Levels Values
Mix ID	5 A-1, A-2, A-3, A-4, A-5

Welch's Test

Source	DF Num	DF Den	F-Value	P-Value
Mix ID	4	5.99830	8.29	0.013

Model Summary

R-sq	R-sq(adj)	R-sq(pred)
73.06%	64.77%	44.86%

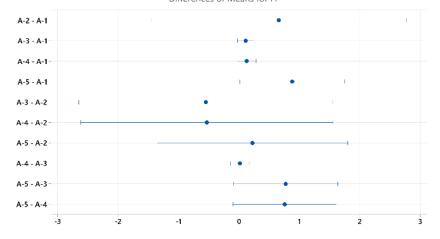
Means

Mix ID	N	Mean	StDev	95% CI
A-1	3	0.0317	0.0382	(-0.0632, 0.1266)
A-2	3	0.691	0.481	(-0.504, 1.885)
A-3	4	0.1434	0.0525	(0.0598, 0.2269)
A-4	4	0.1586	0.0639	(0.0569, 0.2603)
A-5	4	0.915	0.333	(0.386, 1.445)

Grouping Information Using the Games-Howell Method and 95% Confidence

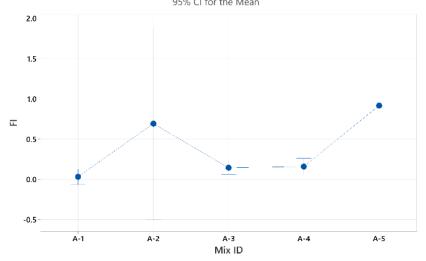
Mix ID	\mathbf{N}	Mean	Grouping
A-5	4	0.915 A	
A-2	3	0.691 A	В
A-4	4	$0.1586\mathrm{A}$	В
A-3	4	$0.1434\mathrm{A}$	В
A-1	3	0.0317	В

Games-Howell Simultaneous 95% Cls Differences of Means for FI



If an interval does not contain zero, the corresponding means are significantly different.

Interval Plot of FI vs Mix ID 95% CI for the Mean



Appendix C – Mix Design A: DCT Statistical Analysis DCT Fracture Energy Vs. Mixture ID

Method

Null hypothesis All means are equal Alternative hypothesis Not all means are equal

Significance level $\alpha = 0.05$

Equal variances were not assumed for the analysis.

Factor Information

Factor	Levels Values
ID	5 Mix A-1, Mix A-2, Mix A-3, Mix A-4, Mix A-5

Welch's Test

Source	DF Num	DF Den	F-Value	P-Value
ID	4	12.2225	1.23	0.348

Model Summary

 R-sq	R-sq(adj)	R-sq(pred)
29.51%	18.24%	0.00%

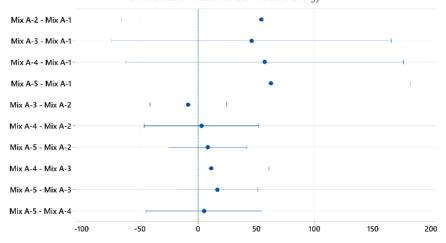
Means

ID	\mathbf{N}	Mean	StDev	95% CI
Mix A-1	6	222.8	74.2	(144.9, 300.7)
Mix A-2	6	277.17	16.64	(259.70, 294.63)
Mix A-3	6	268.83	17.93	(250.02, 287.65)
Mix A-4	6	280.2	30.3	(248.4, 312.0)
Mix A-5	6	285.50	18.62	(265.96, 305.04)

Grouping Information Using the Games-Howell Method and 95% Confidence

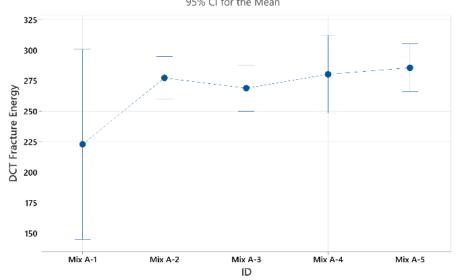
ID	\mathbf{N}	Mean Grouping	
Mix A-5	6	285.50 A	
Mix A-4	6	280.2 A	
Mix A-2	6	277.17 A	
Mix A-3	6	268.83 A	
Mix A-1	6	222.8 A	

Games-Howell Simultaneous 95% Cls Differences of Means for DCT Fracture Energy



If an interval does not contain zero, the corresponding means are significantly different.

Interval Plot of DCT Fracture Energy vs ID 95% CI for the Mean



Appendix D – Mix Design B: IDEAL-CT Statistical Analysis $\underline{\text{CT}_{Index}}$ Vs. Mixture ID

Method

Null hypothesis All means are equal
Alternative hypothesis Not all means are equal

Significance level $\alpha = 0.05$

Equal variances were not assumed for the analysis.

Factor Information

Factor	Levels Values
ID	5 Mix B-1, Mix B-2, Mix B-3, Mix B-4, Mix B-5

Welch's Test

Source	DF Num	DF Den	F-Value	P-Value
ID	4	6.94953	3.12	0.091

Model Summary

 R-sq	R-sq(adj)	R-sq(pred)
42.16%	26.74%	0.00%

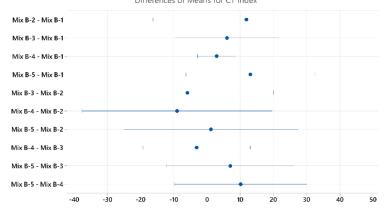
Means

ID	N	Mean	StDev	95% CI
Mix B-1	4	18.93	2.37	(15.16, 22.69)
Mix B-2	4	30.80	11.00	(13.29, 48.31)
Mix B-3	4	24.95	6.43	(14.72, 35.18)
Mix B-4	4	21.825	1.756	(19.031, 24.619)
Mix B-5	4	31.97	7.81	(19.55, 44.40)

Grouping Information Using the Games-Howell Method and 95% Confidence

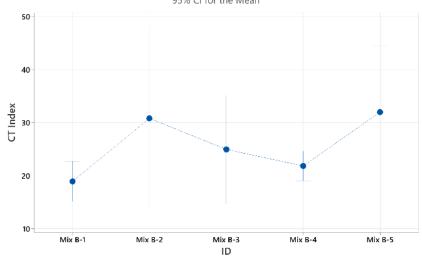
ID	N	Mean Grouping	
Mix B-5	4	31.97 A	
Mix B-2	4	30.80 A	
Mix B-3	4	24.95 A	
Mix B-4	4	21.825 A	
Mix B-1	4	18.93 A	

Games-Howell Simultaneous 95% Cls Differences of Means for CT Index



If an interval does not contain zero, the corresponding means are significantly different.

Interval Plot of CT Index vs ID 95% CI for the Mean



Individual standard deviations are used to calculate the intervals.

IDEAL-CT Fracture Energy Vs. Mixture ID

Method

Null hypothesis All means are equal
Alternative hypothesis Not all means are equal

Significance level $\alpha = 0.05$

Equal variances were not assumed for the analysis.

Factor Information

Factor	Levels Values
ID	5 Mix B-1, Mix B-2, Mix B-3, Mix B-4, Mix B-5

Welch's Test

Source	DF Num	DF Den	F-Value	P-Value
ID	4	7.33463	2.50	0.132

Model Summary

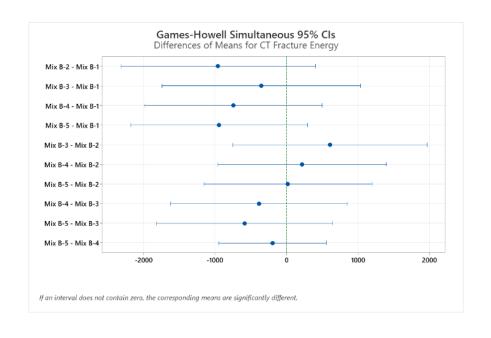
R-sq	R-sq(adj)	R-sq(pred)
49.00%	35.40%	9.33%

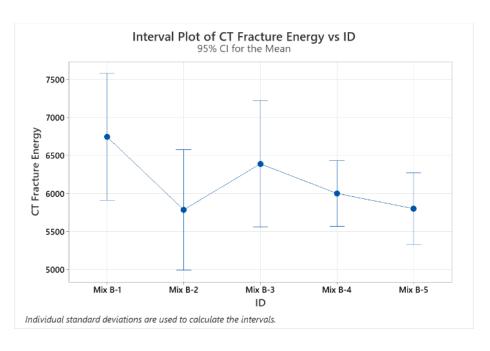
Means

ID	N	Mean	StDev	95% CI
Mix B-1	4	6742	524	(5909, 7576)
Mix B-2	4	5783	498	(4991, 6575)
Mix B-3	4	6388	522	(5557, 7218)
Mix B-4	4	5999	269	(5570, 6427)
Mix B-5	4	5799	296	(5328, 6269)

Grouping Information Using the Games-Howell Method and 95% Confidence

ID	N	Mean Grouping	
Mix B-1	4	6742 A	
Mix B-3	4	6388 A	
Mix B-4	4	5999 A	
Mix B-5	4	5799 A	
Mix B-2	4	5783 A	





IDEAL-CT |m₇₅|/I₇₅ Vs. Mixture ID

Method

Null hypothesis All means are equal
Alternative hypothesis Not all means are equal

Significance level $\alpha = 0.05$

Equal variances were not assumed for the analysis.

Factor Information

Factor	Levels Values
ID	5 Mix B-1, Mix B-2, Mix B-3, Mix B-4, Mix B-5

Welch's Test

Source	DF Num	DF Den	F-Value	P-Value
ID	4	6.98965	7.64	0.011

Model Summary

R-sq	R-sq(adj)	R-sq(pred)
62.98%	53.11%	34.20%

Means

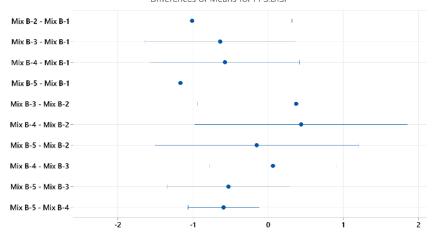
ID	N	Mean	StDev	95% CI
Mix B-1	4	2.411	0.400	(1.774, 3.047)
Mix B-2	4	1.398	0.555	(0.514, 2.282)
Mix B-3	4	1.773	0.345	(1.224, 2.323)
Mix B-4	4	1.8374	0.1224	(1.6426, 2.0321)
Mix B-5	4	1.246	0.201	(0.926, 1.566)

Grouping Information Using the Games-Howell Method and 95% Confidence

ID	N	Mean	Grouping	
Mix B-1	4	2.411 A		
Mix B-4	4	1.8374 A		
Mix B-3	4	1.773 A	В	
Mix B-2	4	1.398 A	В	
Mix B-5	4	1.246	В	

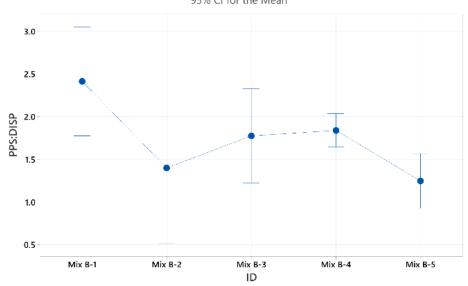
Means that do not share a letter are significantly different.

Games-Howell Simultaneous 95% Cls
Differences of Means for PPS:DISP



If an interval does not contain zero, the corresponding means are significantly different.

Interval Plot of PPS:DISP vs ID 95% CI for the Mean



Appendix E – Mix Design B: I-FIT Statistical Analysis Flexibility Index Vs. Mixture ID

Method

Null hypothesis All means are equal
Alternative hypothesis Not all means are equal

Significance level $\alpha = 0.05$

Rows unused 1

Equal variances were not assumed for the analysis.

Factor Information

Factor	Levels Values
Mix ID	5 B-1, B-2, B-3, B-4, B-5

Welch's Test

Source	DF Num	DF Den	F-Value	P-Value
Mix ID	4	14.2919	10.81	0.000

Model Summary

R-sq	R-sq(adj)	R-sq(pred)
58.81%	53.32%	45.12%

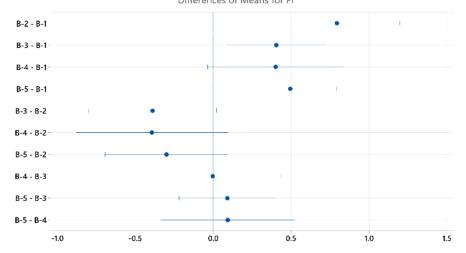
Means

Mix ID	N	Mean	StDev	95% CI
B-1	8	0.4389	0.1977	(0.2736, 0.6041)
B-2	8	1.233	0.299	(0.983, 1.483)
B-3	8	0.8432	0.2133	(0.6649, 1.0215)
B-4	7	0.840	0.298	(0.564, 1.116)
B-5	4	0.9338	0.1113	(0.7567, 1.1109)

Grouping Information Using the Games-Howell Method and 95% Confidence

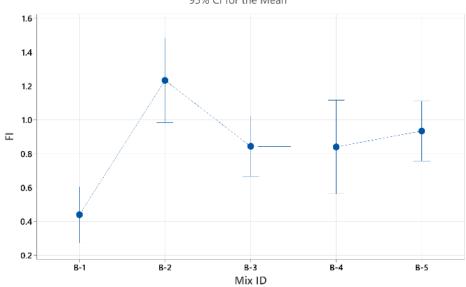
Mix ID	${f N}$	Mean	Grouping
B-2	8	1.233 A	
B-5	4	0.9338 A	
B-3	8	$0.8432\mathrm{A}$	
B-4	7	$0.840\mathrm{A}$	В
B-1	8	0.4389	В

Games-Howell Simultaneous 95% Cls Differences of Means for Fl



If an interval does not contain zero, the corresponding means are significantly different.

Interval Plot of FI vs Mix ID 95% CI for the Mean



Appendix F – Mix Design B: DCT Statistical Analysis DCT Fracture Energy Vs. Mixture ID

Method

Null hypothesis All means are equal
Alternative hypothesis Not all means are equal

Significance level $\alpha = 0.05$

Equal variances were not assumed for the analysis.

Factor Information

Factor	Levels Values
ID	5 Mix B-1, Mix B-2, Mix B-3, Mix B-4, Mix B-5

Welch's Test

Source	DF Num	DF Den	F-Value	P-Value
ID	4	9.95153	9.74	0.002

Model Summary

R-sq	R-sq(adj)	R-sq(pred)
31.46%	19.00%	0.67%

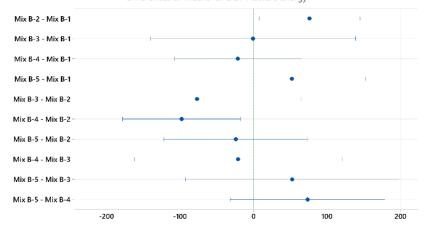
Means

ID	\mathbf{N}	Mean	StDev	95% CI
Mix B-1	5	381.2	35.4	(337.2, 425.2)
Mix B-2	4	457.50	7.68	(445.28, 469.72)
Mix B-3	6	380.7	87.0	(289.4, 471.9)
Mix B-4	6	359.8	49.5	(307.9, 411.8)
Mix B-5	6	433.5	60.0	(370.5, 496.5)

Grouping Information Using the Games-Howell Method and 95% Confidence

ID	N	Mean	Grouping	
Mix B-2	4	457.50 A		
Mix B-5	6	433.5 A	В	
Mix B-1	5	381.2	В	
Mix B-3	6	380.7 A	В	
Mix B-4	6	359.8	В	

Games-Howell Simultaneous 95% Cls Differences of Means for DCT Fracture Energy



If an interval does not contain zero, the corresponding means are significantly different.

Interval Plot of DCT Fracture Energy vs ID 95% CI for the Mean

