DEPARTMENT OF TRANSPORTATION

Feasibility Study of Cold Asphalt Recycling Technologies Using Rejuvenating Asphalt Emulsion: Impact, Implementation, Specification

Hassan Tabatabaee, Principal Investigator
Cargill, Inc.

August 2024

Final Report NRRA202205

National Road Research Alliance

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Technical Report Documentation Page

1. Report No.	2.	3. Recipients Accession No.
MN NRRA202205		

4. Title and Subtitle	5. Report Date
Cold Asphalt Recycling Technologies using rejuvenating asphalt emulsion: Impact, Implementation, Specification	August 2024
	6.

7. Author(s)	8. Performing Organization Report No.
Hassan A. Tabatabaee; Dan Swiertz; Tony Sylvester; Diane Franseen; Cristian Calcanas; Tracie Burton; Kim Gessner	

9. Performing Organization Name and Address	10. Project/Task/Work Unit No.
Cargill, Inc. 13400 15th Ave N, Plymouth, MN 55441 Bitumix Solutions (Subsidiary of H.G. Meigs and Asphalt Materials Inc.) 1220 Superior St, Portage, WI, 53901	11. Contract (C) or Grant (G) No. (c) 1035827

12. Sponsoring Organization Name and Address	13. Type of Report and Period Covered
Minnesota Department of Transportation Office of Research & Innovation 395 John Ireland Boulevard, MS 330 St. Paul, Minnesota 55155-1899	Final Report 14. Sponsoring Agency Code

15. Supplementary Notes

http://mdl.mndot.gov/

16. Abstract (Limit: 250 words)

Cold-recycling processes such as Cold in Place Recycling (CIR) and Cold-Central Plant Recycling (CCPR) offer opportunities for innovation through the use of recycling additives (RAs). The objective of this project was to evaluate the efficacy of rejuvenating asphalt emulsions in the CIR and/or CCPR process in terms of potential performance benefits relative to existing stabilization options. An experimental matrix was designed to include several of the mix design factors known or thought to control mix performance. Rejuvenating asphalt emulsions containing both Bio-based and petroleum-based RAs were produced and compared to a control engineered emulsion with a proven field history of performance. Inclusion of RAs did not negatively impact mixture stability or the mechanism of strength, while generally improving the CT Index of the tested cold recycled mixes compared to the use of a similarly graded control emulsion.

The concept of utilizing a “Balanced Mix Design” approach was explored to quantify the performance attributes of these materials. Mixture stability at 40 °C and mixture IDEAL CT Index at 25 °C were ultimately selected as the performance tests used in the balanced mix design framework. To aid rapid implementation of the results, the recommendations were also written in the form of a specification amendment document, included in the appendix of this report.

17. Document Analysis/Descriptors	18. Availability Statement
Cold in-place recycling, Recycled materials, Recycling, Rejuvenators, Mix design	No restrictions. Document available from: National Technical Information Services, Alexandria, Virginia 22312

19. Security Class (this report)	20. Security Class (this page)	21. No. of Pages	22. Price
Unclassified	Unclassified	75	

2019 Call for Innovation Research Project

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Final Report

Principal Investigator:
Hassan Tabatabaee, PhD
Cargill, Inc.

Co-Principal Investigator:
Dan Swiertz, PE
Bitumix Solutions
(a subsidiary of H.G. Meigs and Asphalt Materials Inc.)

Technical Contributors:

Tony Sylvester, Cargill
Cristian Calcanas, Cargill
Diane Franseen, Bitumix
Tracie Burton, Bitumix
Kim Gessner, Bitumix

March 2022

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Chapter 1 - Project Objectives and Introduction

America's roadway infrastructure is currently rated at a "D" level (A (best) through F (worst) scale) by the American Society of Civil Engineers (ASCE), with an estimated backlog in capital needs for road repair of $420 billion. The ASCE further recommends "support[ing] research and development into innovative new materials, technologies, and processes to modernize and extend the life of infrastructure…" as a means to help reduce the funding backlog and raise the infrastructure grade. Although certainly not "new" technologies, cold-recycling (CR) processes such as Cold in Place Recycling (CIR) and Cold-Central Plant Recycling (CCPR) offer opportunities for innovation that support this recommendation.

The CIR/CCPR processes have traditionally relied on commodity asphalt emulsion products (CSS-1, CSS-1h, etc.) or foamed asphalt as the stabilizing additive, either with or without the addition of cementitious materials. More recently so-called "engineered" asphalt emulsions have become common in some areas of the Country; these products are produced by careful selection of the base asphalt properties and/or emulsifier packages to match the unique needs of recycling processes.

Recent advancements in the use of asphalt Recycling Additives (RAs) in the hot mix asphalt and pavement preservation arenas have demonstrated the efficacy of RAs in increasing pavement or treatment performance relative to control materials. In particular, research into the use of RAs in high recycle content asphalt mixtures is demonstrating that through the proper selection and dosage, these mixtures can perform similarly or even better than control mixtures. This concept has been explored by several researchers using the framework of "Balanced Mixture Design", in which performance properties of the mixture are "balanced" across several modes of failure (e.g., rutting is balanced against cracking resistance) (Epps Martin, et. al, 2017).

The analogous nature of high recycle content mixtures and cold recycling processes such as CIR/CCPR suggests that the use of RAs in these processes might demonstrate particular utility in balancing mixture performance. Progress has been made in identifying key mixture and process-related variables that dictate performance in CIR/CCPR through recent National (NCHRP 09-51, NCHRP 09-62) and State-level research efforts, such as recent work by Braun Intertec for LRRB (Wegman and Sabouri, 2016). Leveraging the knowledge gained in these key projects with that gained from recent research into RAs and balanced mix design is the basis for this project.

In this project it is hypothesized that the use of RAs in asphalt emulsion stabilized CIR and/or CCPR processes can provide performance and/or cost benefits relative to commonly used ("Control") stabilization options. The specific project objectives stated in the project proposal are:

- Evaluate the efficacy of rejuvenating asphalt emulsions in cold recycling processes in terms of potential performance benefits relative to existing stabilization options (e.g., engineered emulsion, foamed asphalt) using concepts of balanced mixture design;
- Provide preliminary usage and design guidelines for the use of rejuvenating asphalt emulsion in Cold Recycling processes;
- Develop a "roadmap" for rapid implementation of a cold recycling test section utilizing rejuvenating asphalt emulsion.

Report Structure

This report is divided into 9 chapters as summarized below:

Chapter 2: Review of Use of Recycling Additives in Asphalt: This chapter presents a literature synthesis on the classification and efforts to standardize RAs for use in asphalt applications. Available literature pertaining to the use of RAs in the cold recycling industry is summarized.

Chapter 3: Review of Performance Testing and Curing Conditions for CR Processes: This chapter summarizes current cold recycling mix design methods and specifications with an emphasis on the NRRA partner states. Literature describing performance testing efforts for cold recycling materials is also summarized.

Chapter 4: Work Plan, Sample Preparation and Testing Methods: Based on the information presented in Chapters 2 and 3, a work plan is presented that includes selection of mix preparation and curing methods, mixture performance testing procedures, and asphalt residue characterization methods.

Chapter 5: RAP Material and Emulsion Characterization: Results from testing the RAP material, recycling additives, and base asphalts used in this study are presented. In addition, the quality control results from the various rejuvenating emulsions are presented.

Chapter 6: Mix Performance Testing and Analysis: This chapter presents the results of the mixture performance testing, including a summary of mixture volumetrics. Results from a sub-study on the effects of elevated mixing and compaction temperatures are presented. Sensitivity of the selected performance test methods to the factors evaluated in this study are discussed.

Chapter 7: Correlation of Binder and Mixture Results: This chapter explores the relationship between residue and mixture properties with the goal of recommending an RA dosage selection tool emulsion manufacturers can use in conjunction with mix designers to formulate rejuvenating asphalt emulsions for cold recycling processes.

Chapter 8: Interpretation and Proposed Balanced Design Framework: The results presented in Chapters 6 and 7 are used in this chapter to propose a balanced mixture design framework for cold recycling mixtures. The results from the performance testing portion of this study are used to propose tentative performance limits for future validation.

Chapter 9: Conclusions and Recommendations for Implementation*:* A summary of major findings is presented in this chapter along with recommendations to validate the proposed performance limits and for rapid implementation of trial projects utilizing rejuvenating asphalt emulsions.

Chapter 2. Review of Use of Recycling Additives in Asphalt

Definition and Categorization of Recycling Agents

With increased service demands from asphalt pavements through ever-increasing volumes coupled with aging highway networks, the need for enhanced durability through smart and sustainable pavement preservation and preventive maintenance is more important than ever. Rejuvenating technologies have been utilized in pavement preservation at both research and commercial capacities for many years, often starting with proprietary applications. However in recent years the push to use such products has become more mainstream, including a diverse range of applications such as rejuvenating fog seals and scrub seals, in-place recycled mixes, and increasingly, hot mix asphalt pavements.

A number of both petroleum- and biological-based additives proposed as recycling agents have been investigated in the literature (1; 2; 3; 4). Different types of categorization may be employed for such material based on the source, manufacturing process, or based on the bitumen fraction most affected by the additive and the expected mechanism of affecting those compatible fractions upon addition to aged bitumen (5; 6; 7).

An example of a source-based categorization system was released recently by The National Center for Asphalt Technology (NCAT) citing five basic categories, as shown in Table 1 (8). It should be noted that although this categorization is convenient, it does not account for the differences in performance that may be achieved using various products in the same category based on physical and chemical properties, chemical modifications, and production process.

Table 1 Types of Recycling Additives for Asphalt (8)

CATEGORY	DESCRIPTION
Paraffinic Oils	Refined used lubricating oils
Aromatic Extracts	Refined crude oil products with polar aromatic oil components
Nathenic Oils	Engineered hydrocarbons for asphalt modification
Triglycerides & Fatty Acids	Derived from vegetable oils *Has other key chemical elements in addition to triglycerides and fatty acids.
Tall Oils	Paper Industry byproducts Same chemical family as liquid antistrip agents and emulsifiers

Categorization based on mechanism is thought be a potentially better way to categorize recycling agents, as it involves the nature of the impact. There are a number of studies and proposals underway to

address this type of categorization. One such proposal based on the bitumen fraction most affected by the additive and the expected mechanism of effect upon addition to aged bitumen (5; 6; 7), is as follows:

- "Soluble Softener", which supplement the "solvent" phase of the bitumen colloidal structure by being most compatible with the low polarity naphthenic aromatic fraction of the bitumen. Such additives reduce the viscosity and modulus of the overall bitumen through lowering the viscosity of the continuous solvent phase, but may have little effect on the intermolecular agglomeration and self-assembly of the polar micelles.

- "Compatibilizers", which have affinity for multiple fractions in the bitumen and may be derived through careful engineering of the source material, whether Petroleum- or bio-based. In addition to reduction in viscosity, these additives are hypothesized to result in a reduction in high molecular weight micelle agglomerations through disruption of the intermolecular associations and molecular self-assembly, similar to the postulated effect of the bitumen "resin" phase.

- "(Phase-) Incompatible Softeners", which often exhibit low compatibility with the low polarity naphthenic aromatic and polar fractions, especially at lower temperatures. This category may include some paraffinic and saturated material with high crystalline fractions. It was speculated that although dispersion of such lower viscosity additives in the bitumen may still achieve a reduction in overall bitumen modulus, increasing the dosages of "insoluble softeners" in asphalt may lead to colloidal instability and the long term durability and phase stability may be compromised (9; 5).

However, although academically relevant, there are no widely approved criteria or methodologies for identification of how a given product should be classified based on category, making this type of categorization less practical for implementation in practice at this time. Studies are underway to develop practical methods for characterizing binders and additive combinations based on compatibility that may aid implementation of such methods in the future.

In interim, and in the absence of mechanism-based categorizations, perhaps one of the earliest methods for classifying RAs may be useful. This method, as described in ASTM D4552 (10), is based on physical properties, chemical compositional properties, and direct aging and oxidative stability of the recycling agents, with corresponding threshold values shown. The purpose of the classification is to address aspects regarding meeting minimum required safety, stability, and compatibility measures, while not directly addressing performance on the final asphalt binder or mixture. The document originally only addressed petroleum-based additives, however it was updated in 2020 to also include bio-based recycling agents. The RAs are further categorized by viscosity, however the viscosity categorization is mostly geared towards providing generic form of identification, rather than being indicative of any specific performance or utility aspect. The specification is shown in Table 2.

Table 2. Classification of Recycling agents per ASTM D4552-20 (10)

Test	ASTM Test Method	RA 0		RA 1		RA 5		RA 25		RA 75		RA 250		RA 500	
		Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Viscosity • 60 °C [140 °F], mm^2/s	D2170	10	49	50	175	176	900	901	4500	4501	12500	12501	37500	37501	60000
Flash Point, COC, °C [°F]	D92	219 [425]	...	219 [425]	...	219 [425]	...	219 [425]	...	219 [425]	...	219 [425]	...	219 [425]	...
Saturates, wt. %[A]	D2007	...	30	...	30	...	30	...	30	...	30	...	30	...	30
Tests on Residue from RTFO 163 °C [325 °F]	D2872														
Viscosity Ratio[B]	"	...	3	...	3	...	3	...	3	...	3	...	3	...	3
Wt Change, ±, %	"	...	4	...	4	...	4	...	3	...	3	...	3	...	3
Specific Gravity at 25 °C [77 °F]	D70 or D1298	0.900	1.100	0.900	1.100	0.900	1.100	0.900	1.100	0.900	1.100	0.900	1.100	0.900	1.100

[A] The suitability of D2007 for measurement of saturates content and determination of compatibility of non-petroleum-based recycling agents has not been established. Additional testing may be required for assessment of the compatibility of non-petroleum-based recycling agents.

[B]

$$ViscosityRatio = \frac{Viscosity\ of\ Residue\ from\ RTFO\ Test\ at\ 60°C\ [140°F]}{Original\ Viscosity\ at\ 60°C\ [140°F]}$$

Use of Recycling Agents in In-Place Recycling

There is very little literature mention of use of RAs in Cold Recycling type applications. Therefore in this section many of the discussed studies and cases are more focused on Hot In-Place (HIR) recycling type applications. However, the research team believes there is much to be learned from HIR rejuvenation processes, and specifications, that may be of use in a cold application. Therefore such literature is reviewed in this section.

The terminology used in the literature can also lead to some confusion, as often any bituminous addition to the CIR is referred to as a "recycling agent" in the CIR literature, in which case the intent is clearly different from the more widely used definitions of recycling agents as additives incorporated into the bitumen, as was described in the previous sections.

The NCHRP synthesis report number 421 (11) references RAs as being defined by the aforementioned ASTM D4552 if in oil form, or by the ASTM D5502 if in emulsion form. These RAs can be used in conjunction with or instead of new binders to improve performance. The report also references the "Pacific Coast Asphalt Specification" as defining RAs as being "hydrocarbons with physical properties that restore aged asphalt to current specifications". However, no direct reference is cited for the definition.

The ARRA cold recycling manual (12) refers to emulsified recycling agents as being a normal tool for use in CIR mix designs. The wording implies that this is mostly referring to cationic asphalt emulsions, and not RA oils. However, the guidance provided with regards to the use and efficacy of RAs is still deemed useful and applicable.

The dosage is based solely on viscosity and the effectiveness of the given RA in reducing the viscosity of the aged asphalt binder. The time and temperature of interaction between the RA and asphalt are deemed an important factor. Further factors are degree of aging of the aged asphalt, mechanical impact of mixing, compaction, and traffic, and the climate conditions. The manual recommends that the mechanical properties of the mix be determined both before and after the final curing, rather than simply relying on blend charts.

Looking a bit further afield and into HIR use of RAs, one can refer to the general viewpoints outlined by ARRA with regards to incorporation of RAs in HIR mix designs (12):

1. Only use an RA, relying on its ability to effectively combine with the aged asphalt binder.
2. Only use a soft virgin asphalt binder, to create a combined "average" binder with adequate properties.
3. Use both an RA and a soft virgin asphalt binder
4. Use the final recycled mix properties such as resilient modulus or stability, rather than binder properties, to determine the final mix selection.

This document also makes some interesting comments with regards to the impact mechanisms and performance needs of an RA when used in an HIR, mainly, the ability to penetrate into the aged asphalt at the temperature and conditions of HIR mixing. If the RA fails to properly penetrate it may form a lubricating layer on the RAP particles, which can cause instability and appearance of "over-asphalted" wet mix. The report further mentions an uncited FHWA studies from the 1970s in which a two-part dye system was used to demonstrate the ability of RAs to fully and uniformly diffuse and distribute into an HIR mix, if proper temperature and mixing time was provided. The amount of diffusing dramatically slowed when temperatures were cooled (12). Other sources have specifically listed adequate temperature for mixing the rejuvenating agent and admixtures, cited as typically being between 230°F and 320°F (13).

Optimization of the mix design are possible through either the binder itself or making the final mixes at different RA contents and extracting and recovering the binder for comparisons. The Hveem stability was also noted as being more sensitive to RA content than the Marshall stability, although both registered the impact. Alternatively, it is noted that by testing the Resilient Modulus through diametric loading, tensile loads become the predominant mode of loading, decreasing the impact of aggregate structure and therefore showing greater sensitivity to the impact of an RA on the properties of the binder (12).

In one of the only examples of research specifically focused on use of RAs in cold recycling, Swiss researchers used a number of vegetable oil based products with and without added asphalt binder to produce cold mix samples (14). The mix design deferred from typical CIR design used in North America in that the asphalt binder or additive were not emulsified or foamed when added to the RAP. Instead the RAs or an RA-binder blend were added directly to the RAP followed by the addition of water (about 5-6% water by weight of the mix). Mixes were cured at 23 and 40°C for multiple days, showing that curing, as measured by compressive strength, was achieved after 14 to 30 days, depending on curing temperatures. The mixes showed sensitivity to moisture and saturation, but seemed to perform well in both the lab and field test sections when dry, as shown in Figure 1. The researchers suggested that good drainage in the underlying layer would be necessary to prevent such damage.

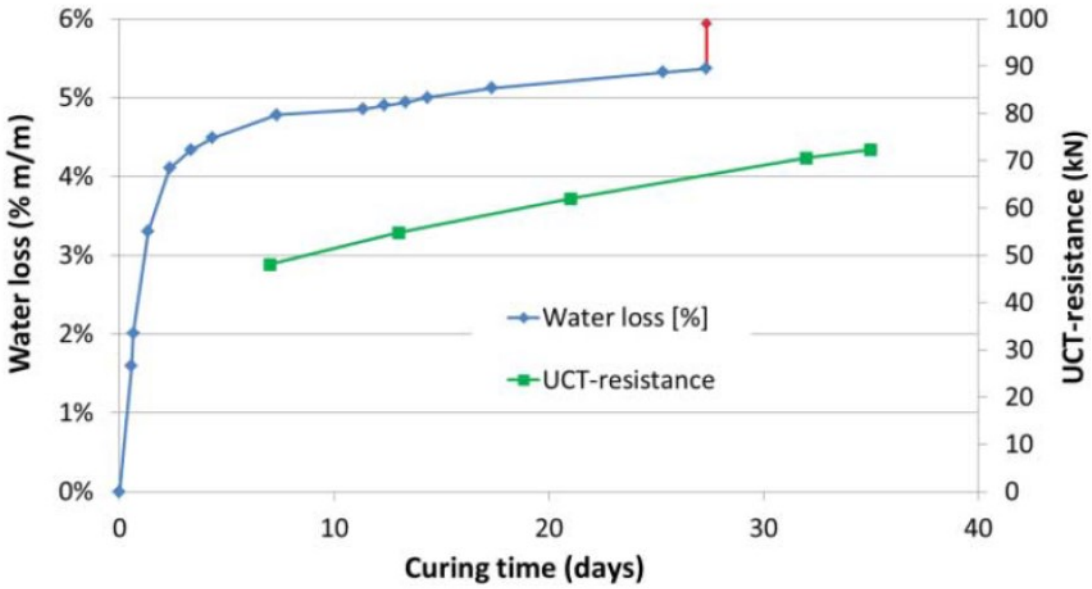


Figure 1. Rate of water loss and compressive strength gain during curing at room temperature (14)

The aforementioned study is of use and relevance to the current project as it is one of the only examples of well documented RA curing studies in a cold recycling setting. However it is anticipated that the results may vary from that expected to be achieved in the present project due to key expected differences in material type and design, specifically the dosage levels of the RA, the use of emulsified asphalt and/or RA, and the compaction process and excess moisture during compaction, all which have the potential to significantly affect the curing process. The materials and methods will be further discussed in detail in later chapters.

Chapter 3- Review of Performance Testing and Curing Conditions for Cold Recycling (CR) Processes

According to the Asphalt Recycling and Reclamation Association's Basic Asphalt Recycling Manual (ARRA-BARM), no unified or nationally accepted design methodology exists for CR processes. As a result, materials, processes, and design requirements vary across the U.S. and abroad (12). The purpose of this section is to summarize the existing state of mix design methodologies among agencies, associations, and private entities as well as summarize landmark literature exploring the use of performance-based methods to evaluate CR materials. In addition, two recent research efforts evaluating recycling additives in Hot Mix Asphalt are also summarized since they may be directly applicable to this project. The information in this section will be used to guide the research team in selecting test methods and curing conditions for the present study.

Synthesis of Select Existing CR Mix Design Methods and Specifications

Table 3 summarizes existing CR design methods for NRRA partner agencies, other select State Agencies, as well as relevant association and private industry information – collectively called "entities" in this section. Information for the various State Agencies was obtained by searching current DOT Standard Specifications, special provisions, and DOT design manuals for each State. Other design information was found by searching relevant industry publications (e.g., ARRA-BARM, Wirtgen Cold Recycling Manual), published specification databases (e.g., AASHTO), and resources available via internet search. In all, 15 entities were reviewed, 13 of which had a published mix design processes with testing required. The purpose of this summary is not to be exhaustive of all available design methods, but to provide a representative database of methodologies to guide this project.

All of the entities reviewed specify a gradation analysis on the bulk RAP (black rock gradation), and at least two specify a washed gradation after extraction. Mankato is unique among entities reviewed in also specifying RAP aggregate quality using sand equivalent and Fine Aggregate Angularity. Three of the entities specify a Modified Proctor Test (AASHTO T180) to determine the optimum moisture content for compaction of the RAP material. The Modified Proctor Test is a standard method in the geotechnical engineering community, as is of relevant note for this study since most of the entities specify or allow gyratory compaction for sample preparation (the Modified Proctor Test relies on impact for compaction).

Sample preparation generally follows one of two methods: Superpave Gyratory Compactor (SGC) with a compactive effort of 30 gyrations or Marshall Compaction with a compactive effort of 75 blows. Two of the Agencies reviewed allow either SGC or Marshall, and the FHWA-FLH method specifies 35 gyrations. The ability of the SGC to simulate field compaction of CR mixtures has been reported extensively in the literature with little consensus to the most field-replicable approach (15). Most Agencies specify reporting of volumetric properties following compaction and curing, typically using AASHTO T166 (saturated surface dry method) for bulk specific gravity (Gmb) and AASHTO T209 (so-called "Rice" method) for Maximum Specific Gravity (Gmm), although at least one research study reported problems using these methods for CR mixtures, and instead recommended vacuum sealing methods (15).

Table 3. Summary of Required Mix Design Tests and Curing Conditions for Select Agencies, Associations, and Design Documents

	Entity	Required Testing	Curing Conditions
NRRA Agencies	California[1]	- Asphalt content of RAP and Ratio of Residue to Cement - 4" Marshall Stability and Retained Stability after moisture conditioning - RAP Coating Test - Raveling Test	- Stability: 140 F to constant weight, but between 16-48 hours (note: a reduction in retained stability percentage is allowed for higher levels of dry stability - Raveling: ASTM D7196 at 50 F
	Illinois[1]	- 4" or 6" Marshall Stability and Retained Stability after moisture conditioning - Raveling Test	Not specified
	Iowa	- No mix design; specified additive rate (0.30 gallons/SY/in)	NA
	Michigan[1]	- Modified Proctor for Optimum Moisture Content - 4" or 6" Marshall Stability and Retained Stability after moisture conditioning - Raveling Test	Not specified
	Mankato[1]	- Washed (after ignition) gradation, sand eq., and FAA of RAP - 4" Marshall Stability and Retained Stability after moisture conditioning - IDT for Thermal Cracking - Raveling Test	- Stability: 140 F to constant weight, no less than 16 hours; bring to ambient between 12-24 hours - IDT: 60 C for no less than 48 hours, no more than 72 hours - Raveling: Ambient lab temp (65-75 F) for 4 hours
	Missouri	- No mix design procedure could be located	NA
	North Dakota[1]	- 4" Marshall Stability and Retained Stability after moisture conditioning - IDT for Thermal Cracking - Raveling Test	- Stability: 60 C to constant weight between 16 and 48 hours; bring to ambient between 12-24 hours - Raveling: 4 hours at 10 C and 50% humidity
	Wisconsin[1]	- IDT at 25 C; TSR - RAP Coating Test - Minimum Virgin Asphalt Content	Not Specified
Select Other Agencies	Kansas[1]	- 4" Marshall Stability and Retained Stability after moisture conditioning - IDT for Thermal Cracking - Raveling Test	- Stability: 140 F to constant mass, between 16-48 hours; cool for 18+-6 hours - Raveling: Lab temperature for 4 hours
	Colorado[1]	- Hveem Stability - IDT (Lottman) for Moisture Resistance - IDT for Thermal Cracking - Raveling Test - Fracture Energy (DCT) and Hamburg Wheel Tracking at 50 °C[4]	- Stability: 140 F to constant mass, between 16-48 hours; cool for 12-24 hours - Fracture Energy and Hamburg, when applicable: 48-72 hours at 60 C - Raveling: 10 C and 50% humidity
	Virginia[1]	- Modified Proctor for Optimum Moisture Content - 4" or 6" Marshall Stability and Retained Stability after moisture conditioning - IDT for Thermal Cracking - Raveling Test	- Stability: 140 F to constant mass. - Raveling: 20 gyrations; cured 4 hours at 50 F and 50% humidity
	FHWA - FLH 524[2]	- IDT with moisture subset per T283 - Raveling Test	- IDT: 140 F to constant mass; between 16-48 hours; cool to ambient 12-24 hours - Raveling: 4 hours at 50 F and 50% humidity
Association/Company	ARRA[1]	- Marshall Stability with moisture subset or IDT TSR- - Raveling Test - Thermal Cracking - BBR (M320) or IDT (T322)	- Stability: 140 F to constant mass or specified time, usually 48 hour max.; cooled overnight to ambient- Raveling test: 4 hours at 50 F and 50% humidity
	AASHTO PP86 & MP31[1]	- Choose EITHER: Marshall Stability with moisture subset or IDT with moisture subset - Minimum ratio of asphalt content to cement - Raveling Test	- Stability or IDT: 60 C to constant mass; between 16-48 hours; cool at 25 C between 12-24 hours - Raveling: 4 hours at 50 F and 50% relative humidity
	Wirtgen[3]	- Modified Proctor for Optimum Moisture Content - IDT, dry and moisture conditioned - Unconfined compression strength - Triaxial (California Bearing Ratio) Shear Testing	- IDT: 72 hours at 40 C; allow to cool to ambient - For High Volume: Pill placed in plastic bag; 48 hours at 40 C; remove from bag, cool to ambient

[1]Volumetric analysis reported; Superpave Gyratory Compactor effort of 30 gyrations; California & Virginia allow 75 blow Marshall; Virginia does not directly specify reporting of volumetrics.

[2]Volumetric analysis reported; Superpave Gyratory Compactor effort of 35 gyrations

[3]Volumetric analysis reported; Marshall Method with effort of 75 blows

[4]Included in design requirements at discretion of regional engineer and for "high volume traffic locations or when required to direct-load the recycled material from the recycling train into the laydown paver hopper"

In terms of mixture testing, 10 of the 13 entities that included mixture testing specify the Marshall Stability test, with several of the entities allowing either 4" or 6" specimens. There is support in the literature for a standard correction of stability values between 4" and 6" samples, although this correction was developed for hot mix asphalt (16); the research team for this project hypothesize that differences in surface area to volume ratios between 4" and 6" specimens may impact curing rate and should be taken into account when designing the experiment for this project. The entities that specify Marshall Stability usually include a provision for a moisture conditioned subset to measure moisture susceptibility via a minimum retained stability percentage.

Five of the 13 entities specify an Indirect Tensile Test (IDT) at intermediate temperature (e.g., 25 °C) along with a moisture conditioned subset to measure moisture susceptibility following AASHTO T283, ASTM D4867, or modifications thereof. Moisture conditioning generally follows a similar approach to that used in the hot mix asphalt industry: samples are vacuum saturated to a specified percentage of fully saturated, conditioned in a hot water (e.g., 60 °C) bath for a specified time, returned to ambient lab temperature (e.g., 25 °C), and tested.

The Colorado DOT is unique among entities reviewed in specifying the Hamburg Wheel Tracking Test (AASHTO T324) and Disc-Shaped Compact Tension (DCT) tests for special design scenarios (generally high traffic). The researcher team found reference to using the Hamburg test and Asphalt Pavement Analyzer (APA) for CR mixtures in at least one other literature study (15). Of note, the Wirtgen Cold Recycling Manual also lists unconfined compressive strength and triaxial testing for CR mixtures, the only entity reviewed to do so (17). This requirement appears to be the result of international design standards.

Interestingly, six of the 13 entities specify AASHTO T322 IDT for low temperature thermal cracking evaluation. For some of the entities this test result is listed as "Report Only". This finding was particularly interesting because AASHTO T322 is not widely used in practice in the hot mix asphalt community and is usually used in applications of mechanistic structural design of pavement. As mentioned, use of the Disc-DCT appears in one of the specifications reviewed (Colorado) and low temperature Semi-Circular Bend (SCB) testing of CR mixtures has been reported in the literature to evaluate thermal cracking susceptibility.

The ARRA-BARM mentions use of the asphalt binder Bend Beam Rheometer (BBR, AASHTO M320) results to measure thermal cracking resistance. Use of blended binder properties to predict performance of CR mixtures also has support in at least one other reviewed publication (12; 11). The research team for the NCHRP 09-51 project used BBR specimens fabricated from mixture samples for low temperature evaluation (18).

Eleven of the 13 entities specified a form of the Raveling Test, ASTM D7196. This test is typically used to measure the rate of curing in CR mixtures and is usually the test method that determines the minimum amount of stabilizing additive (emulsion or foamed asphalt) used in the mix design. As such, preparation of samples for this test usually follows a different approach than for the other test methods. No other test intended to measure curing characteristics was found during this review. A minority of the entities reviewed also included RAP Coating tests, minimum virgin asphalt content requirements, and ratio of virgin asphalt to active filler requirements. Iowa simply specifies a target additive rate of 0.30 gallons/SY/in for all CIR projects.

Curing conditions used during the mix design process can generally be broken down into two categories: fully cured or partially cured. When curing conditions are specified, full curing is always

specified for Marshall Stability, IDT specimens, Hamburg specimens, and DCT specimens. Full curing is most typically defined as curing samples in a 60 °C (140 °F) oven to a substantially constant mass, but no less than 16 hours and no more than 48 hours, then allowing samples to cool for approximately one day (usually specified as 12-24 hours). Interestingly, curing conditions were not found for Illinois, Michigan, or Wisconsin despite these entities having performance test requirements. Partial curing is always used for preparing Raveling Test specimens, usually specifying a temperature, time, and humidity. Most commonly specified is 10 °C (50 °F) for four hours; when humidity is specified, 50% is usually used. Mankato specifies ambient laboratory curing for four hours, without specifying humidity. The Wirtgen Cold Recycling Manual includes a provision for curing samples intended for use on higher volume roadways; in this instance samples are cured in a sealed plastic bag (i.e., in the presence of moisture) (17).

Synthesis of Select Literature Relating to Performance Testing and Curing of CR Mixtures

In this section several landmark studies relating to performance testing and curing of CR mixtures and use of recycling additives are summarized. The references listed below are not meant to be exhaustive, but rather demonstrate a chronological progression of performance testing of CR mixtures over the last approximately 10 years with the goal of providing this research project justification for selecting certain methods. In addition, two recent studies on use of recycling additives for high RAP mixtures are included because they may contain parallels to this project. References are summarized individually, although the information is applied holistically to develop a testing plan.

Charmot, Evaluation of the Fracture Energy Properties of Cold Recycling Asphalt Aggregate Mixtures (2009)

The purpose of this dissertation was to apply then recently developed asphalt mixture fracture mechanics principles generated from the Disc Shaped Compact Tension (DCT) Test to CIR mixtures in an attempt to better explain differences in field performance of these mixtures. Major conclusions were:

- Transverse cracking, particularly reflective cracking, is defined as an important metric of success for CR mixtures;
- Fracture energy related properties are shown to be sensitive to mix design factors and are correlated with field performance of CIR mixtures.
- The Disc-Shaped Compact Tension Test (DCT - ASTM D7313) was used to successfully characterize CR mixtures and was shown to correlate with reflective cracking propensity in field projects; and was therefore recommended by the author for more intensive evaluation.
- The author notes that an indicator of mixture stability/rut resistance is also needed (e.g., the “Balanced Mixture Design” concept) (19).

Stroup-Gardiner, NCHRP Synthesis 421 (2011)

Although not a research project involving testing, this synthesis provides a thorough “State-of-Practice” summary of the CR industry at that point in time. Both Agency and private sources were reviewed for this synthesis. With regard to material testing, the following important information is presented:

- A framework for using blended asphalt binder properties is presented;
- The most commonly used mix design method used by State Agencies is to do nothing, although several reported using either SGC or Marshall methods;
- Load-carrying capacity (stability) of CR mixtures is most typically measured with either Marshall Stability or IDT, although there is mention of the Asphalt Pavement Analyzer and Hamburg Wheel Tracking Test being used;

- It is overall concluded that more standardization needs to occur before any unified design specification can exist (11).

Cox & Howard, MDOT: Cold In-Place Recycling Characterization Framework and Design Guidance for Single or Multiple Component Binder Systems (2015)

This report provides a comprehensive review of available performance testing methods and curing conditions for CR mixtures. The overall objective of this study was to determine properties of CR mixtures that control performance in high-traffic design scenarios.

- CIR compaction using 30 gyrations is recommended based on comparisons to field compaction;
- Asphalt Pavement Analyzer (APA) wheel tracking via typical Mississippi DOT protocols and Indirect Tension Test (IDT) testing were recommended by the authors;
    - The use of the Hamburg Wheel Tracking Test for CIR mixtures is reported for the Texas DOT, but otherwise the authors could not find other documented literature;
    - Extensive use of the Marshall Stability test for CIR is documented, although the test may not fully capture the benefits of using active fillers (e.g., cement);
- Indirect Tensile Strength and using fracture energy measurements at intermediate temperature is recommended in a balanced mix design framework;
- The authors recommend curing CIR samples in a humidity-controlled oven at 40 °C and between 35-50% humidity to simulate field conditions (in Mississippi) (15).

Wegman & Sabouri, MnDOT: Optimizing Cold In-Place Recycling (CIR) Applications Through Fracture Energy Performance Testing (2016)

The objective of this study was to characterize CR mixtures using two performance tests methods: the SCB and DCT in order to better understand the correlation between laboratory design and mixture performance. The authors introduced a newly derived parameter to estimate fracture energy, although the derivation can be considered similar to other fracture energy parameters (area under a load-displacement curve). Although the authors concluded that the fracture energy approach was viable to characterize CR mixture material, the bulk of the data set includes RAP material from only one design while varying stabilizing additive content and type and active filler content.

Although the testing showed differences in average fracture energy among the design iterations, statistical significance between the samples within a given laboratory is questioned (Figure 2); in Figure 1, dotted red lines are used to demonstrate the overlap in data; considering the variability of the “Foam” data set, the average fracture energy value the three other data sets all fall within the error bar range of Foam. A statistical analysis was conducted by the authors for inter-laboratory variability between two of the labs, but no such analysis was presented between samples within a single laboratory. The authors acknowledge a larger data set is needed to support the use of this method.

Based on the findings of this study, the following conclusions pertinent to the present project are offered:

- Use of fracture-energy derived parameters is further supported;
- Sample handling and geometry can significantly affect the results of such testing;
- The use of active filer (cement) did not appear to appreciably change the fracture energy measured for the mixtures in this study, and in fact resulted in the lowest measured fracture energy (20). This finding may further support the need for a stability/rutting test at higher temperature in any “balanced mixture design” framework.

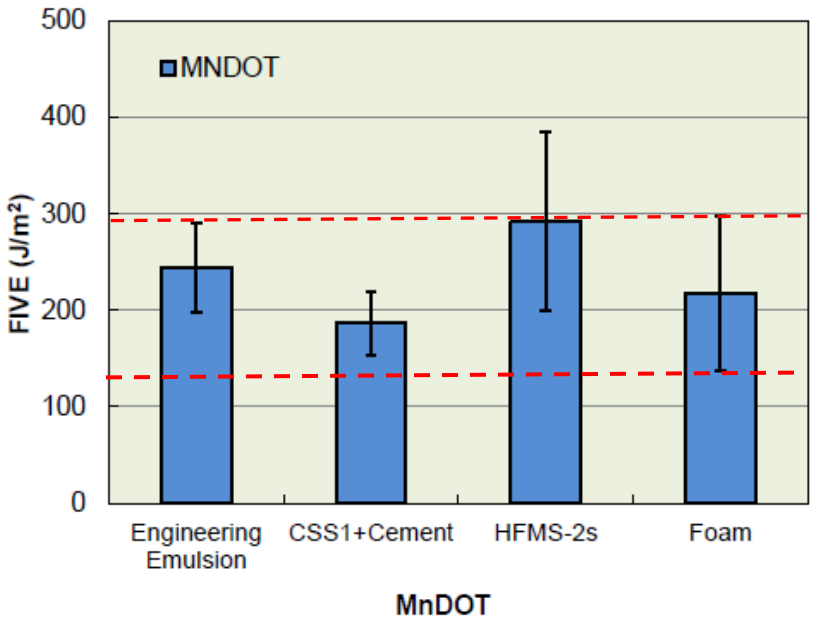


Figure 2. Fracture energy analysis for four design iterations using the same RAP source. Note the differences in average fracture energy, but relative overlap in error bars. From (20), with red lines added by the current project research team.

Schwartz et al. NCHRP 09-51: Material Properties of Cold In-Place Recycled and Full-Depth Reclamation Asphalt Concrete (2017)

The main objective of this large national research effort was focused on the evaluating the structural properties of CR mixtures in the lab and field for pavement design purposes. Pertinent to the present study, however, was the finding that the stiffness master curves for both asphalt emulsion and foamed asphalt generally overlapped and no statistically significant difference was found between the two stabilizing additives; practically speaking, the emulsion stabilized mixtures were less stiff at high temperature relative to the foamed asphalt. Interestingly, "no strong correlations were found between mixture characteristics (e.g., volumetrics, gradation, density) and stiffness". In general, active filler such as cement reduced temperature susceptibility and benefited mixture stiffness. Rutting of CR mixtures is a primary performance concern as well (18).

In general, the results of this study suggest use of asphalt emulsion should provide similar performance results as foamed asphalt, particularly for cured specimens under the present study conditions. Rutting/stability will be primary area of study and the use of active fillers may be considered if marginal values of stability are encountered.

Wegman & Sabouri, MnDOT: Evaluating Effective Asphalt Content in CIR Mixtures (2019)

Wegman & Sabouri note that it is relatively common practice to reduce the design asphalt emulsion content of CR mixtures during hot weather to "ensure constructability" in the field. The concern with such practice is potentially reduced performance of the CR mixture due to insufficient residue. The authors hypothesized that greater RAP binder "activation" at high temperature was the causal factor. To simulate this phenomenon in the laboratory, three different mixing temperatures (temperature of the RAP at time of mixing) were used: ambient lab temperature, approximately 110 °F, and approximately 125 °F. Note that the specified Superpave high temperature grade for most of Mankato is 58 °C or approximately 136 °F. Findings indicated:

- Higher mixing temperatures reduced the optimum emulsion content determined in the mix design, with higher mixing temperature resulting in:
    - Higher compacted sample density;
    - Higher dry stability at the determined optimum emulsion content;
    - Generally higher retained stability;
    - Reduced raveling resistance (Figure 2);
    - Higher (closer to zero) critical low temperature for cracking, but higher overall IDT strength;
- Use of fracture energy testing determined that there was no statistical difference in mean fracture energy values between the three mixing temperatures (Figure 3).

The authors concluded "…that reducing the emulsion content of the CIR mixtures during the heat of the day does not necessarily deteriorate the mixture properties." (21) This is an important analogy to the present study since it is hypothesized that use of recycling additives may likewise reduce the optimum emulsion content by way of RAP binder "activation". Those mixture properties that were negatively affected by a reduction in stabilizer content (e.g., raveling) are therefore of higher concern in the present study. Overall, this study further supports the use of a balanced mixture design concept for CR mixtures.

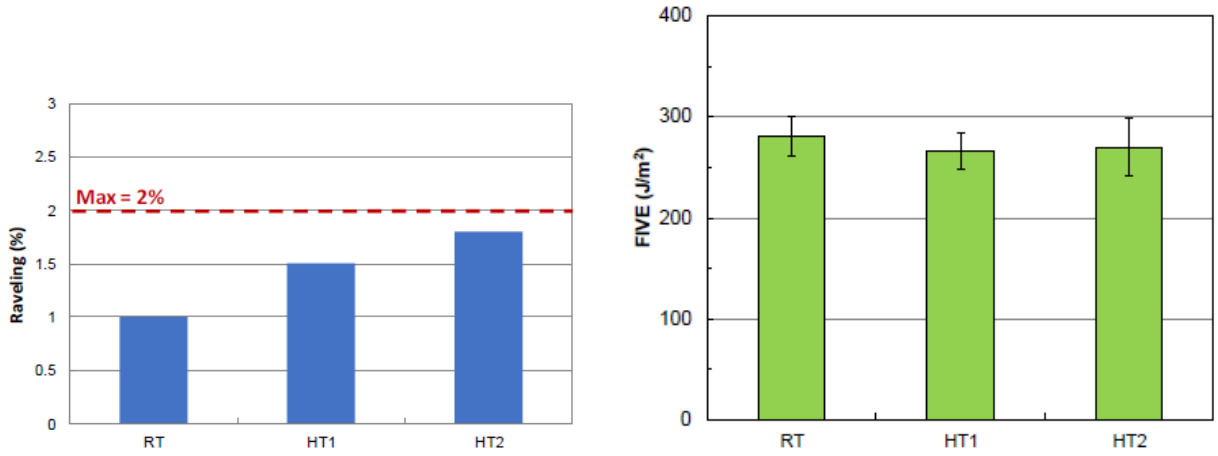


Figure 3. Effect of mixing temperature (HT2 > HT1 . RT) on raveling resistance and fracture energy supporting the balanced mixture design concept. From (21).

Dong & Charmot, Proposed Tests for Cold Recycling Balanced Mixture Design with Measured Impact of Varying Emulsion and Cement Contents (2019)

The authors of this study have published several other relevant articles relating to CR mixture testing; this reference was chosen in particular because it is the most recent and references much of their respective earlier work. This article presents a "Balanced Mixture Design" approach to CR mixture design and demonstrates the efficacy of such an approach for various emulsion-stabilized CIR materials, with and without cement. Major findings are:

- A balance between rutting (stability) and cracking is required for optimal performance of CR mixtures;
- A wheel tracking rutting test at 60 °C was used to evaluate rut potential of CR mixtures;
    - Reducing emulsion content and increasing cement content had a positive effect on rutting resistance of the CR mixture;

    - CR mixture rut resistance can meet or exceed minimum asphalt concrete limits and the authors believe a wheel-tracking test is critical for high volume designs;
- The IDEAL-CT cracking test was used to measure cracking indices of various CR mixtures
    - Low emulsion and/or high cement content CR mixtures exhibited “quasi-brittle” behavior;
    - ANOVA testing demonstrated the IDEAL-CT can differentiate between mixture design factors such as emulsion content and cement content;
- The authors conclude that a balance between rutting and cracking is required for CR mixtures;
- The authors recommend including moisture damage resistance as part of a CR balanced mixture design framework (22).

Casillas et al. Asphalt Emulsion Cold In-Place Recycling Mix Design Practices: Designing a Semi-Bound Pavement Material (2020)

This reference is a white paper summarizing the history and current State-of-Practice for the design of CR mixtures. The primary contribution of this white paper to the current project is the concept of better understanding the role of cohesion gain during curing of CR mixtures (Figure 4). Since most of the design and testing information summarized thus far specified a single curing condition, little information is gained with regard to the transition of CR mixtures from essentially unbound granular materials to semi-bound or bound flexible layers. For the present study, this concept may be critical to better understanding the effect of utilizing recycling additives for CR processes (23). For example, if final cured stability is similar between traditional mixtures and “rejuvenated” mixtures but initial stability is significantly different, early-life field performance could similarly be drastically different. Practically speaking, since the use of recycling additives differs from standard practice, measuring performance using methods developed for standard practice may be misleading.

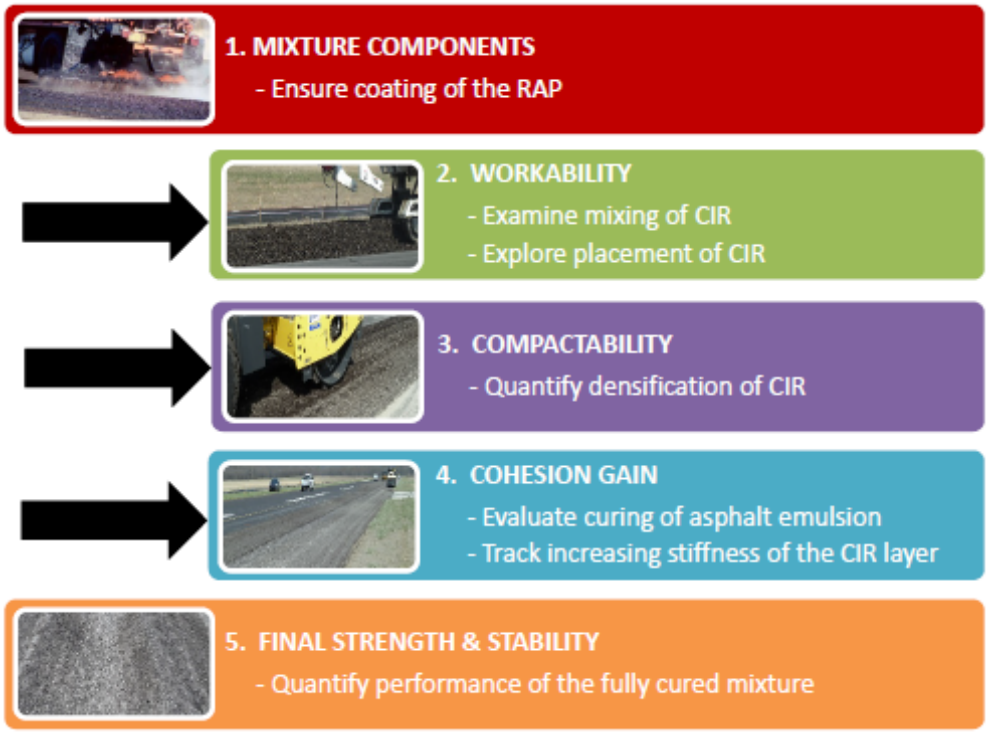


Figure 4. Proposed design considerations for CR mixtures, from (23)

Diefenderfer et al. NCHRP 09-62: Rapid Tests and Specifications for Construction of Asphalt-Treated Cold Recycled Pavements (2020)

The objective of this national research effort was to produce a series of tests to evaluate when CR mixtures could be considered ready for trafficking and/or surfacing. Although primarily a field-based study, the authors concluded that shear and raveling-based properties were most practical to quantify time to opening. With regard to the present study, this recommendation suggests a relative evaluation of curing rate should be considered as this appears to be of primary concern in the field (24). The authors hypothesize the raveling test could be used in such a framework with the objective of comparing mixtures without recycling additives (control) to those with recycling additives. The findings of NCHRP 09-62 might be particularly useful if a field evaluation project is planned as a result of the present research opportunity.

Epps-Martin et al. NCHRP 09-58: Evaluating the Effects of Recycling Agents on Asphalt Mixtures with High RAS and RAP Binder Ratios (2020)

Although this national research effort focused on warm and hot mix asphalt mixtures, direct analogies can be made to the present study, namely use of mixture performance coupled with binder testing to quantify the efficacy of recycling additives. Major findings of NCHRO 09-58 relevant to this study include:

- Use of blended binder properties to estimate dosage of recycling additives and predict subsequent mixture performance is confirmed;
    - Extensive use of the Glover-Rowe parameter at 15 °C and ΔTc determined from BBR testing is noted in this study;
- Balancing the mixture blended binder high temperature PG is recommended to select optimum recycling additive dosage, suggesting high temperature rutting/stability is a critical design parameter for mixtures containing recycling additives;
- No significant difference in recycling agent efficiency was noted when the recycling agent was added directly to the RAP as opposed to blended with the virgin mixture binder, although a clear effect on RAP binder availability was measured for the factors of conditioning time, conditioning temperature, and RAP binder stiffness (25). In other words, for the present study it is expected that each unique RAP source and mixing condition could have a different “optimum” recycling additive dosage

Bahia et al. Recycled Material Research Center: Long-Term Performance of Asphalt Concrete Mixed with RAP and RAS (2020)

This study again focused on the effects of recycling additives on hot mix asphalt but provided findings useful to the present study. The central mixture performance test used in this study was the IDEAL-CT (ASTM D8225); the test was used to evaluate mixtures at the standard 25 °C, but also at 0 °C as a surrogate for more time-, material-, and cost-intensive low temperature cracking tests such as the DCT. Although the greater objective of this study was to measure the effects of long-term aging on mixture performance, useful correlations between mixture performance and blended binder properties were discovered.

Figure 5 shows the correlation between the CT Index value measured at a respective temperature and a blended binder property. At 25 °C, CT Index is reasonably correlated with the Superpave fatigue parameter G*sin(δ) measured at the same temperature and at two levels of aging. CT Index measured at 0 °C was found to be highly correlated with blended binder m-value at the target PG temperature (26). In other words, if the blended binder is controlled to a certain m-value at a desired climate, low temperature cracking should likewise be controlled. This is a related concept to the ΔTc value for limiting age-induced

cracking in which strongly "m-controlled" binders are generally believed to be more prone to such distress. Findings generally support to concept of using blended binder properties to estimate low temperature performance.

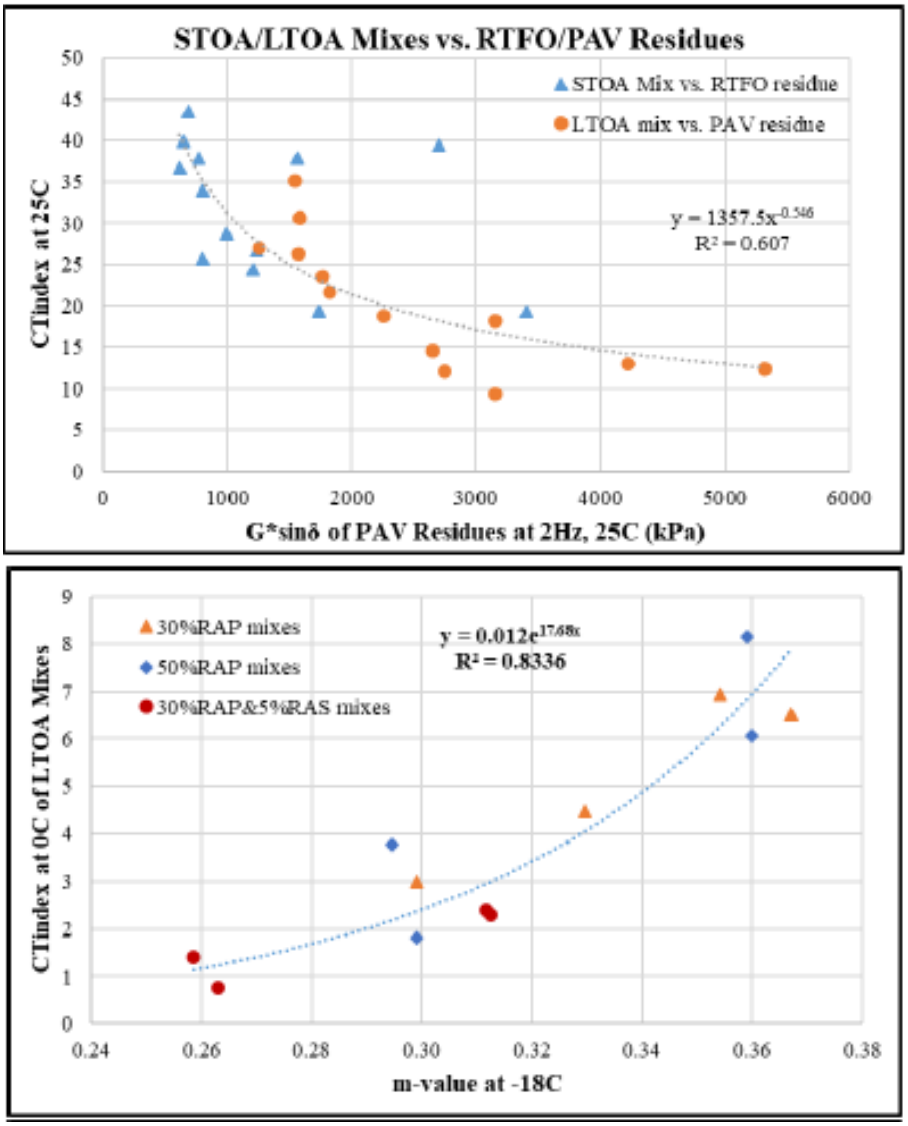


Figure 5. Use of blended binder properties at intermediate (top) and low (bottom) temperatures to estimate mixture performance. From (26)

Chapter 4 - *Work Plan, Sample Preparation and Testing Methods*

This chapter presents the work plan based on the project objectives and synthesis presented in this report. The chapter is divided into two subsections: materials and test methods.

Material Selection Plan

It is clear from the literature reviewed that the efficacy of a given recycling additive depends at least in part on the recycled binder properties (chemical and rheological). The selection of more than one distinct RAP sources for this project is therefore justified. It For this project two RAP sources were selected from two different climate regions within the NRRA member agency area. Both sources were sampled from field operations, one from a CIR project in South-Central Mankato (PG 58-28 or PG 58-34 region) and one from a CCPR project in Central Illinois (PG 64-22 region). Both sources also have associated mix design information available, which may aid the research team if longer term follow up for performance is warranted.

As described in previous chapters, recycling agents at the highest level can be broadly divided into two types: Bio-Based (inclusive of vegetable-based oils and tall oils) and Petroleum-Based (inclusive of aromatic and paraffinic oils). For the present study, a few factors are considered for the selection of the recycling agents to be tested:

- One representing bio-based RAs, and another representing Petroleum-based RAs.
- The specific additives have a demonstrated history of being commercially used in asphalt emulsion applications in pavement preservation applications. The reason for this factor is to bypass the necessity to perform screening and verification of the suitability of a given RA for use in CIR emulsion formulations, as emulsion quality and compatibility is outside the scope of the current project.

The research selected and sourced the oils for use in this study that meet this criteria and have years of field history in emulsion and rejuvenating pavement preservation applications. The commercial name and sourcing of the oils is not disclosed in the project.

Although several types of stabilizing additives exist in the CR industry, the focus of this study will be on asphalt emulsion. It has been reported in the literature that fundamental differences exist between the stabilizing mechanism of asphalt emulsion relative to foamed asphalt. Focusing on one stabilizing technology will allow the research team to gain a clearer understanding of the effects of RAs without biasing the results with the mechanism of stabilization. The use of active fillers (e.g., Portland Cement) are not included in the work plan. The final selection of materials is given in the Table 4.

Table 4. Selection of Raw Materials

Variable	Level	Description
RAP Source	2	Mankato RAP (CIR Project) Illinois RAP (CCPR Project)
Stabilizing Additive	1	Engineered Asphalt Emulsion
Recycling Additive	3	None – Engineered Emulsion only Bio-Based – Rejuvenating Emulsion-1 Petro-Based – Rejuvenating Emulsion-2

Sample Preparation and Mixture Test Methods

Sample Preparation and Curing

Since the inclusion of RAs into traditional stabilizing materials will inherently increase the unit cost of the stabilizing material, the cost-benefit of using such materials may rely on the ability of the RA to reduce the required dosage of stabilizing material via increased RAP binder activation. This is a concept similar to what was presented in Wegman & Sabouri (20), although in that study mixing temperature was the causal factor in reducing additive content. In the present study, the mixing temperature is fixed for the main factorial in order to isolate the effects of the RA, and a second, higher mixing temperature, will be used for a subset of mixes to test the impact of production temperature on binder activation as it also relates to RA usage.

Testing and curing methods chosen for this study represented a balance between practical, well-established methods and methods that will help to satisfy the objectives of this study (e.g., demonstrate the balanced mixture design concept). “Curing” in the parlance of CIR and CCPR industries refers to the setting of the emulsion. However, the researchers also anticipate that a second type of curing, that of the diffusion of the RA into the RAP, may also be relevant in the present study.

For the initial testing factorial, two curing intervals were selected based on consultation with the literature and a limited laboratory experiment. The first interval selected is referred to as the “Fully Cured” or “Full Cure” protocol, which was developed based on a summary of available literature: samples are placed in a 60 °C oven immediately after compaction for 48 hours, left to anneal at laboratory temperature for 18 ± 6 hours, then processed for testing.

A curing condition between that of freshly made mix and Fully Cured mix was desired, so a dummy mixture using representative project materials was produced, massed, and immediately placed into a 60 °C oven; curing temperature was held constant to eliminate this variable from consideration. The pill mass was recorded at several time intervals up to 140 hours. Following the 140 hour curing period, the final mass of the pill was within 0.01% of the predicted mass based on removing 100% of the water from the RAP and emulsion; it was therefore assumed that this was a fair approximation of the terminally cured mass of the sample.

The percent change in pill mass was plotted against time as shown in Figure 6. The 48 hour curing interval resulting in a mass loss of approximately 90% of the terminal value. To avoid the high rate of change portion of the graph and to address practicality concerns, an intermediate curing period of 24 hours is selected, resulting in an approximate mass loss of 70% of the terminal value. Both samples would also then undergo a further 18 ± 6 hours annealing at lab temperature, which may slightly increase mass loss further. In summary, by selecting 24 hours and 48 hours of curing at 60 °C, the effective curing as measured by mass loss represents approximately the 75% curing condition and the approximately 100% curing condition. It is likely these percentages change somewhat between RAP sources and additive levels but for the purposes of this research, the team believes this magnitude of difference is sufficient to demonstrate RA effects on curing, if present.

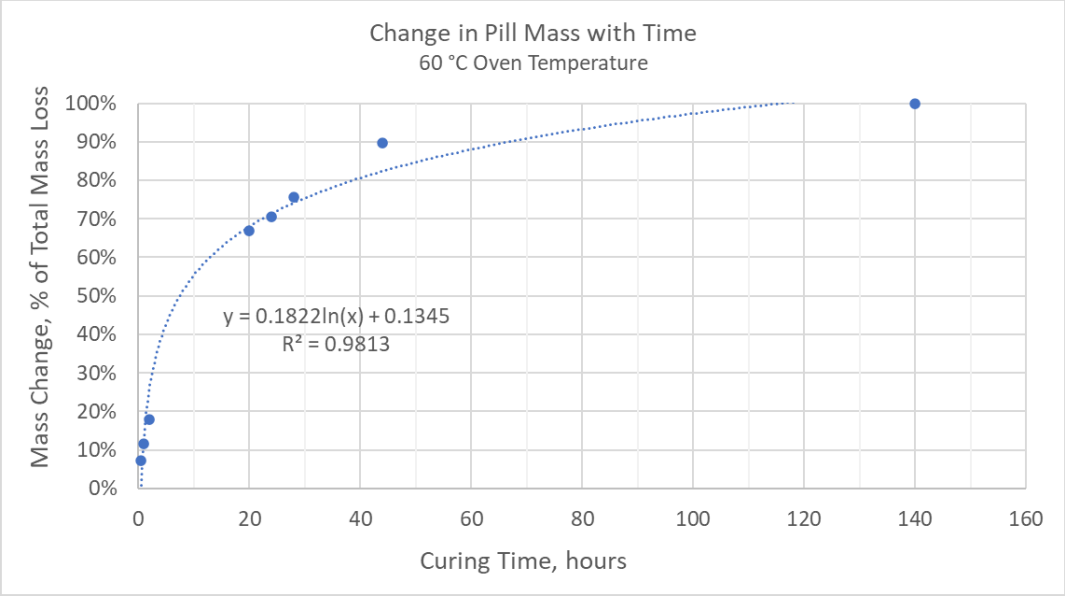


Figure 6 Mass loss curve for Illinois RAP prewetted to 2% water with 3% added engineered emulsion.

The two finalized curing protocols for the base factorial design are given in Table 5. Mixing and compaction protocols generally followed MnDOT standard procedure (Grading and Base Manual procedure), except the RAP is prewetted at least 24 hours prior to mixture production to facilitate better lab productivity and consistency. For all performance tests, samples are brought to testing temperature by placing in a forced draft oven for two hours at the testing temperature prior to the test.

Table 5 Finalized selected Curing Protocol

Curing Interval	Step 1	Step 2	Step 3	Step 4
Partial Cure	Mix* and Immediately Compact** Samples	60 °C Oven for 24 hours	Lab Temperature for 18 ± 6 hours	Process for Testing
Full Cure	Mix* and Immediately Compact** Samples	60 °C Oven for 48 hours	Lab Temperature for 18 ± 6 hours	Process for Testing

*RAP is prewetted to 2% moisture by bulk dry weight prior to mixing with emulsion.

**All samples compacted to 30 gyrations; sample mass is adjusted to result in a mixture height of 95 ± 5 mm following compaction.

Initially the research team envisioned utilizing the raveling test (ASTM D7196) to evaluate the effects of curing time on the samples. As a cost-share item the research team evaluated the sensitivity of that test to several important variables and determined that it is better utilized as a mix design verification test rather than a research tool for this study. Prior experience of the research team suggested that an indirect testing geometry may better suited to understanding cuing effects, so the IDEAL CT (ASTM D 8225) test was ultimately selected. Since utilization of a "Balanced Mix Design" concept is one of the goals of this study, the 6" Stability (ASTM D5581) was also utilized at each curing interval. Therefore, for the baseline factorial of mixtures, the Table 6 performance tests were selected.

Table 6. Performance Testing Framework

Service Temperature Range / Distress	Proposed Testing Method(s) - Mixture	
	Method	Curing Condition
High Temperature / Rutting	6" Stability (D5581) @ 40 °C	Partial Cure Full Cure
Intermediate Temperature / Cracking	IDEAL CT (D8225) @ 25 °C	Partial Cure Full Cure

Binder Test Methods

Binder Performance Grading

The initial performance grades (PG) for the virgin asphalt were measured following standard AASHTO M320 methodology using a TA DHR2 Dynamic Shear Rheometer (DSR), and a ATS BBR3 Bending Beam Rheometer (BBR). Multiple levels of aging was carried out on the asphalt using an ATS PAV-3 Pressure Aging Vessel (PAV), with each level consisting of 20hrs of conditioning at 2.1 MPa of pressurized air at 100°C.

Extraction and Recovery

The binder from RAP and loose mix samples was extracted using two step centrifuging using a standard explosion-proof centrifuge in accordance with ASTM D2172, followed by a high-speed centrifuge to remove the fines. Toluene was selected as the solvent of choice based on extensive prior experience of use for such applications.

The asphalt was recovered from the solution using an Abson recovery method following ASTM D1856, modified for ideal toluene removal conditioning using methodology described by ASTM D7906 for asphalt recovery from Toluene solutions.

Mastercurve Analysis

For the extracted binders, the intermediate and low temperature grade parameters, mastercurve, and Black space parameters were measured on a TA DHR2 Dynamic Shear Rheometer using a 4-mm spindle following ASTM D7175, and a standard 25mm spindle for the high temperature PG.

The Christensen-Anderson model (27), was used to fit the isotherms to a master curve using a minimization technique to determine the shift factors and derive the mastercurve shape parameters, as shown below in equation [1].

$$G^* = G_g^* \left(1 + \left(\frac{\omega}{\omega_c}\right)^{log2/R}\right)^{R/log2} \quad [1]$$

In which:

- G* is the complex modulus in Pa at frequency ω in Hz;
- G^*_g is the glassy modulus asymptote variable;
- ω_c is the cross-over frequency;
- R is the Rheological Index or "R-Value".

Iatroscan

The Iatroscan test is an increasingly popular method to measure the so-called SARA fractions of asphalt binders (Saturates, Aromatics, Resins, Asphaltenes). The Asphaltene content is defined as the N-Heptane insoluble fraction of the binder, following ASTM D3279. The fractionation of the maltene phase is then carried on the N-Heptane soluble portion using the Iatroscan, based on the principles of Thin Layer Chromatography. In this method the solution is "spotted" onto "chromarods" using a syringe, followed by series of conditionings and solvent elution steps that sequentially separate the different fractions based on solubility with the eluting solvent. The saturates are separated through an N-Pentane elution, followed by a Chloroform-Toluene blend (90:10 by volume) elution to separate the aromatic phase (sometime referred to as "Cyclics" as they also contain naphthenic ring structures). The portion of the original spotting that is un-eluted and remaining on the original spotting location on the rod is considered the polar aromatic (resin) fraction. The Iatroscan employs a Flame Ionization Detector (FID) to quantify the fractions separated by each elution to different points on chromarod.

Differential Scanning Calorimeter

A TA DSC250 Differential Scanning Calorimeter (DSC) was used to measure the glass transition range of aged and rejuvenated asphalt using a modulation method. The heating rates, rest rates, and increment periods were selected:

- Hold for 1 minute at 150°C to erase thermal history
- Cool from 150°C to -100°C at a rate of 20°C/min
- Hold for 5 minutes at -100°C to achieve equilibration
- Modulate from -100°C to 100°C in 2°C modulation periods at an average heating rate of 5°C/min.

The modulation analysis can be used for to separate the thermally reversible and irreversible response from the spectra. Analysis of the DSC data required the development of a protocol for reduction and smoothing of the initial dataset. Data below -88°C was discarded to remove the impact of test artefacts occurring at the start of the modulation step. The modulated step collected approximately 55,000 datapoints over the 200°C span. The data was first uniformly reduced to 1000 data points (~1 point per every 0.2°C). Smoothing was performed using the TA TRIOS software's "Least Square Moving Window" smoothing technique, for a 40 data point window.

Transition temperatures were determined as the local maxima on the derivative of the heat capacity spectra. The "total" glass transition for the entire range was defined as the temperature corresponding to the midpoint of the enthalpy change between the tangents ("ΔH mid-point Tg"). The "transition region" was defined by the temperatures at which reversible heat flow curve approximately achieves its linearity at either tangent. It is conceivable that valid methods other than that utilized in this study may be devised to define the limits in a consistent manner.

Chapter 5 – Asphalt, *RAP, and Emulsion Characterization*

Reclaimed Asphalt Pavement (RAP)

It is clear from the literature that the efficacy of a given recycling additive depends at least in part on the recycled binder properties (chemical and rheological). The selection of more than one distinct RAP source for this project was therefore deemed justified. With the help of TAP members, the research team located and sampled two sources of RAP. Both sources were sampled from field operations, one from a CIR project in South-Central Mankato (PG 58-28 or PG 58-34 region) and one from a CCPR project in Central Illinois (PG 64-22 region). The associated mix design information for both RAP sources are also available as reference. The material was solvent extracted and graded using the Dynamic Shear Rheometer 4-mm spindle test method. Gradations were determined per AASHTO R30 and shown in Figure 7 along with black rock gradations.

Figure 8 and Figure 9 show the mastercurves derived from each extracted RAP binder. Table 7 shows the calculated binder properties corresponding to these materials. It can be seen the IL RAP has a lower binder content, stiffer low and intermediate temperature PG. Looking at the overall rheological and Black Space properties the IL RAP can be seen to have slightly less desirable properties across the board. In absence of specific threshold values for most rheological measures of quality at this time, one may simply put that the IL RAP shows slightly "lower quality" than the MN RAP. This trend is not unexpected, as the IL RAP is sourced from a predominantly PG-64-22 region, compared to that of the MN RAP that likely came from a pavement originally using a PG58-28 binder. The impact of such differences in binder "quality", whether the RAP, the emulsion base, or the rejuvenated blend, will be further explored in the context of CIR mix performance measures throughout this project.

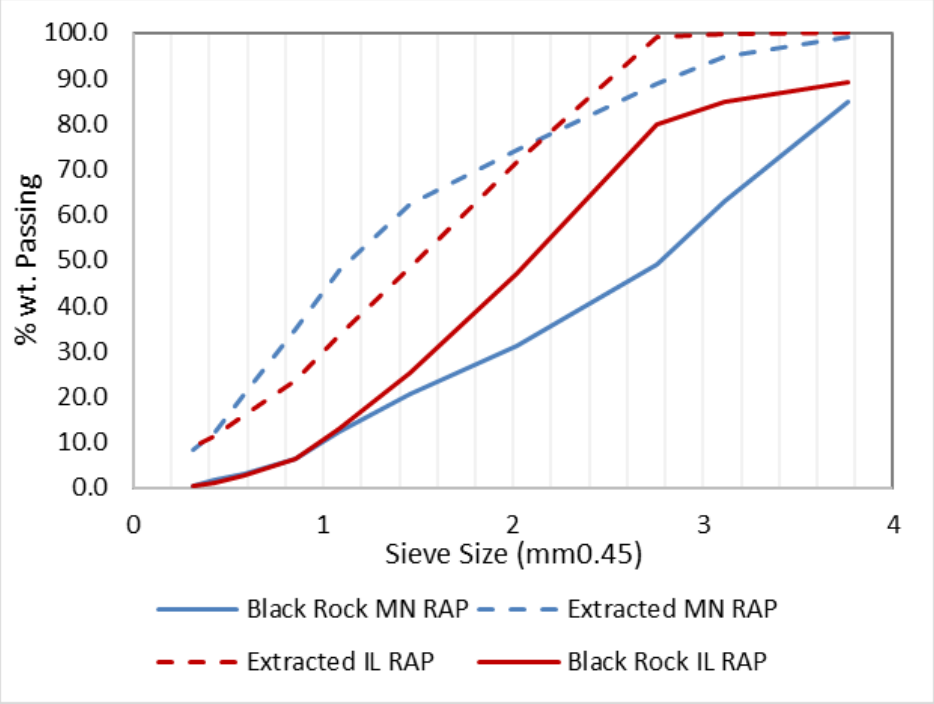


Figure 7 RAP gradation curves

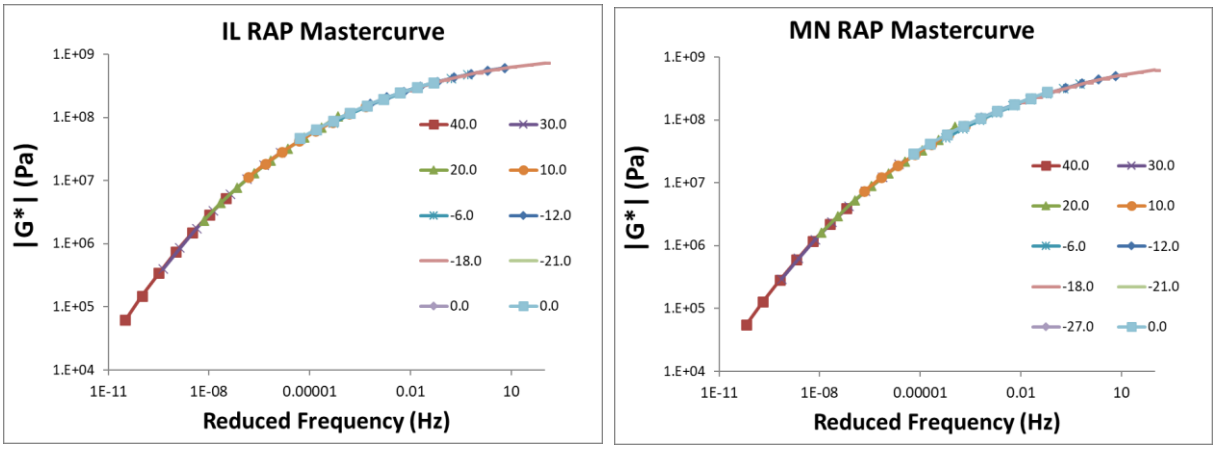


Figure 8 Extracted RAP binder stiffness mastercurves (Although the graphs seem qualitatively similar, they are numerically different as shown by analysis results in Table 7)

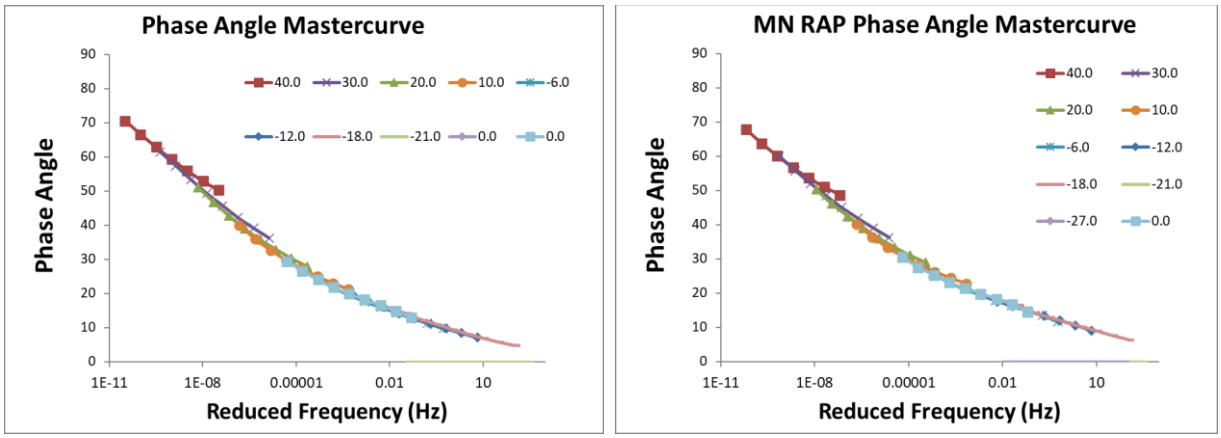


Figure 9 Extracted RAP binder phase angle mastercurves (Although the graphs seem qualitatively similar, they are numerically different as shown by analysis results in Table 7)

Table 7 Measured RAP binder properties and mastercurve analysis parameters

Property	IL RAP	MN RAP
Moisture Content (%)	1.09%	1.81%
Std.+ Hi-speed Centrifuge Binder Content (%)	4.55%	6.42%
Standard PG	**PG 82-10**	**PG 82-16**
High Temperature PG (As extracted)	82.5	84.3
Low Temperature PG (T_S)	-19.8	-24.4
Low Temperature PG (T_m)	-15.4	-17.7
ΔTc ($T_S - T_m$)	-4.4	-6.6
Intermediate Temperature PG	29.9	26.5
Glover-Rowe |G*| (Log kPa)	3.5	3.3
Glover-Rowe Phase Angle (°)	47.8	47.1
Glover-Rowe Parameter (kPa)	1974	1290
Temperature at δ=30°, 1.59 Hz (°C)	13.8	12.6

Base Binders

Two base binders were selected for emulsion production in this project, a PG58-28, representative of common practice in northern NRRA states, and a PG64-22 representative of the upper Midwest and to be used to produce the rejuvenating emulsions. Both binders were sourced from the upper Midwest. Table 8 shows the performance grading results for these binders. It can be seen that for either binder high temperature PG is on the higher side of the grade. The PG58-28 has a broader Useful Temperature Interval (UTI) and a positive ΔT_c, marking it as a potentially somewhat higher quality binder, however both binders comfortably meet all PG requirements.

Table 8 Performance Grading results for project base binders

Binder Description	UTI (HT – LT PG)	High Temp PG (Unaged)	High Temp PG (RTFO Aged)	RTFO	Low Temp PG (Stiffness)	Low Temp PG (m Value)	ΔT_c
	°C	°C	°C	Mass Δ	°C	°C	
PG 58-28	91.7	62.5	63.5	-0.484%	-29.2	-29.4	0.2
PG 64-22	89.9	67.0	68.3	-0.013%	-26.2	-22.9	-3.3

Binder Description	Original High Temp. PG (|G*|/sinδ, kPa)			RTFO High Temp. PG (|G*|/sinδ, kPa)			Low Temperature PG (S(60))			Low Temperature PG (m(60))		
	58	64	70	58	64	70	-12	-18	-24	-12	-18	-24
PG 58-28	1.76	0.82	0.41	4.58	2.07	0.98		261.1	517.0		0.316	0.249
PG 64-22	3.30	1.46	0.69	8.85	3.84	1.75	182.6	368.5		0.308	0.255	

Recycling Agents

At the highest level, recycling agents can be broadly divided into two types: Bio-Based (inclusive of vegetable-based oils and tall oils) and Petroleum-Based (inclusive of aromatic and paraffinic oils). For the present study the research selected and sourced one of each of the two broad categories of oils. The selected RAs had years of field history in emulsion and rejuvenating pavement preservation applications, eliminating the need for determining basic suitability for emulsion applications. The commercial name and sourcing of the oils is not disclosed in the project. The ASTM D4552-20 (10) properties of the two recycling agents are shown in Table 9.

Table 9 RA Properties in accordance to ASTM D4552-20

Property	Bio' Oil (B1)	Petro' Oil (P1)
Rotational Viscosity at 60°C, cP	29.3	143.5
Specific Gravity at 25°C, g/ml	0.93	0.98
ASTM D3279 Asphaltene Content, %wt.	0.2%	0.2%
Iatroscan Resin Content, %wt.	56.1%	16.9%
Iatroscan Cyclics Content, %wt.	43%	63%
Iatroscan Saturates Content, %wt.	0.6%	19.8%
RTFO Aged Viscosity at 60°C, cP	30.6	195
RTFO Aging Index (Viscosity Ratio)	1.05	1.36
RTFO Mass Loss, %wt.	0.38%	4.57%

The RAs were dosed into the PG64-22. This allows for RA dosing to be set at a point that would create similar grade properties as that of the PG58-28 binder, therefore allowing to determine what impacts beyond overall asphalt softness are imparted to cold mixes through the use of RAs. This will be discussed in the later sections. RA dosage were selected based PG properties of the modified binder,

generally following guidance proposed in NCHRP 09-58 (25). The test results are shown in Table 10 and Table 11, while the dosage optimization process can be seen in Table 10.

The following overall dosing approach was taken: The lower RA dosages were determined as to approximately match the HT PG of the PG58-28 binder (2.4% for B1, 4% for P1). The high dosage for B1 was matched to the low dosage of P1 (4%) in order to have an equal dosage comparison. And finally, the high dosage for P1 (6.7%) was chosen to have an equal HT PG to that of the high dosage of B1. These equivalences are shown in Figure 10.

Table 10 Performance Grading results for base binder modified using different RA dosages

Binder Description	RA Content (%)	High Temp PG (Unaged) °C	High Temp PG (RTFO Aged) °C	RTFO Mass Δ	Low Temp PG (Stiffness) °C	Low Temp PG (m Value) °C	ΔT_c
PG64 + 2.5% B1	2.5	62.8	63.6	0.011%	-29.6	-27.4	-2.2
PG64 + 4% B1	4	60.6	61.2	0.006%	-32.2	-30.3	-1.8
PG64 + 5% P1	5	62.2	63.2	-0.357%	-27.9	-26.8	-1.1
PG64 + 6.7% P1	6.7	61.9	62.9	-0.442%	-28.5	-27.6	-0.9
PG64 + 8% P1	8	59.4	60.7	-0.544%	-29.1	-28.3	-0.8

Table 11 Performance Grading parameters for base binder modified using different RA dosages

Binder Description	Original |G*|/sinδ, kPa			RTFO |G*|/sinδ, kPa			S(60), MPa			m-value (-)		
	52	58	64	52	58	64	-12	-18	-24	-12	-18	-24
PG64 + 2.5% B1		1.87	0.86		4.70	2.09	106.6	241.6		0.353	0.294	
PG64 + 4% B1	3.16	1.38	0.65	7.77	3.34	1.52		181.9	374.4		0.320	0.269
PG64 + 5% P1	4.01	1.73	0.79	10.62	4.51	1.97	135.0	305.3		0.353	0.287	
PG64 + 6.7% P1	3.89	1.65	0.76	10.20	4.30	1.90	120.2	278.5		0.360	0.296	
PG64 + 8% P1	2.72	1.19	0.56	7.43	3.16	1.41		264.7	516.0		0.303	0.246

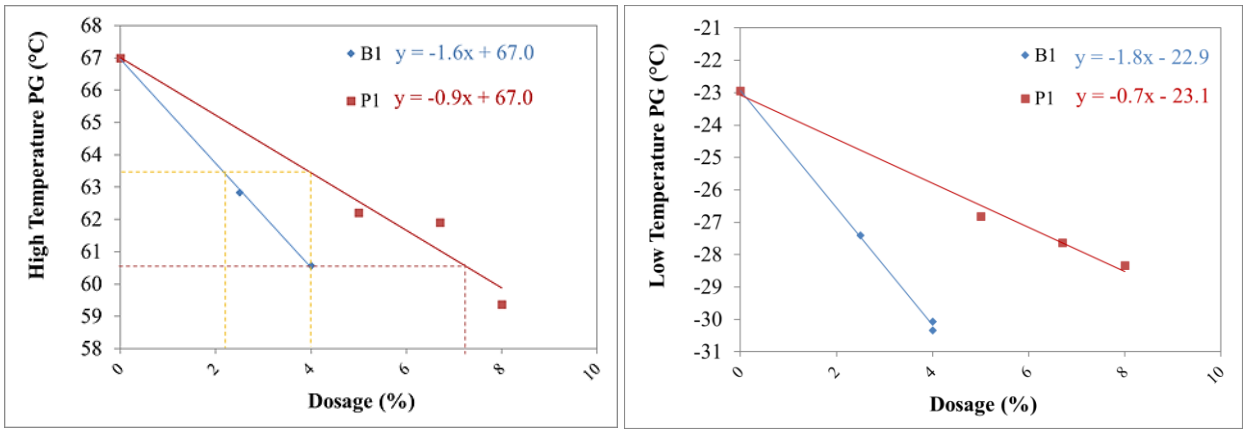


Figure 10 PG of base binders containing different dosages of RA. Study dosages determined based on predicted equal HT PG.

Emulsions

As described in the previous sections, the RAs were incorporated by blending with the PG64-22 base binder prior to emulsification, as this is most common and practical in the rejuvenating emulsion industry. The control was made using the PG58-28 base binder. The emulsions were produced in the lab using a Charlotte G5 emulsion mill and a cationic emulsifier commonly used for production of engineered CIR

emulsions at a target residue content of about 65%. Table 12 shows the results of the quality control testing performed for the resulting emulsions, in accordance with common practice for CIR emulsions.

Table 12 Quality control testing results for study CIR emulsions

PROPERTY		AASHTO / ASTM TEST METHOD	RESULTS
Emulsified Asphalt			
Saybolt Furol Viscosity	25.0 °C	T59 / D7496	29.8 seconds
Storage Stability, 24 hour	-----	T59 / D6930	0.2 %
Sieve	-----	T59 / D6933	0.00 %
Distillation, % Residue, 20 minutes	175 °C	T59 / D6997	65.1 %
Distillation Residue			
Penetration, 100 g, 5 seconds	25.0 °C	T49 / D5	104 dmm

(a) PG58-28 control

PROPERTY		AASHTO / ASTM TEST METHOD	RESULTS
Emulsified Asphalt			
Saybolt Furol Viscosity	25.0 °C	T59 / D7496	32.1 seconds
Storage Stability, 24 hour	-----	T59 / D6930	0.1 %
Sieve	-----	T59 / D6933	0.00 %
Distillation, % Residue, 20 minutes	175 °C	T59 / D6997	65.3 %
Distillation Residue			
Penetration, 100 g, 5 seconds	25.0 °C	T49 / D5	97 dmm

(b) PG64-22 + 2.4%B1

PROPERTY		AASHTO / ASTM TEST METHOD	RESULTS
Emulsified Asphalt			
Saybolt Furol Viscosity	25.0 °C	T59 / D7496	27.1 seconds
Storage Stability, 24 hour	-----	T59 / D6930	0.0 %
Sieve	-----	T59 / D6933	0.00 %
Distillation, % Residue, 20 minutes	175 °C	T59 / D6997	64.4 %
Distillation Residue			
Penetration, 100 g, 5 seconds	25.0 °C	T49 / D5	132 dmm

(c) PG64-22 + 4%B1

PROPERTY		AASHTO / ASTM TEST METHOD	RESULTS
Emulsified Asphalt			
Saybolt Furol Viscosity	25.0 °C	T59 / D7496	34.5 seconds
Storage Stability, 24 hour	-----	T59 / D6930	0.1 %
Sieve	-----	T59 / D6933	0.01 %
Distillation, % Residue, 20 minutes	175 °C	T59 / D6997	65.4 %
Distillation Residue			
Penetration, 100 g, 5 seconds	25.0 °C	T49 / D5	89 dmm

(d) PG64-22 + 4%P1

PROPERTY		AASHTO / ASTM TEST METHOD	RESULTS
Emulsified Asphalt			
Saybolt Furol Viscosity	25.0 °C	T59 / D7496	29.3 seconds
Storage Stability, 24 hour	-----	T59 / D6930	0.0 %
Sieve	-----	T59 / D6933	0.00 %
Distillation, % Residue, 20 minutes	175 °C	T59 / D6997	64.8 %
Distillation Residue			
Penetration, 100 g, 5 seconds	25.0 °C	T49 / D5	104 dmm

(e) PG64-22 + 6.7%P1

Comparing the residue distillate penetration for the RA blends to that of control PG58-28 it is apparent that matching the HT PG did not result in matching penetrations. This is not unexpected due to the different base binders and the different RA modifications, however, further in the study, mixture performance results will be assessed against the various distillate rheological properties in order to determine which parameter is better suited as a dosage determinator for design purposes.

Analytical Evaluations

The binders with and without RAs and at various aging conditions were further subjected to various analytical testing procedures. The purpose of this testing was to create a fundamental baseline of knowledge about the properties of the raw materials in order to use towards interpretation of mixture performance testing and evaluations performed later in the project.

The tests conducted were the Differential Scanning Calorimeter (DSC) and SARA fractionation using the Iatroscan, as described in previous sections. Additional testing may be considered and added based on observed performance. These methods are not anticipated to be part of any specification proposal for the design and use of rejuvenated CIR and are mainly for research and data interpretation in this study in order to aid in the creation of meaningful practical design guidance.

The SARA fractionation results for the neat binders at aged and unaged conditions are also presented in Table 13 and Figure 11. The results show that as the binders aged the asphaltene content increased, while the “cyclics” and “resins” significantly decreased. The “saturate” phases also showed a slight reduction.

It is also noteworthy that for the unaged neat binders the SARA fractionations were rather similar despite the difference in performance grade. This is an interesting demonstration of how SARA fractionation is a useful tool to assess general asphalt trends, it is too coarse to be accurately predictive of binder physical and rheological properties without the context of additional data. To this end the researchers find the DSC test to be a good complimentary analysis method to along with SARA provide a clearer picture of binder physical-chemical properties.

Table 13 Iatroscan SARA fractionation Results for base binders before and after aging

Sample	Saturates	Cyclics	Resins	Asphaltene
PG64-22-Unaged	8.5%	51.8%	23.8%	15.8%
PG58-28-Unaged	6.9%	53.4%	23.5%	16.1%
PG64-22-RTFO+PAV	6.8%	37.1%	30.3%	25.8%
PG58-28-RTFO+PAV	5.4%	39.4%	31.8%	23.3%

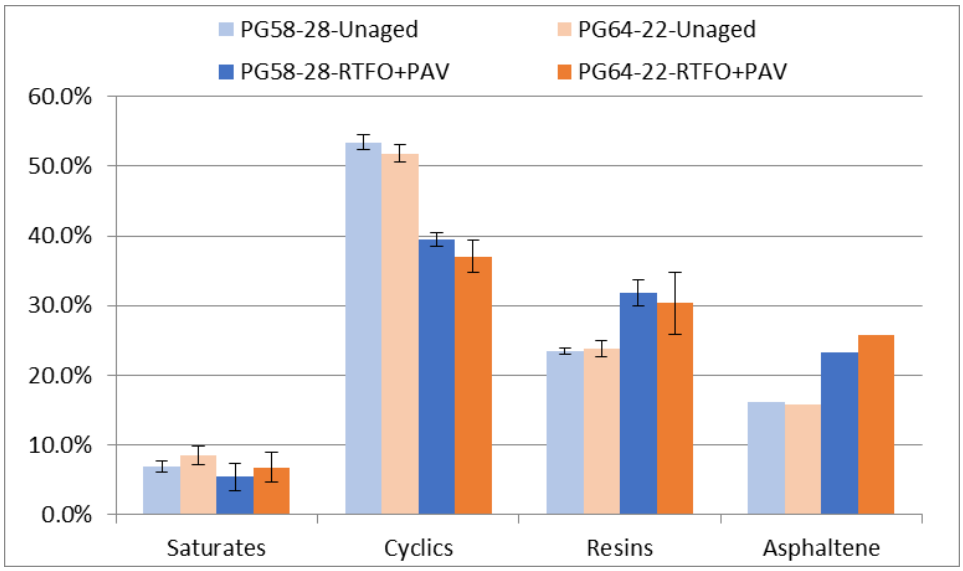


Figure 11 Comparison of SARA fractions of the base binders (error bars represent standard deviation of 4-5 replicate rods)

Figure 12 shows the definition of terms used for analysis of the DSC curves. The "half height" and "inflection" glass transition temperatures were defined through the TRIOS software analysis package, while the Tgα, Tgβ, and Tgγ values were defined through the deconvolution analysis of the Heat Flow derivative curve, as shown more clearly in Figure 13. The approximate locations of these points are also shown in Figure 12. The results are presented in tabular form in Table 14.

The results show that although the actual locations of the glass transition temperatures are relatively similar for both binders, the relative "strength" of the transitions, as characterized by the area under the deconvoluted curves of the heat flow derivative, are rather different. With the stiffer binder having a more prominent β transition. The growth of the β is also noted with aging.

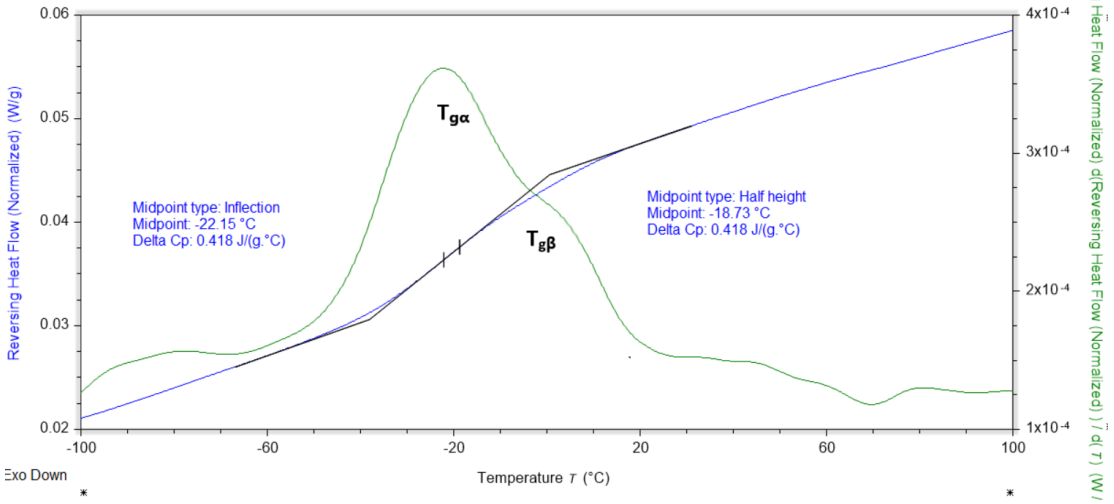


Figure 12 Definition of terms used for analysis of DSC curves for the Example of the RTFO aged PG64-22 binder

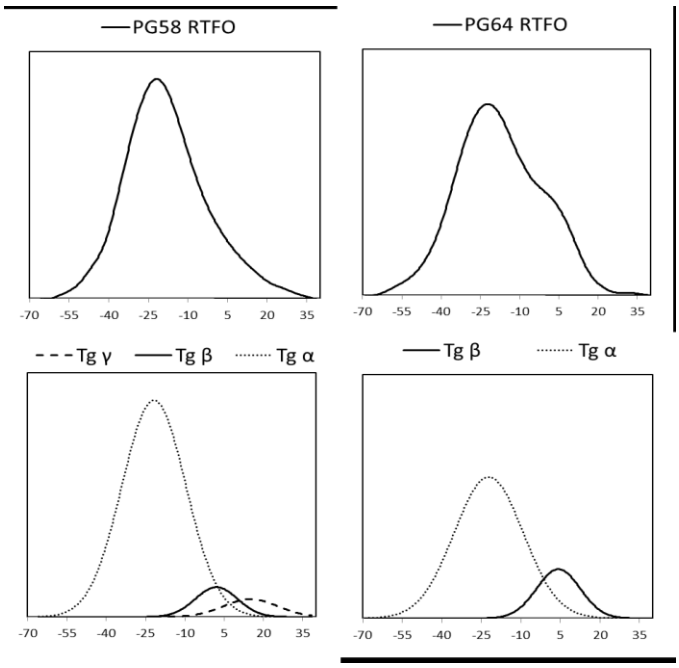


Figure 13 Comparison of Deconvolution curves between the tested PG58 and PG64 binders

Table 14 DSC Analysis Results for base binders at various aging conditions

Binder Description	$T_{g\ Half\ cp}$ °C	$T_{g\ Inflection}$ °C	$T_{g\alpha}$ °C	$T_{g\beta}$ °C	$T_{g\gamma}$ °C
PG64 Unaged	-18	-22.46	-22.15	4.18	-
PG64 RTFO	-18.73	-22.15	-22.21	4.26	-
PG64 RTFO + PAV	-19.97	-23.99	-24.01	2.96	-
PG58 Unaged	-21.68	-23.35	-22.77	1.88	13.23
PG58 RTFO	-19.44	-21.96	-21.85	2.02	15.07
PG58 RTFO + PAV	-18.96	-22.2	-21.74	5.80	24.00

The analytical results from the base binder comparisons show that both the PG58-28 (control binder) and the PG64-22 (base for RA binders) have similar chemical fractionation makeup, while having somewhat different long term oxidative aging mechanisms. With the PG58-28, oxidative aging resulted in more growth in the “Resin” fraction, as apposed to the “Asphaltene” fraction. This manifested itself as widening of the glass transition region in the thermal analysis spectra, as the tertiary transition shifted to higher temperatures with aging. With the PG64-22, aging had a more pronounced impact on the increase of the “Asphaltene” fraction. The asphaltene fraction itself does not exhibit a glass transition, although it is postulated that if certain associations occur, it will have an impact on the transition behavior of other fractions. As a result the Tg evolution of this binder with aging is less straight forward.

Overall it is concluded that within the context of cold recycling in which conditioning is focused on the early life properties, both binders should exhibit relatively similar compatibility behaviors, therefor limiting the potential confounding effects of base binder differences when comparing the performance of the mixes containing the control PG58-28 base binder to the cold recycling mixes containing the RA-treated PG 64-22, as will be further explored in later chapters.

Chapter 6 – Mix Performance Testing and Analysis

Mix Design Verification and Evaluation of Mixture Density

The mix designs for each RAP source were supplied at the time of material collection by the consulting laboratories that conducted the designs. For each RAP source the Theoretical Mixture Maximum Specific Gravity (Gmm) was measured at 3% added emulsion for each the Bio-High and Petro-High emulsions following AASHTO T209 with the supplemental dry back procedure; it is believed that if the Gmm values for these blends substantially compare to the mix design values it can be assumed that (a) the RAP material delivered and material preparation procedure are representative of the design materials, and (b) the inclusion of RAs does not influence the ability to accurately measure Gmm. Results are shown in Table 15.

Table 15. Comparison of Mix Design Gmm with RA Emulsion Gmm at same Emulsion Content (3%)

Blend:	**Mankato**	**Illinois**
Mix Design	2.333	2.405
Bio-High, Avg of 2	2.331	2.400
Petro-High, Avg of 2	2.318	2.400

Although some differences are noted for the Mankato Petro-High blend, it is believed that the accuracy of the other values to the mix design suggest this is most likely a sampling variation. Based on these results, it is concluded the materials delivered are representative of the mix design, and that the preparation procedure substantially matches the mix design. For the following analysis, the mix design Gmm was used at each emulsion content to calculate the Gmm for the current project by linear interpolation.

The average Mixture Bulk Specific Gravity (Gmb) for each blend was measured following AASHTO T166, Method A modified to utilize a one minute soaking period as opposed to four minutes (following MnDOT CIR design procedure protocol). All samples were measured following the long term curing protocol outlined earlier. The Air Voids (Va) for each sample blend was then calculated. In general the Va shown below is calculated from an average of five Gmb specimens (two for stability, three for IDEAL CT). Voids for samples prepared at lab temperature are in Figure 14.

Samples for each RAP source and emulsion formulation show a logical trend of decreasing Va (higher density) with increasing emulsion content. It does not appear the inclusion of RAs categorically increases or decreases density for a given emulsion content; in other words, use of RAs in this manner does not substantially affect the mechanisms of compaction and no adjustment to lab procedure or field compaction protocols is expected. In general, the samples exhibit Va levels comparable to values cited in the literature for CIR mixtures and for the mix designs associated with these RAP sources. The mix designs for these RAP sources were completed in two different laboratories independent of this project, but values of air voids from those mix designs are included in Figure 14 for reference.

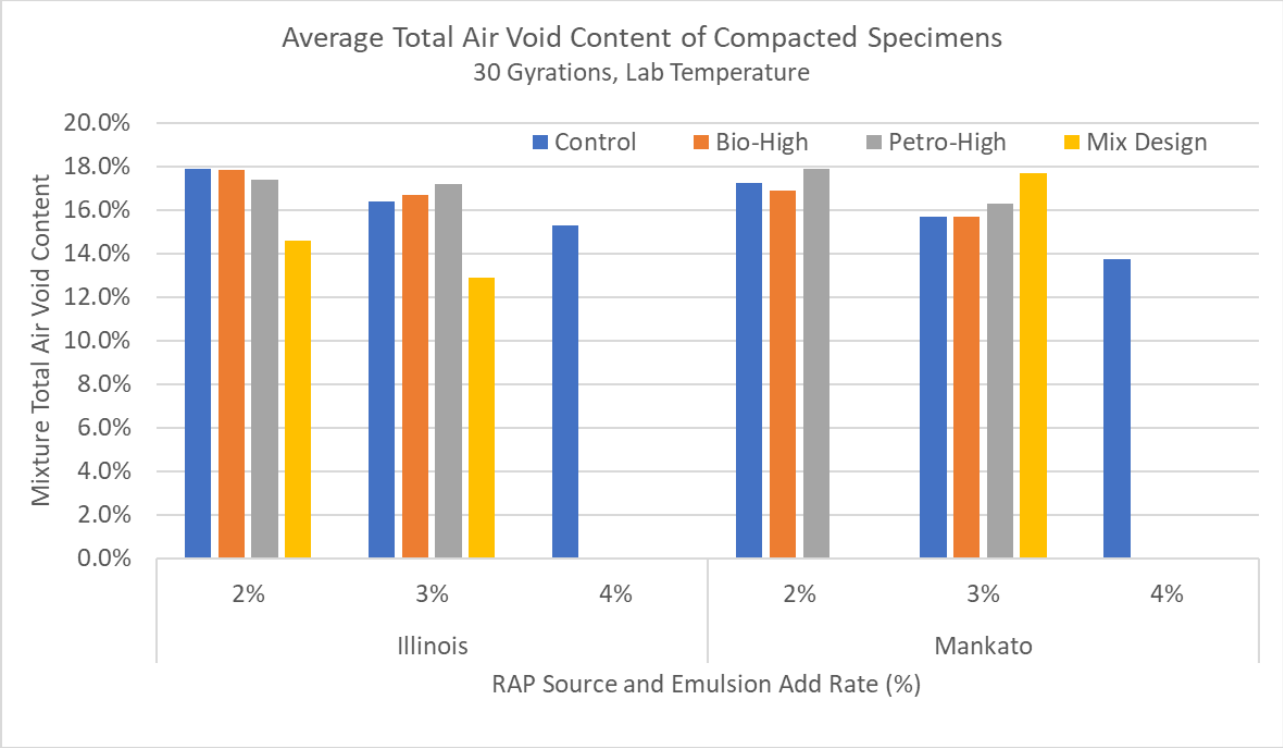
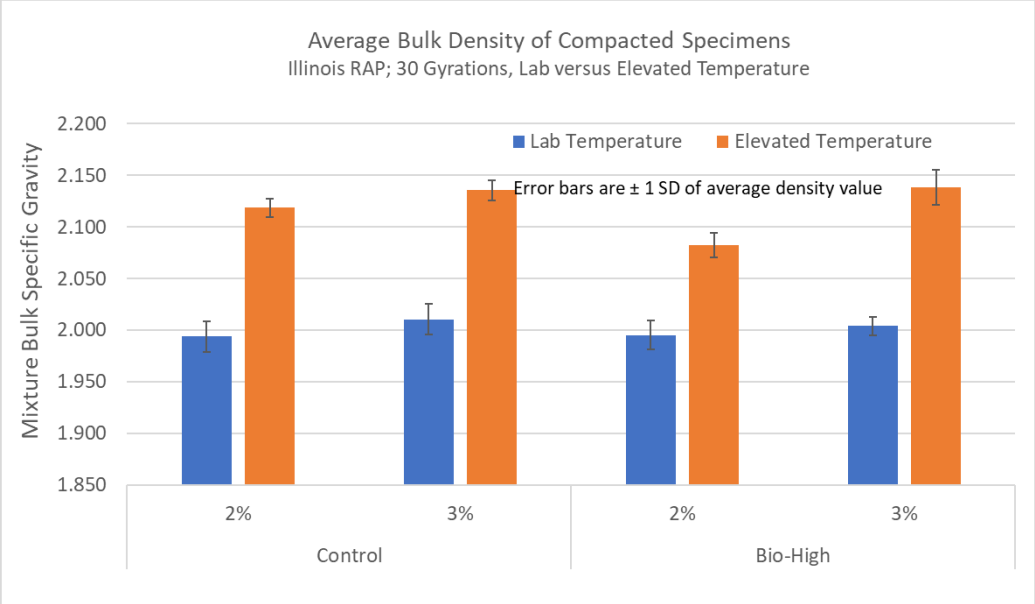


Figure 14. Mixture Air Voids for Lab Temperature Samples

A subset of mixtures was produced at elevated temperature, which for this study is defined as 110 °F mixing and compaction temperature. The relative Gmb and air voids for some of these mixtures is shown in Figure 15. The elevated temperature resulted in mixtures achieving between about 5%-8% lower air voids relative to the same blend produced at lab temperature. This change in Va is greater than the change in Va between 2% and 4% emulsion for both RAP sources shown above (about 3% Va decrease for each source). This is not unexpected and confirms findings presented by Wegman (2019) that mixing and compaction temperature is critical to recycled mixtures' performance. This finding further illustrates the importance of lab protocol for production of these samples. Standard practice is to produce specimens at "lab temperature", but if field conditions are expected to vary significantly from lab temperature, this may ultimately produce a non-representative design. The impact of temperature on performance for this study will be further presented below.



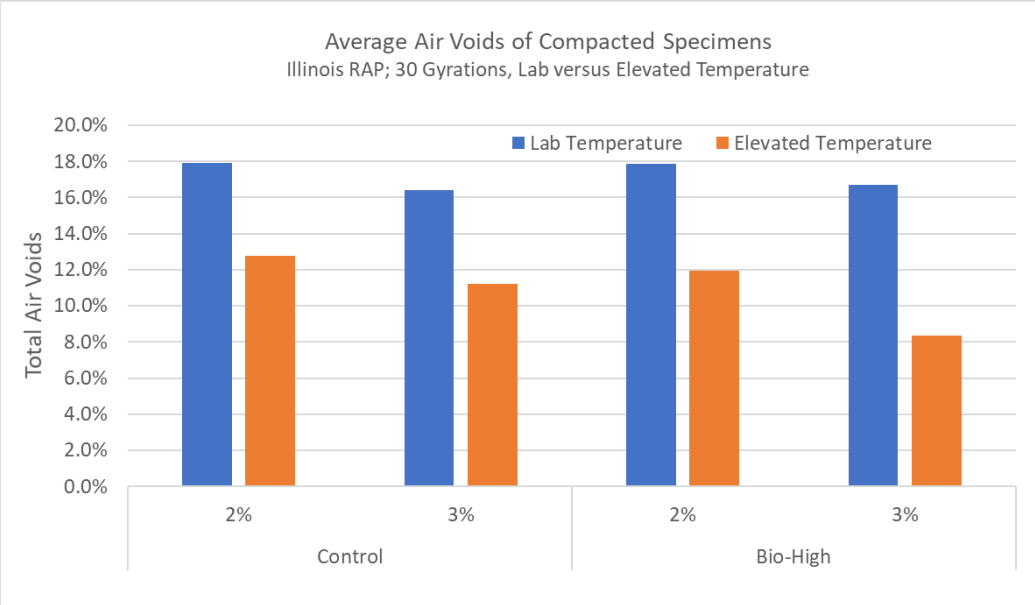


Figure 15. Effect of Mixing and Compaction Temperature on mixing bulk density (top) and air voids (bottom).

Mixture Stability at 40 °C

Effect of RAs on Long Term Cured Mixture Stability

Stability following the full-cure protocol was conducted on two samples per blend at each emulsion content. The measured stability is corrected to 95 mm for all samples following the procedure in ASTM D5581. Results for the Control, Bio-High, and Petro-High blends are shown in Figure 16 . Error bars for all bar charts shown in the stability section represent the range in stability values observed (the ASTM d2S value) for the given blend combination. The first observation made is that commonly cited minimum limit of 2,500 lb for CIR stability at 40 °C (corrected for 150 mm specimens) is comfortably met for all blends. It does not appear the inclusion of RAs at the levels used in this study results in a collapse of stability, excessive mixture softening, or similar phenomenon. Logical trends of decreasing stability with increasing

emulsion content are noted, with some differences between RAP source and emulsion blend. In general, when accounting for testing variability many of the blend differences do not appear to be practically significant.

The implications of this data are that current minimum limits on stability do not need to be modified to account for RAs. Although it appears stability may not be a robust indicator of effective dosage, stability should be checked in a balanced design approach. Use of stability as a dosage determination test is explored in a subsequent section.

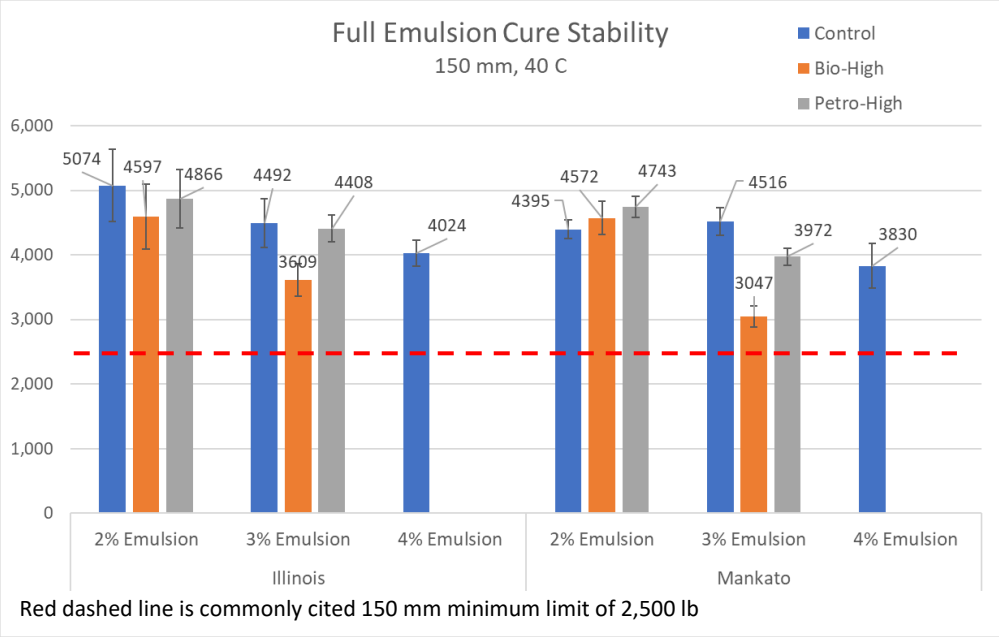


Figure 16. Full emulsion cured stability at 40 °C

Effect of Curing Condition on Stability

Although it does not appear RAs create long term stability-related concerns, a check was conducted on short term cured specimens to ensure early life stability is not negatively impacted. Results shown below confirm that mixtures containing RAs do not exhibit significantly different short term stability properties relative to control materials. It appears the mechanism of strength (as measured by stability) gain for RA emulsions is at least similar to control materials (i.e., RAs do not appear to retard the curing of cold recycled mixtures). It is therefore assumed that the general construction process would not need to be modified to allow the effective use of RA materials.

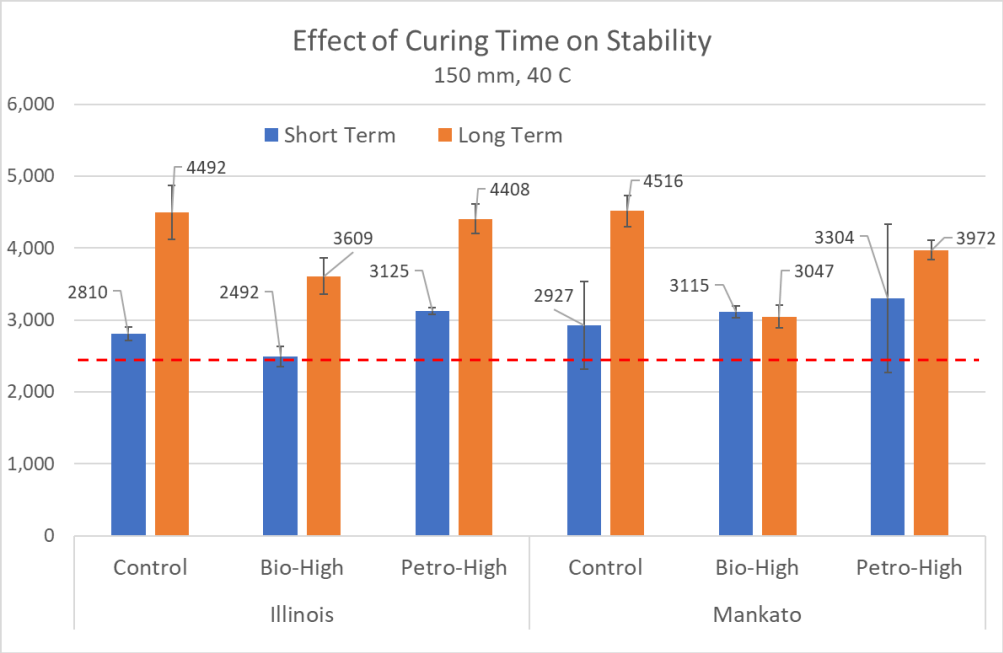


Figure 17. Effect of curing level on stability

Effect of RA Dosage on Stability

Blends utilizing the two levels of RAs at two emulsion contents were tested to determine the sensitivity of the stability test to RA dosage (residue properties). Results shown in Figure 18 indicate that although a logical trend is found for all blends with respect to emulsion content, it does not appear stability is practically sensitive to RA dosage within the dosage levels used in this study. This finding further confirms that stability is most likely not an effective tool to determine RA dosage, but is likely better suited to check against over-dosing of an RA in a balanced design framework.

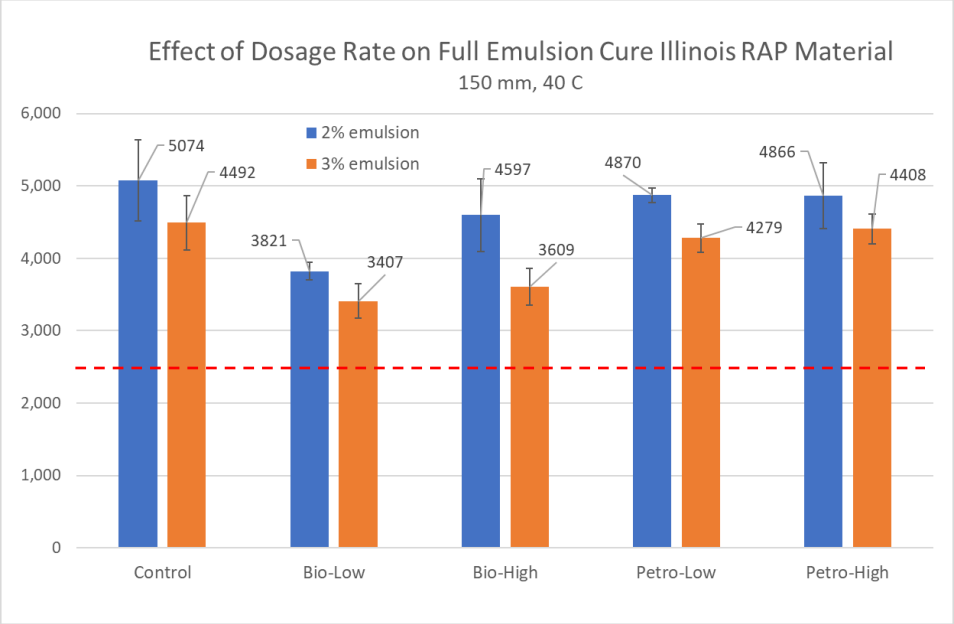


Figure 18. Effect of RA dosage on mixture stability

Effect of Mixing and Compaction Temperature on Stability

The effect of mixing and compaction temperature on a subset of Illinois RAP long term cured samples is shown in Figure 19. The "Lab Temp" specimens are the same as shown above, whereas the "110 °F" specimens are those mixed and compacted at 110 °F. Between a 24% and 45% increase in stability was measured for the samples tested in this project. This finding is consistent with Wegman (2019), although the relative increases noted in the Wegman study for a given emulsion content were closer to 60%. That could be an artifact of sample size differences (150 mm diameter vs. 100 mm diameter in Wegman study).

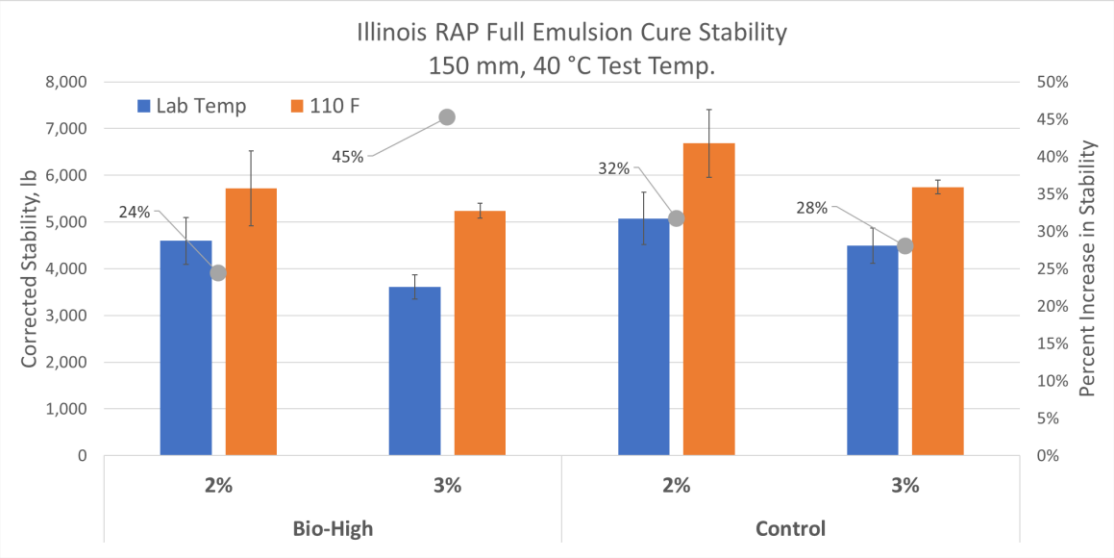


Figure 19. Effect of mixing and compaction temperature on mixture stability

Interestingly, the most readily noted change due to increasing mixing and compaction temperature is the increase in sample density at a given emulsion content. It seems logical that increasing mixture density will result in greater stability as more aggregate-to-aggregate contact is created. However, when interpreting Figure 14, which shows that increasing emulsion content can similarly increase density, a corresponding decrease in stability is observed (Figure 20). This finding suggests that changing the mixing and compaction temperature must contribute more than density alone. Two hypotheses to explain this behavior are increased binder activation (Wegman) and increased aggregate reorientation/mobility at higher temperature. A higher residue content produces a specimen with lower air voids, but also a specimen with increased binder film, which may contribute a lubricating effect, thereby reducing stability.

When the elevated mixing and compaction temperature sample subset is plotted along with the lab temperature dataset (Figure 20), both sample subsets show a similar sensitivity to air voids (slope of best fit). This suggests that the mechanism controlling stability are similar at each production temperature, but also further illustrates the importance of simulating typical field conditions in the laboratory for these mixtures sine the magnitude of stability is changed.

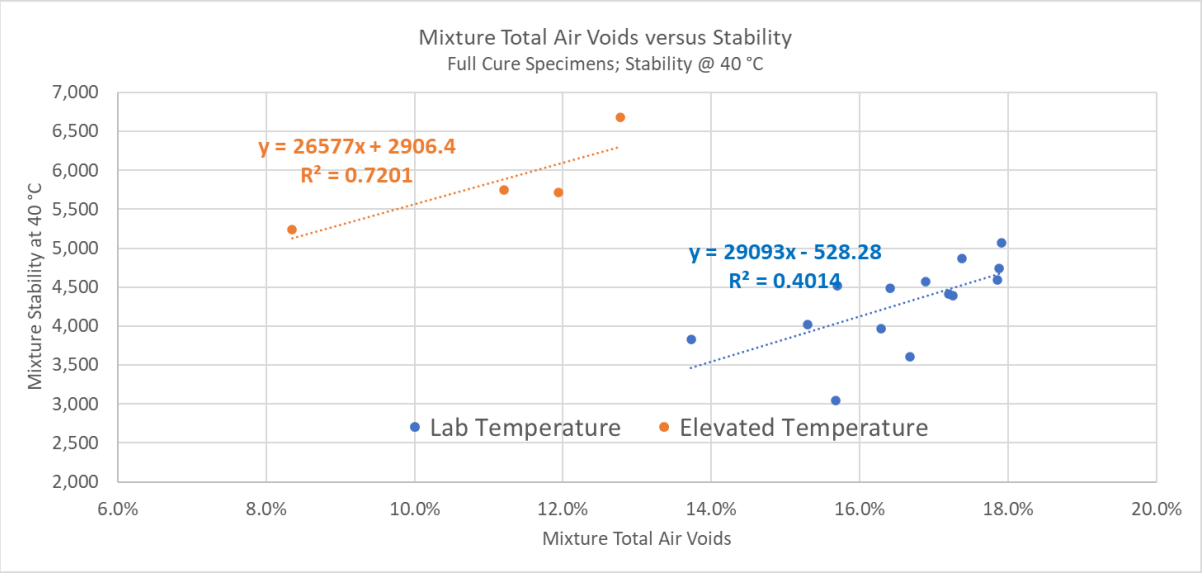


Figure 20. Sensitivity of stability at two mixing and compaction temperatures to air void content

Summary of Findings Related to Mixture Stability

The following is a summary of the major findings related to mixture density and stability:

- The use of RAs in this study does not appear to influence the compaction behavior of cold recycled mixtures, and compacted air void level is similar to control mixtures;
    - Field operations do not need to be adjusted when using RAs in the manner prescribed in this study;
- RAs do not appear to negatively impact mixture stability or the mechanism of strength gain with respect to stability during curing for the materials tested in this study;
    - Current mix design minimums for stability should be maintained when using RAs
- Stability alone does not appear to be a robust RA dosage indicator but should be checked to avoid over dosage in a balanced design framework;
- The effect of mixing and compaction temperature on stability is pronounced, resulting in a significantly higher stability independent of RA usage. The mechanism for this increase in stability is not mixture density alone.

Mixture CT Index

Effect of RAs on mixture CT Index

The CT Index (ASTM D8225) of the long term cured specimens is presented in Figure 21; ASTM D8225 was followed, except that the nominal sample height was adjusted to 95 mm as opposed to the commonly used 62 mm in the HMA industry. The reason for increasing the sample thickness was twofold: First, since stability is also run on 95 mm nominal specimens, the entire sublot of performance test specimens can be produced at one time without changing batch weights. Second, since cold mixtures tend to exhibit lower tensile strength than HMA, sample thickness was increased to accommodate the precision of the testing load cell. The calculation of CT Index allows for a sample thickness normalization, which was accounted for in this project. Error bars in this section represent ± 1 standard deviation from the average value, as

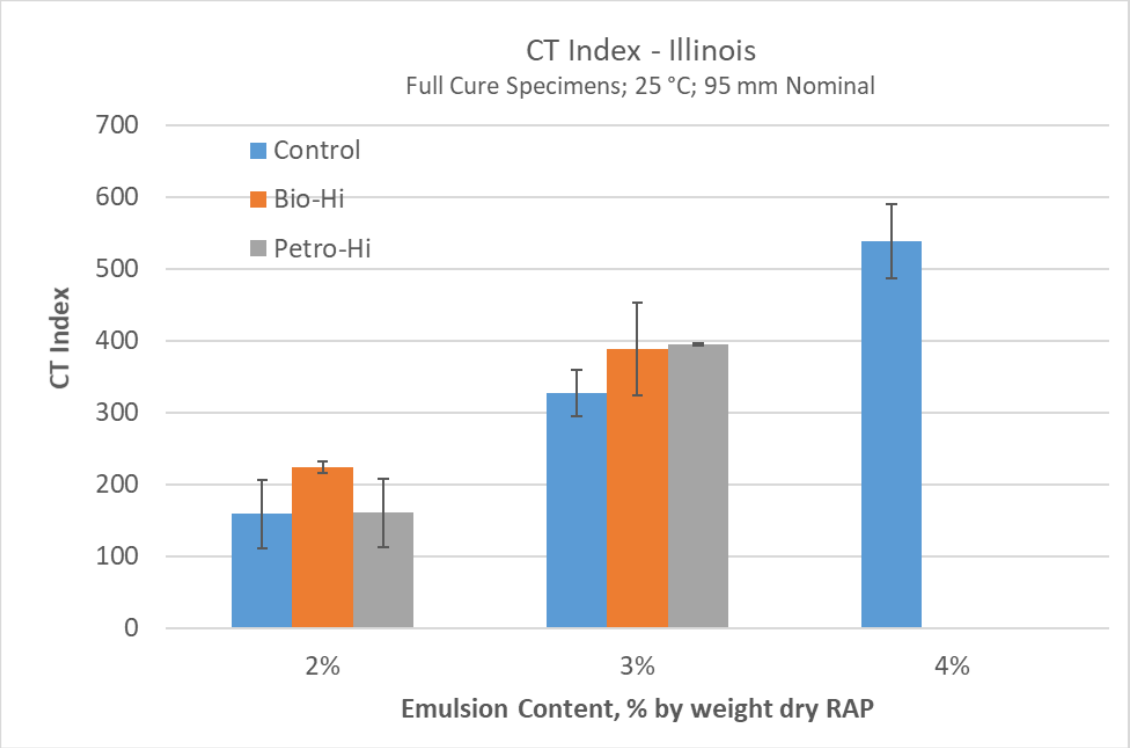
generally three specimens were tested per combination. A higher CT index is indicative of increased cracking resistance.

The first observation made from Figure 21 is that the relative magnitude of CT Index values are considerably higher than what is commonly observed in the HMA industry. There could be several causal effects contributing to this behavior; it is important to keep in mind that cold-recycled mixtures are sometimes classified as “non-continuously bound” as opposed to HMA, which is classified as “continuously bound”. Essentially the cured emulsion residue-RAP binder film is not continuous within the recycled mixture, which in turn affects the mechanisms by which these materials respond to load. In the case of the CT Index, cold-recycled mixtures typically exhibit lower peak load, but higher post-peak slope relative to HMA, thereby producing an over net-increase in CT Index. It is important that the actual CT Index values obtained for cold mixtures therefore not be directly evaluated against those from HMA without consideration of the mechanisms of cracking in these mixtures.

Data in Figure 21 shows CT Index is clearly sensitive to emulsion content, with increasing emulsion content producing a logical increase in cracking resistance. It is hypothesized that this phenomenon is related to the mixture response becoming more influenced by residue properties as residue percentage increases and the mixture becomes more continuously bound. The researchers believe that mixture post-peak slope is the primary indicator of film continuity and is the driver for CT Index. Figure 22 shows the relationship between Post Peak Slope and CT Index for all sample combinations and both RAP sources. The coefficient of determination for the combined dataset is nearly 90%. Interestingly, there is significant overlap between RAP sources and between emulsion blends within a given RAP source, illustrating that both binder quantity, but also quality drives performance.

The most notable difference between the Illinois RAP and Mankato RAP is the measured asphalt content. The Mankato RAP at 6.4% is almost 2% higher than the Illinois RAP at 4.6%. The authors hypothesize that both quantity of residue and quality of residue are important, and the effects of RAs are only practically realized after the “residue demand” for a given RAP is met. The residue demand could be related to the aggregate gradation within the RAP among other factors. Figure 23 illustrates this concept by isolating the Mankato RAP; the data in Figure 23 is blocked by emulsion content and shows that independent of formulation at 2% emulsion, the sensitivity of CT Index to Post Peak Slope is minimal. At 3% however, the effect of Post-Peak Slope on CT Index is pronounced: once the total residue demand is met, quality becomes the more important driver of cracking resistance. In other terms, quantity of residue is represented by color in Figure 23, quality of residue is the labeled data points.

Figure 21 data suggests that a practitioner could lower the added emulsion content and achieve similar CT Index values as a control material at a higher emulsion content. For the Illinois RAP source, this “shift” is approximately between 0.20-0.38% for the Petro-High and Bio-High emulsions, respectively. For the Mankato source the shift is closer to 1% emulsion.



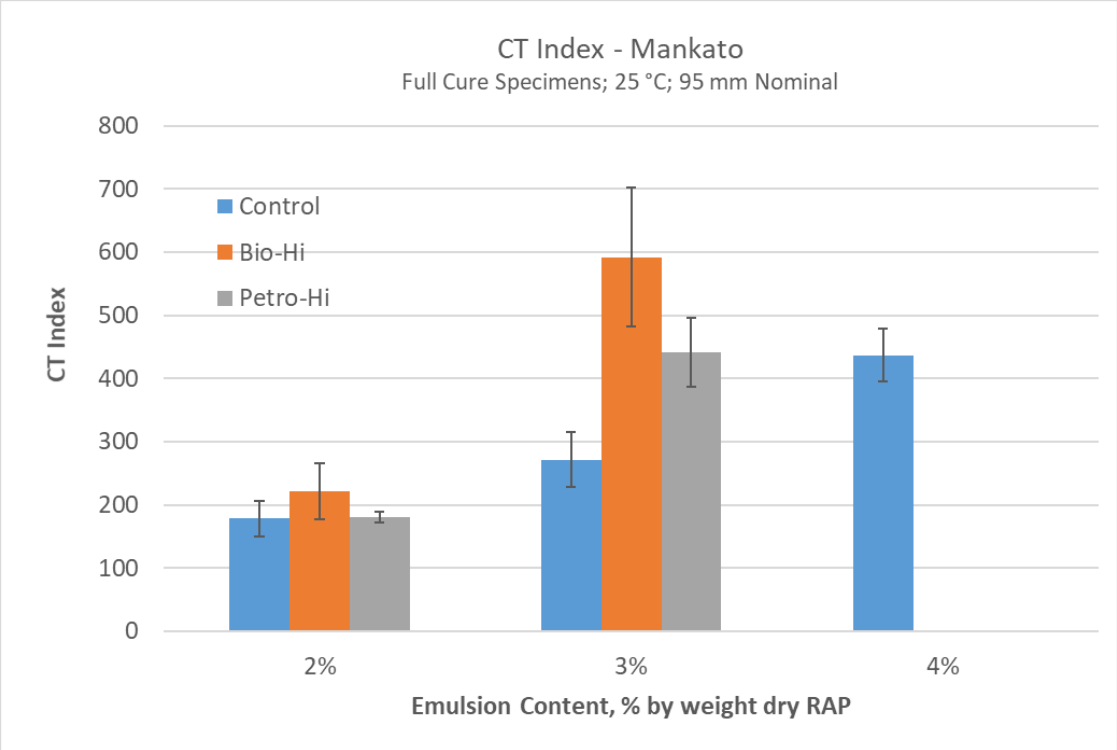


Figure 21. CT Index of fully cured specimens at 25 °C

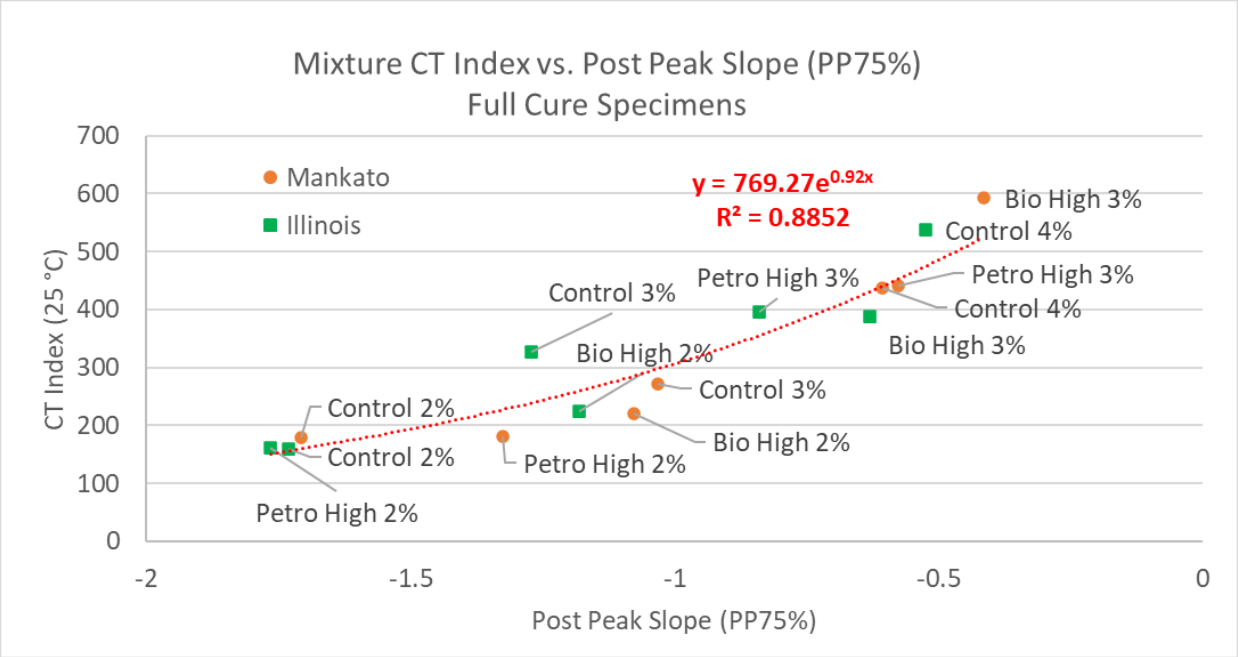


Figure 22. Correlation between CT Index and Post Peak Slope for both RAP sources and all emulsion combinations

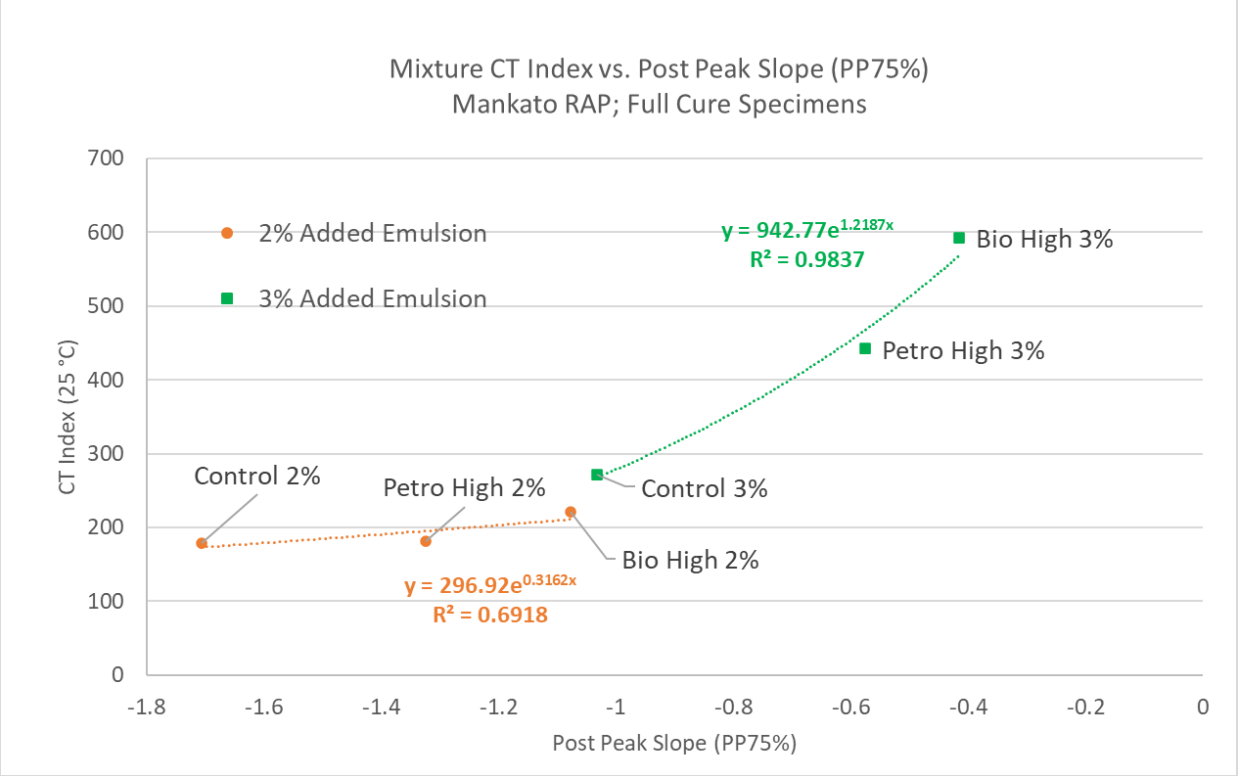


Figure 23. CT Index and Post Peak slope for Mankato RAP

Effect of RA dosage on mixture CT Index

Figure 21 clearly illustrates that CT Index can be used to demonstrate efficacy of RAs. It remains to be shown whether CT Index could be used as a robust dosage determination tool. Figure 24 shows the effect of RA dosage on the Illinois RAP material at 3% added emulsion. Results show that on average CT Index could be used to determine a minimum effective dosage, although variability in the testing is a challenge. At sufficiently low RA dosages, it does not appear the CT Index can detect the changes the RA affects upon the mixture. The research team believes that in the context of a balanced design framework CT Index could be used to quantify asphalt binder quality, but that dosage should be determined by other means, such as residue rheological properties. More information on a proposed balanced design framework will be outline in later sections.

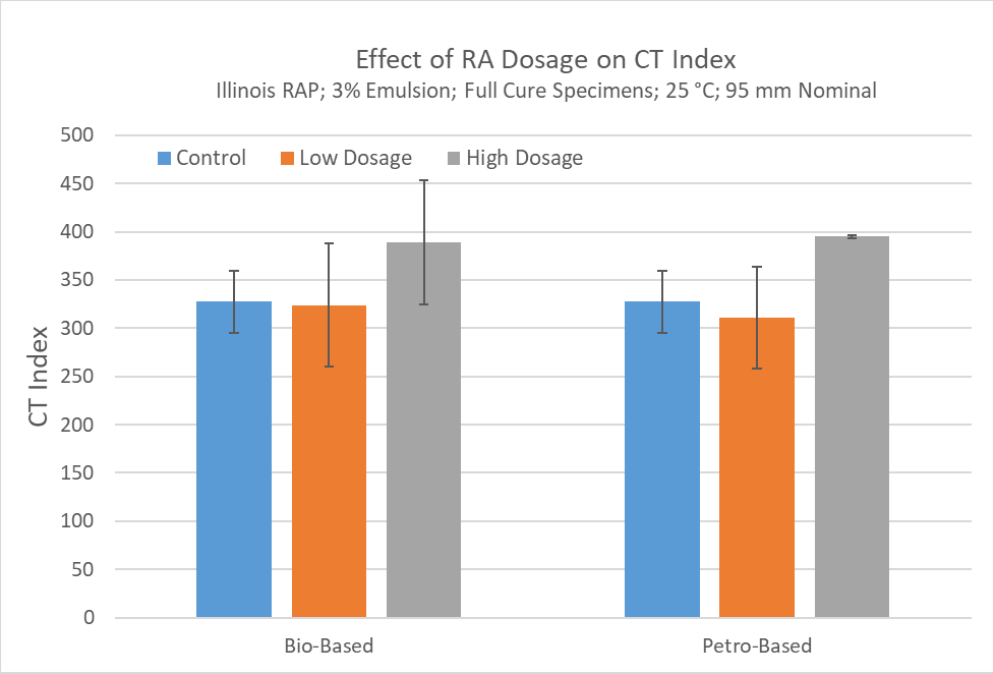
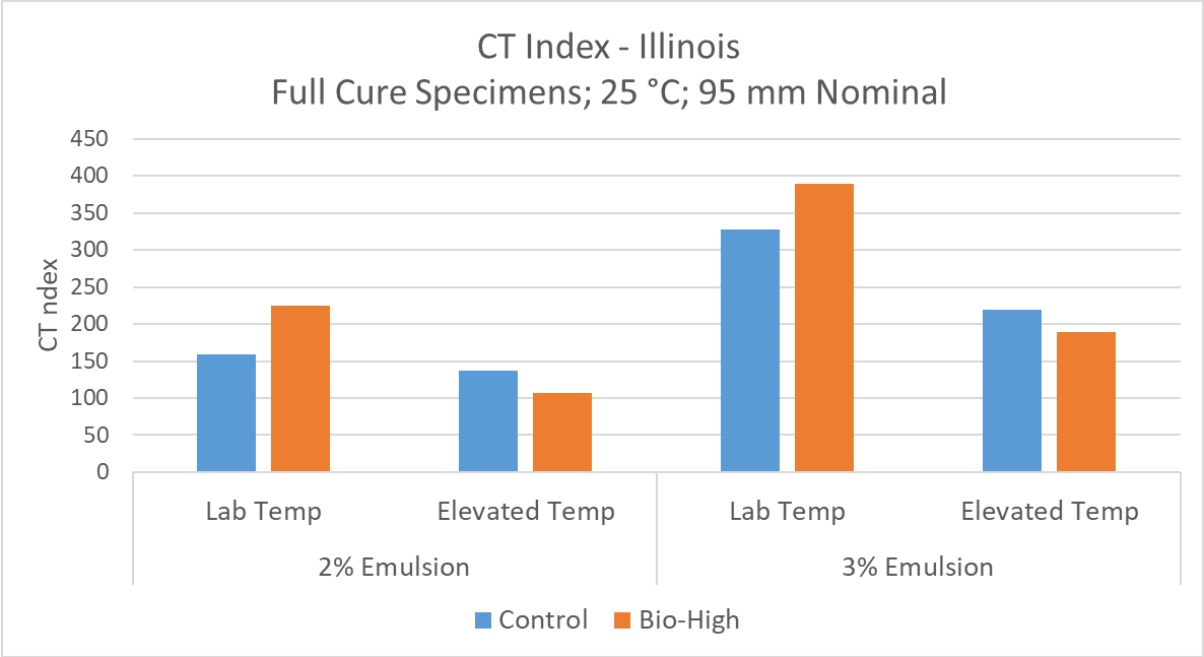


Figure 24. Effect of RA dosage on CT Index

Effect of Mixing and Compaction Temperature on CT Index

Mixture density is shown to be strongly influenced by mixing and compaction temperature in this study; it is known from the growing HMA CT Index database being generated by practitioners and researchers alike that CT Index is often counterintuitively shown to decrease with increasing density (lower air voids). Increased mixing temperature may increase the efficiency of RAs since more binder activation may be present at higher temperature. Figure 25 shows mixtures produced at 110 °F relative to the same mixture combination produced at lab temperature.

For all combinations, production at elevated temperature produces an average CT Index equal or lower to the CT Index of the corresponding lab temperature specimen. Notably, the effect of the RA appears to be muted at increased mixing and compaction temperature. Nevertheless, there was concern among the project committee that increasing mixing temperatures might cause the RA to over soften the mixture, and the combined stability and CT index results do not substantiate this concern. If anything, the use of RAs appears to be at least conservative if mixing temperature change throughout the day.



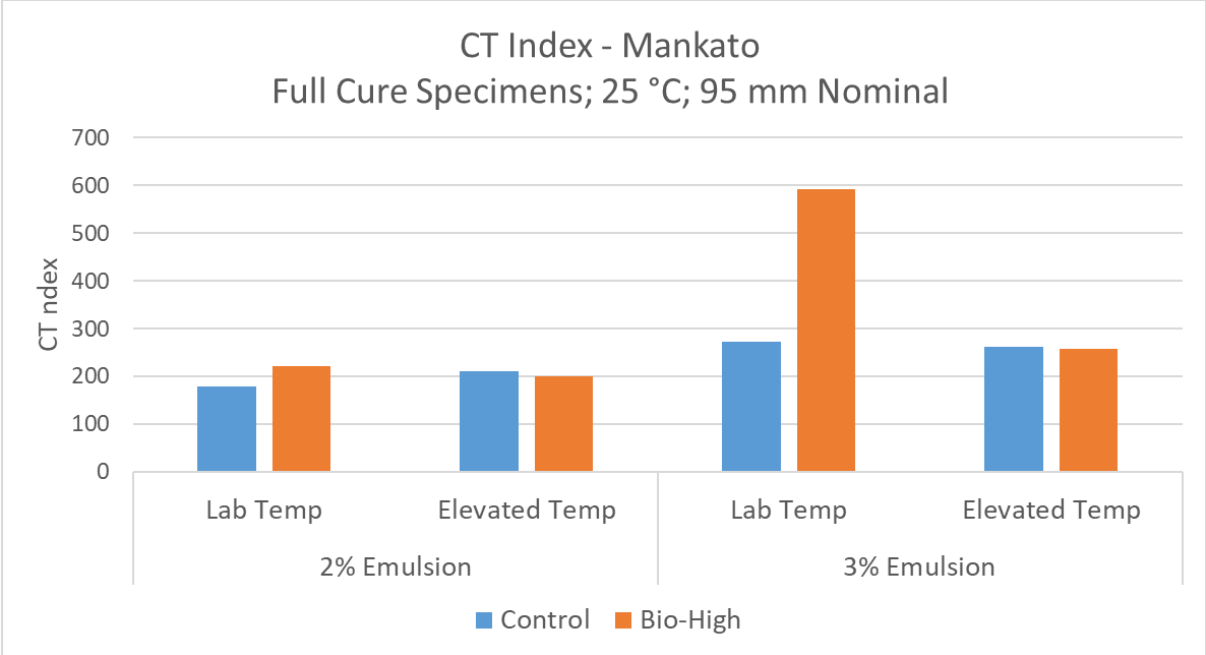


Figure 25. Effect of mixing and compaction temperature on CT Index

In a similar exercise to the stability section, CT Index is plotted against mixture air voids in Figure 26 - top. Although the correlations within a given mixing and compaction temperature are poor, the general trend is opposite of what is commonly cited in the HMA industry; for this dataset, CT Index increases with increasing density, whereas CT Index is commonly cited as decreasing with increasing density in the HMA industry. Curiously, it appears that for a similar level of air voids, the CT Index for lab temperature produced mixtures is higher than for elevated temperatures; the sensitivity (slope of regression line) of CT Index to air void level is also affected by mixing and compaction temperature, which is different from what was observed for stability.

When Post Peak Slope is plotted against air voids (Figure 26 – bottom), it is noted that there is more definite overlap in data between the two preparation temperatures. Considering the strong relationship between CT Index and Post Peak Slope (Figure 22), this observation suggests that differences in CT Index as a result of mixture preparation temperature are at least partially caused by mixture stiffness. Findings presented for mixture stability (Figure 19), confirm that indeed the mixtures produced at higher temperature result in higher stiffness. In other words, since preparation temperature is shown to impact mixture air voids, which is confounded with mixture stiffness, preparation conditions are critical during the design process.

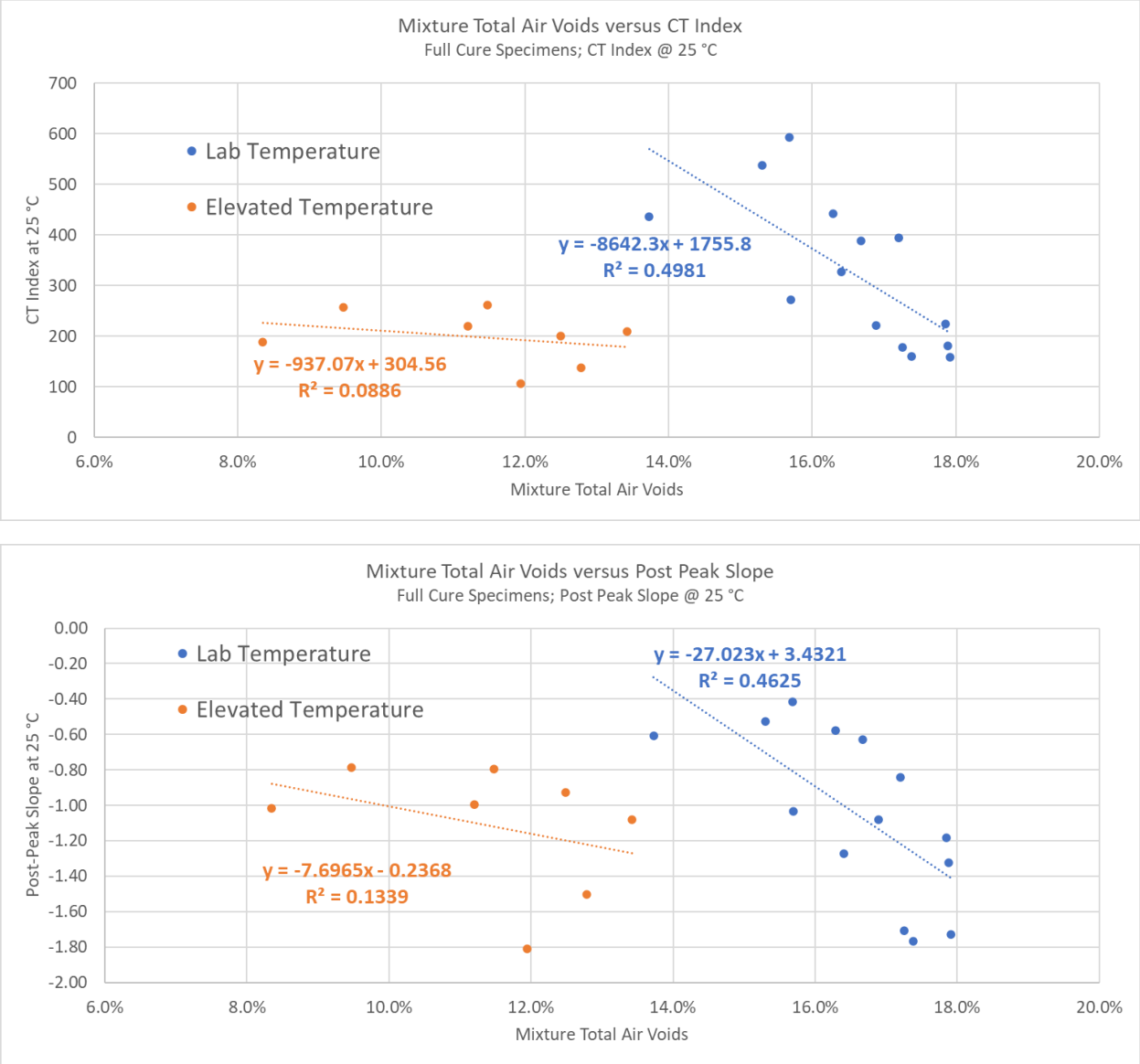


Figure 26. Mixture air voids vs. CT Index (top) and Post Peak Slope (Bottom) at two mixing and compaction temperatures

In the cold recycling industry, mixture air voids is not a design value as it is in HMA. Based on Figure 26, if CT Index is to be run at the recommended design emulsion content, then density will inherently vary design to design. The question of whether to a controlled air voids for CT Index testing of

cold recycled mixtures is a topic that needs to be further evaluated. A potential solution is to benchmark several designs with a history of performance and propose a minimum CT Index based on the designs themselves and allow density to be a factor designers can use to adjust performance. This is the approach outlined by the authors in subsequent sections.

Summary of Findings Related to Mixture CT Index

The following is a summary of the major findings related to mixture CT Index Testing;

- The CT Index test run on 95 mm nominal height specimens is sensitive to RAP source, emulsion content, emulsion formulation, and to a lesser extent RA dosage rate. Repeatability of the test is comparable to published precision estimates from the HMA industry.
- Overall, the inclusion of RA in the CR emulsions resulted in an improvement in the CT Index for both types of RA.
- Results show that there appears to be a balance between residue quantity and quality that ultimately controls cracking resistance measured with the CT Index. Specimens with higher total residue are generally more sensitive to the inclusion of RAs. It appears that CT Index is controlled primarily by the post peak slope during the test and attempting to increase CT Index should focus on this parameter.
- Results show that there may be interrelated effects of mixture preparation temperature, which effects mixture density, and RA effectiveness. This finding speaks to the need to understand project specific parameters such as local climate at the time of construction.
- CT Index is affected by mixture density, although mixture density is not a design factor during the mix design process for cold recycled mixtures; it is proposed that mix designers use mixture density as a variable to adjust CT Index values during a balanced mix design process.

The findings presented in this chapter support the concept of using a “Balanced Mix Design” approach for cold recycled mixtures. Of the existing mix design methods found during the literature search, the research team could not identify a measure of durability/cracking that was practical or sensitive enough to mixture factors for such an approach, hence the inclusion of CT Index in this study. It appears utilizing the existing high temperature (40 °C) stability test along with the proposed CT Index protocol could be a viable solution. Stability, CT Index, and ITS samples, if run, can all be made to 95 mm; additionally, since CT Index is essentially an ITS test in which more analysis is conducted on the testing trace, inclusion of this test as a report value may not add any additional specimens. The next chapter attempts to pull all of this information together along with residue testing results into a design framework.

Chapter 7 – Correlation of Binder and Mixture Results

Binder Analysis Overview

As part of the test plan in this project, two main sets of binders were tested an analyzed:

1. **Emulsion base binders** (referred to as a "Emulsion Residue" herein), tested using standard PG methodology utilizing the BBR for low temperature characterization.
2. **Extracted binder** derived from the RAP or from the lab produced mix samples used in performance testing. These samples were tested "as-extracted", meaning that they had been previously subjected to the lab mix conditioning and curing protocols previously described. Low temperature properties were derived using 4-mm cryogenic DSR method. No additional aging was carried out on the samples in order to be able to better relate binder and mixture performance without the confounding effect of additional conditioning.

The figures below show a high level summary of all the results achieved from the analysis of these samples. The grouping of the base binders and extracted binders are denoted on the graphs for clarity. Subsequent sections will focus on relating these properties to mix performance measures.

In the naming convention, "3%" denotes the emulsion loading in the mix, "LT" means Long Term curing. "Low" and "High" denotes the RA dosage level, corresponding to the low and high dosages determined in earlier chapters, while "Bio" (B1) and "Petro" (P1) represent the bio-based and petroleum-based RAs used in this project.

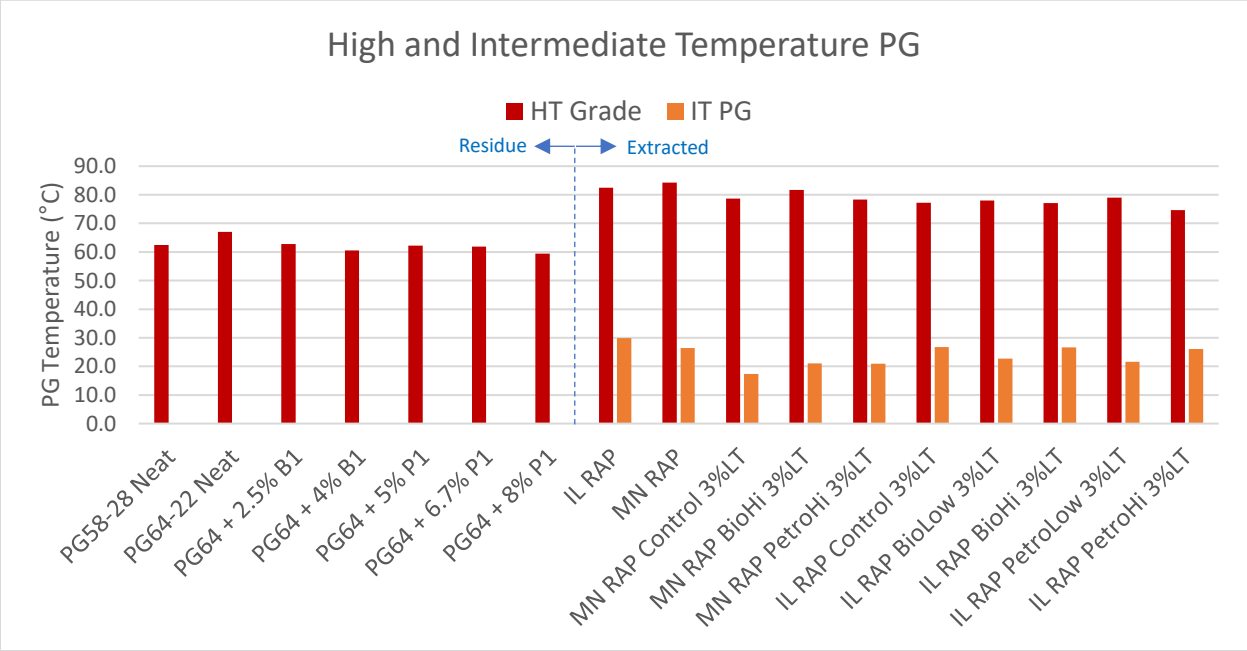


Figure 27. High and Intermediate Temperature Performance Grade of emulsion base and extracted CMA binder

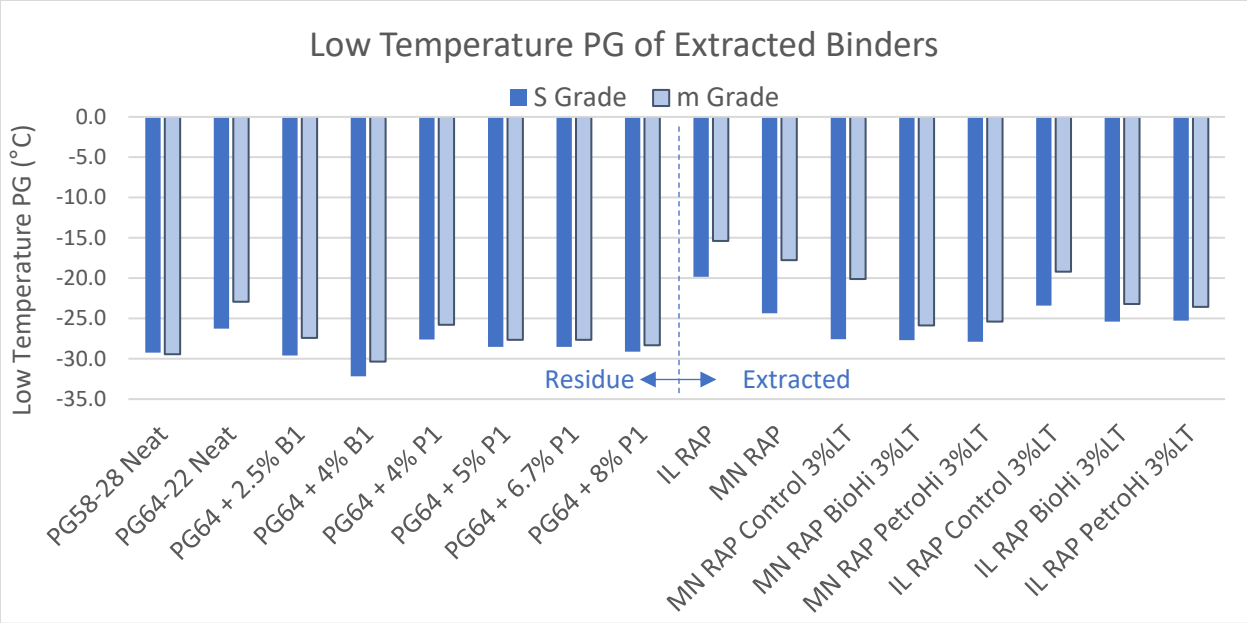


Figure 28. Low Temperature Performance Grade (Stiffness and Relaxation) of emulsion base and extracted CMA binder

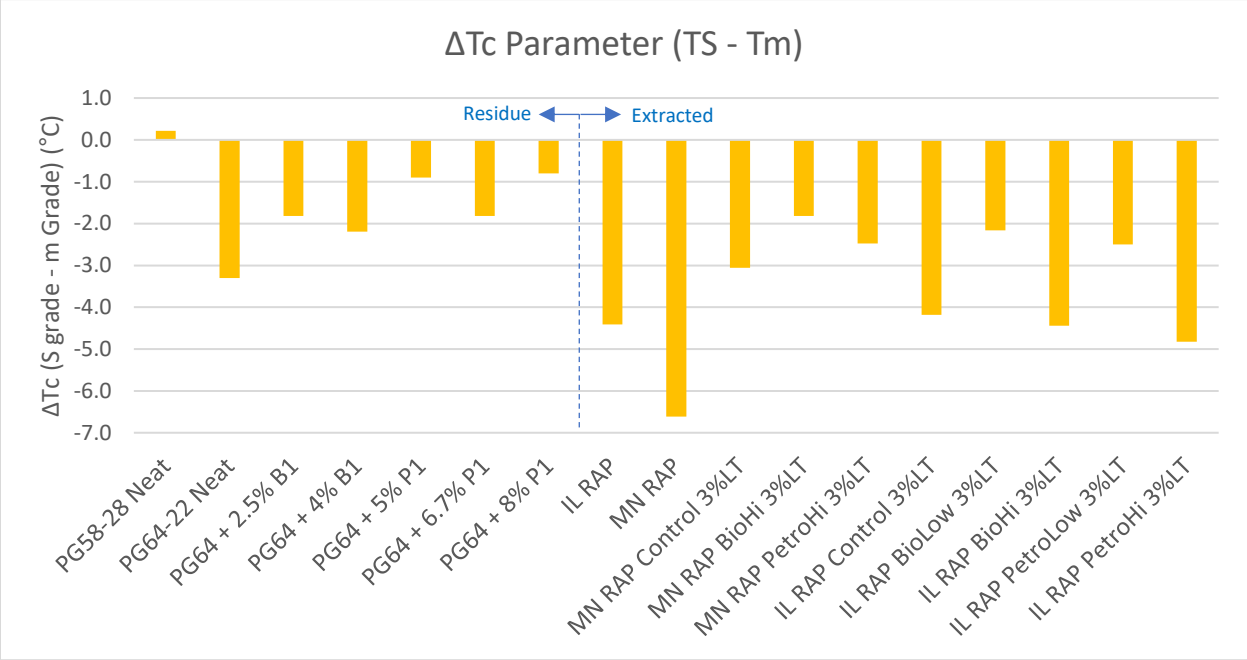


Figure 29 The "Delta Tc" parameter, measured as the difference between the Stiffness and Relaxation Low Temperature Performance Grades (S grade – m grade)

Figure 29 shows that despite the more positive Delta Tc for the control binder (PG 58-28) than the rejuvenated binders, the resulting extracted CR binders typically showed better Delta Tc values for the rejuvenated binders, especially for the case of the B1 RA. These results could be indications of better compatibilization of the aged binder by the inclusion of either rejuvenator.

Figure 30 and Figure 31 show additional rheological measures for the extracted binders, namely the "Glover-Rowe" parameters, and the Critical Phase Angle temperatures. These measures have been used in various studies to represent binder fatigue and durability parameters. It can be seen that overall, the MN RAP mixes had better rheological quality measures from the start, with the addition of RA having limited impact on properties. However with the lower quality IL RAP, use of the RAs, especially B1, had a marked impact on these measures.

Figure 32 shows that the aforementioned rheological measures correlated quite well with the low temperature relaxation grade (m grade), therefor it was decided to drop these parameters from further mix correlations in interest of use of the more familiar m-grade.

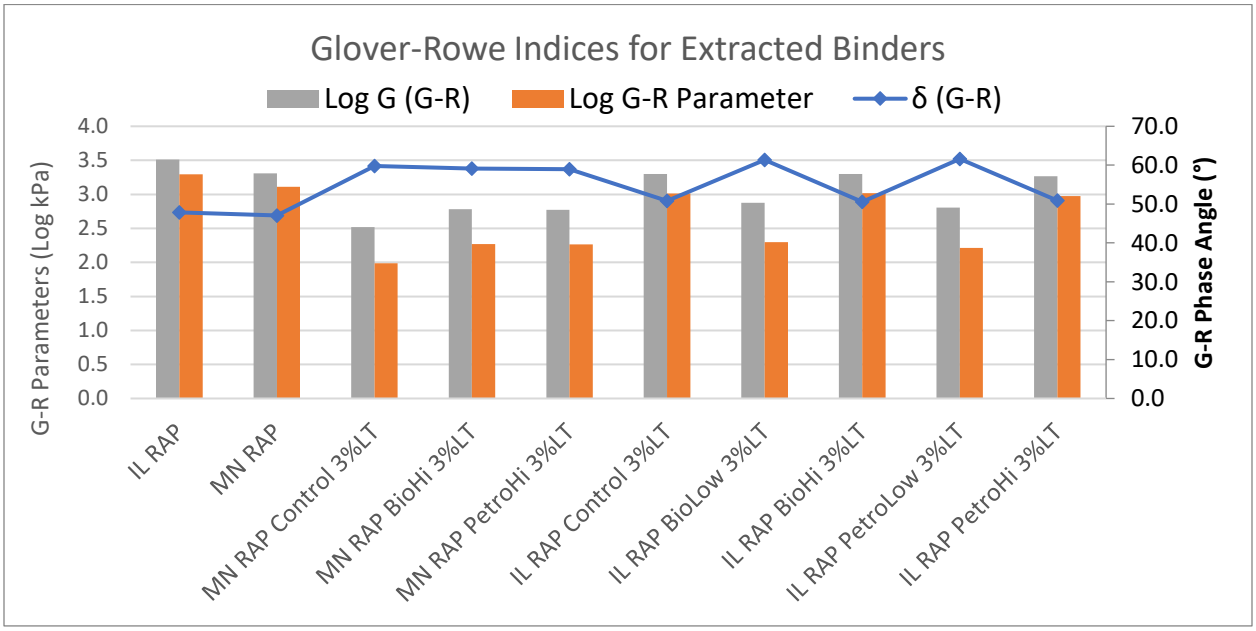


Figure 30 Glover-Rowe Parameters for the extracted binders

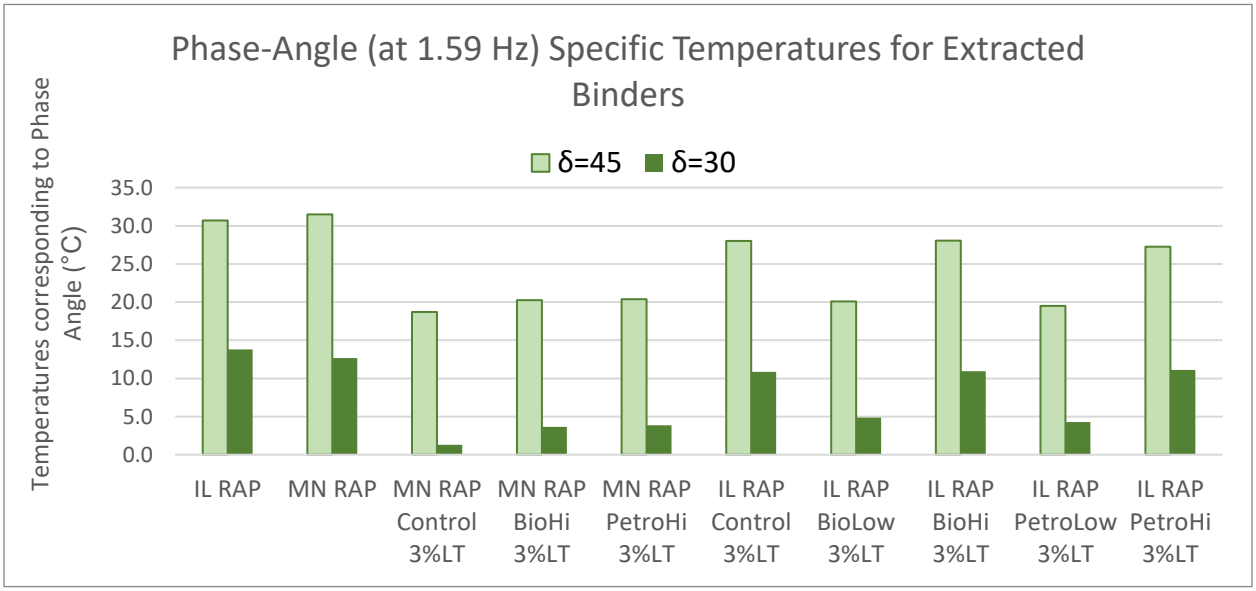


Figure 31 Phase Angle Critical Temperatures as 1.59 Hz (10 Rad/sec), measured for the extracted binders
Emulsion Residue vs. Extracted Binder Properties

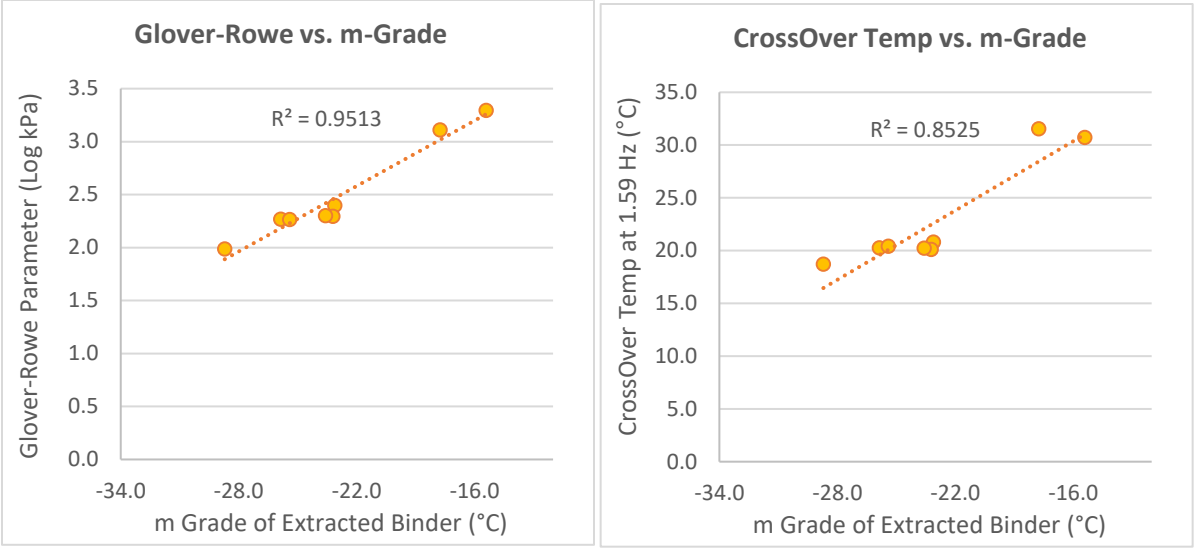


Figure 32 Correlation between Low Temperature m-grade and other rheological measures

Marshall Stability Comparison to Binder Properties

The mixture stability, as previously presented, were compared to the high temperature performance grades. Figure 33 shows that a rational relationship existed between the residue High Temperature PG, and that of the mix stability, with higher PG resulting in higher stability. The impact of the emulsion residue grade is less pronounced at the lower emulsion content (2%), as expected. In all cases the stability is meeting the required target values.

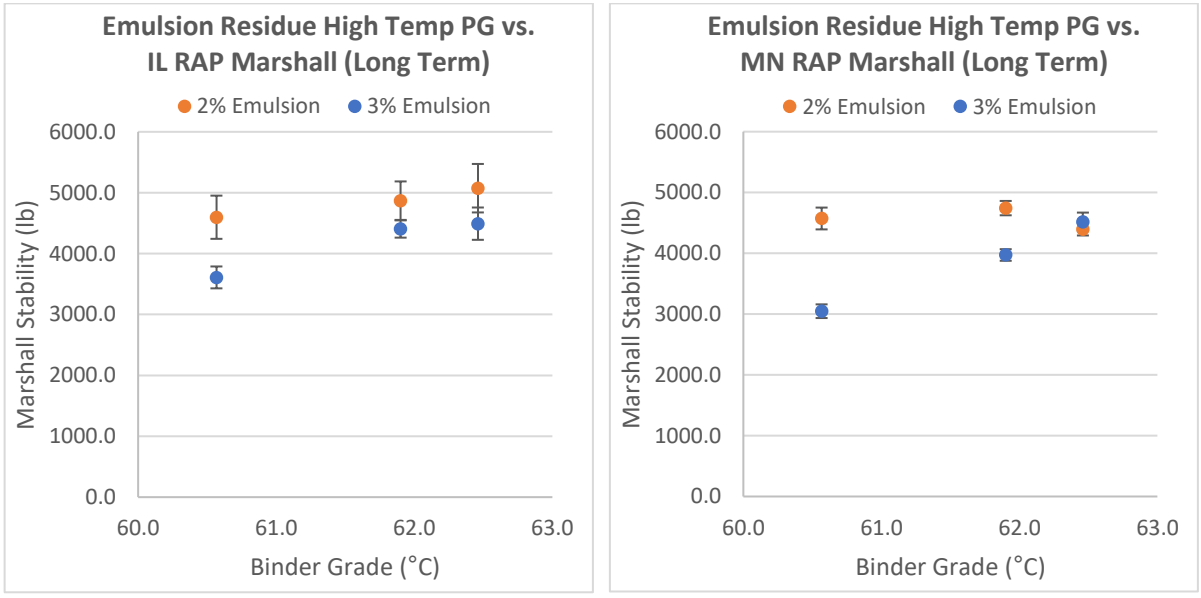


Figure 33 Relationship between Emulsion base binder PG and the mixture stability (error bars represent standard deviation of 3 replicates)

Looking at the relationship with the extracted binder grades (Figure 34), one does not see much of a relationship, as the change in stability values were relatively modest.

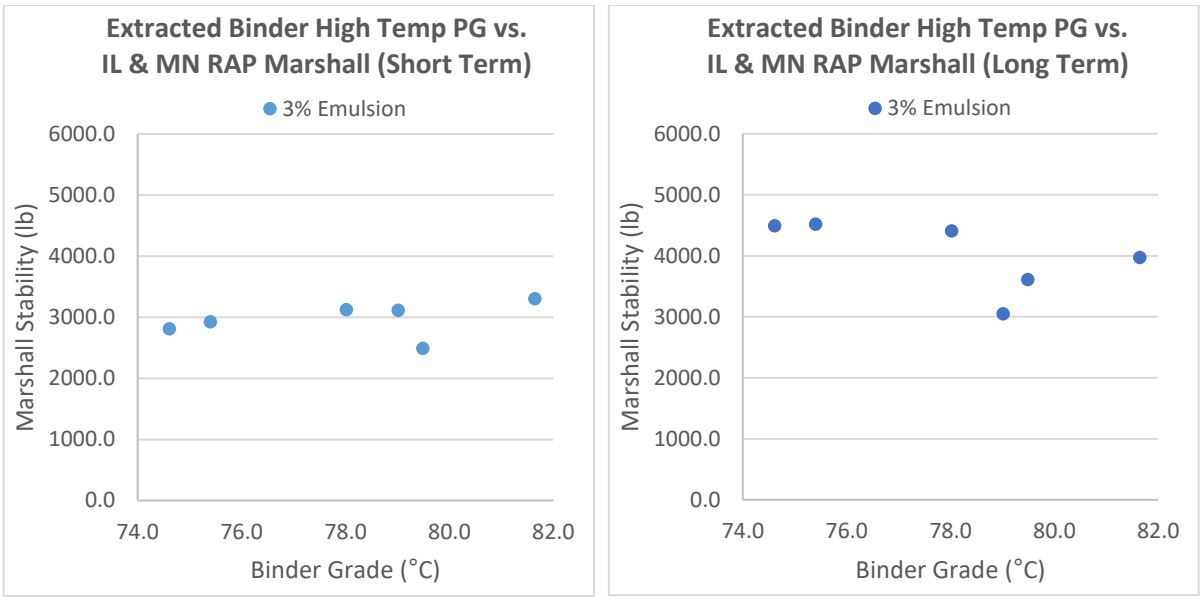


Figure 34 Relationship between Extracted binder PG and the mixture stability

IDEAL-CT Performance Comparison to Binder Properties

The IDEAL-CT test results were plotted against the extracted binder properties, as shown in Figure 35. Improvement of both the relaxation controlled low temperature PG well with the significant improvement in the CT Index, and extremely well with the Post Peak slope of the IDEAL-CT. This corresponds to earlier observations of the sensitivity of post peak slope to RA impact.

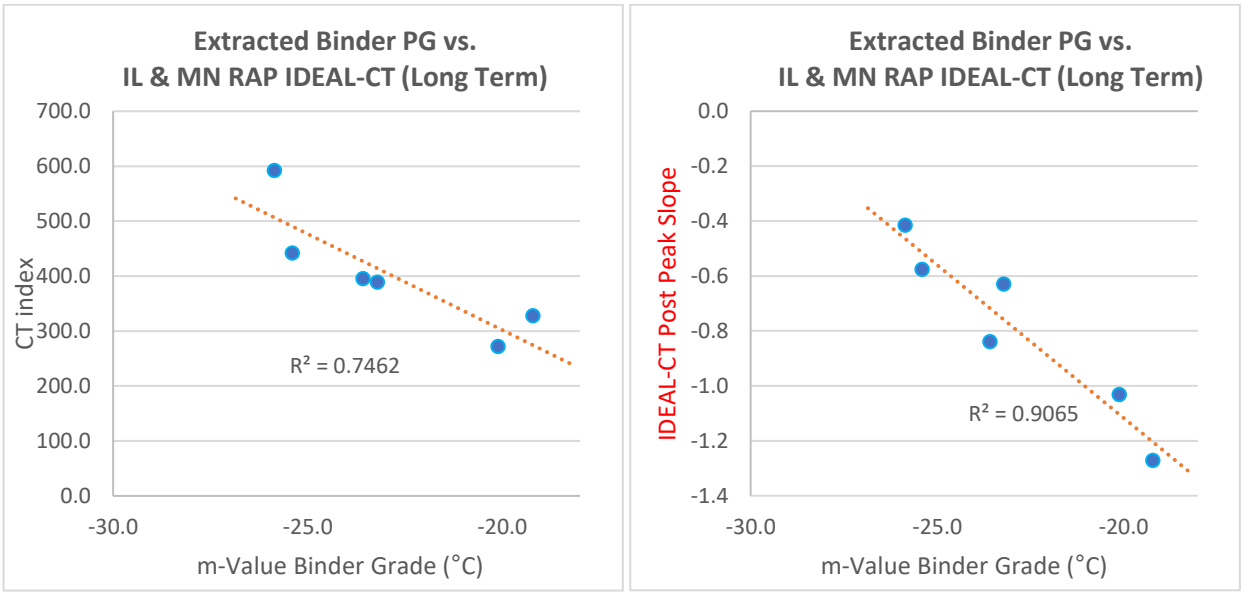


Figure 35 Correlation between low and intermediate performance grade and CT Index

Figure 36 shows that the Delta Tc parameter did not provide very strong predictive power in terms of the value of the CT Index, and even less so with the Post Peak Slope. ON the other hand for the Intermediate Temperature PG and the Glover-Rowe Parameter, a significantly stronger correlation existed between the extracted binder and the post peak slope, than with the CT-Index. This does not necessarily

mean that the Delta Tc is not useful in providing a high level understanding of binder quality, especially if extended aging is involved. As indicated earlier, the Delta Tc values of both RAP types showed a marked improvement with rejuvenation, however, it seems like the ability to precisely quantify or rank short term aged samples in terms of cold recycled mix performance does not seem evident in the present data set.

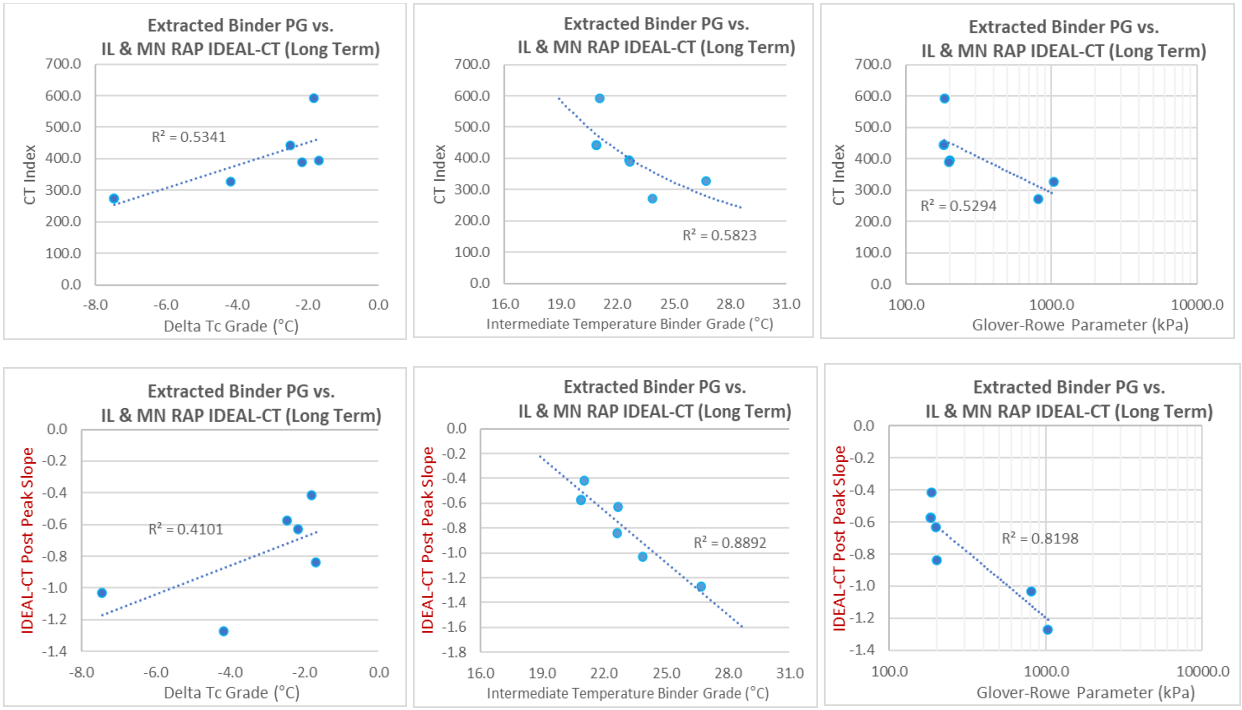


Figure 36 Correlation between Delta Tc, Intermediate Temperature PG, and the Glover Rowe parameter grade, with CT Index and the IDEAL-CT Post Peak Slope.

The present chapter provided an overview of binder performance and analysis results, and established the presence (and in some cases, the lack of) relationship between various binder parameters and the cold recycled mix performance. The findings of this chapter are used in the next chapters to create recommendation for the dosing and design of rejuvenating cold recycling emulsions.

Chapter 8: Interpretation and Proposed Design Framework

Balanced Mix Design

Findings presented in Chapter 7 support the concept of using a balanced performance testing framework for cold recycled mixtures containing RAs. In this study, the stability test is used to measure deformation resistance ("rutting") and the CT Index test is used as a measure of durability ("cracking"). Figure 37 shows the fully cured specimen stability and CT Index data collected during this study (lab temperature preparation). The determination coefficient (R^2 = 76%) shown in Figure 37 includes both RAP sources and all available data points. The data in Figure 37 clearly demonstrates the balance between stability and CT Index as it relates to RAP source, emulsion content, and efficacy of RAs. The determination coefficient for mixture stability and Post-Peak Slope for all specimen combinations is 69.9%, indicating that although CT Index is substantially controlled by Post-Peak behavior for these mixtures, more information can ultimately be gleaned from specifying CT Index; this finding validates Figure 22. Similarly, it is more practical to specify CT Index due to industry familiarity with the parameter, rather than to base a specification based on a less familiar testing metric.

It is envisioned that minimum limits on mixture stability and CT Index could be applied to such a chart to establish quadrants within the chart representing all permutations of stability and CT Index (High Stability/High CT Index, Low Stability/High CT Index, etc.). The data presented in Chapter 7 confirms that current minimum limits on mixture stability can be maintained with the use of RAs to prevent over-softening. The data presented in this study was generated using 150 mm specimens as opposed to the more commonly used 100 mm specimens in the CR industry. Two Agencies that allow use of 150 mm samples for stability (Illinois and Virginia) have a specified minimum limit of 2,500 lb. Consulting published literature, Kandhal and Brown recommend a ratio of 2.25 when converting between 100 mm and 150 mm stability specimens; a minimum stability of 1,250 lb for 100 mm samples (several Agencies specify this as the minimum 100 mm stability limit) would equate to 2,813 lb for 150 mm specimens. Based on this information, a 40 °C Stability limit for 150 mm specimens of 3,000 lb is proposed.

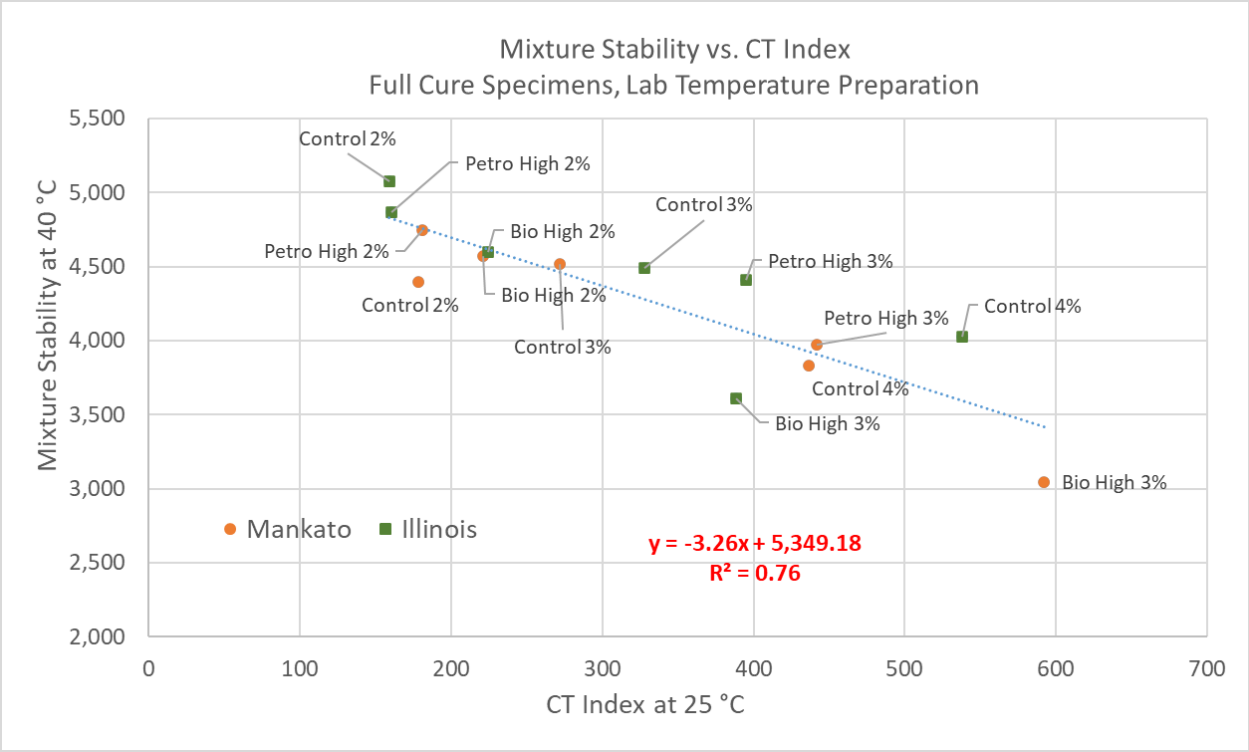


Figure 37. Mixture Stability vs. CT Index for all mixtures

Since CT Index is a relatively new test, and particularly for the CR industry, a minimum proposed value is less evident. The design emulsion content for both RAP source mix designs used in this project were reported as 2.5% by the consulting labs conducting the designs. By all indications both designs represent "typical" cold recycling designs for the respective regions; although performance may not be evident for several years, at the time of this project (approximately 18 months post-construction) no negative reports of performance have been received.

The CT Index value versus emulsion content for the control emulsion is shown in Figure 38 for both RAP sources. Best fit lines show excellent determination coefficients for both sources, albeit with different sensitivity to emulsion content (slope). Interpolating to obtain the CT Index resulting from the design emulsion content of 2.5% results in CT Index values of 231 and 247 for the Mankato and Illinois sources, respectively. For the purposes of this study, an average of those values (239) will be used for further calculations. The pooled standard deviation for all control CT Index specimens is approximately 42 CT Index units. The one standard deviation range of CT Index from the mean value at 2.5% emulsion content is approximately 200-280. For the purposes of this project, a minimum CT Index value of 200 is proposed for consideration and further discussion. The justification for this value is that it should not preclude the use of traditionally satisfactorily performing materials and allow some mix design tolerance, but also should be relatively conservative to avoid under-asphalting mixtures.

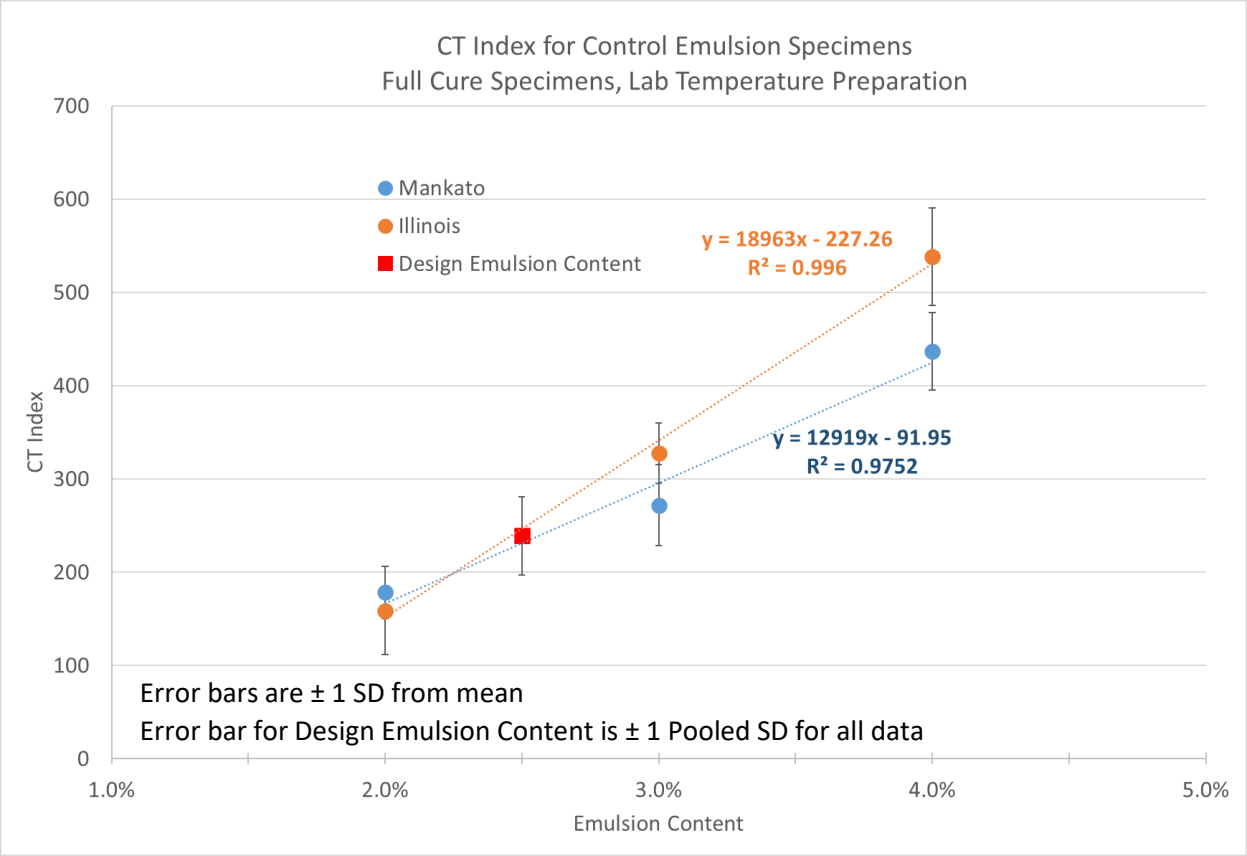


Figure 38. Determination of minimum proposed CT Index based on Control materials

Figure 37 is reproduced below as Figure 39 with the proposed limits for stability and CT Index included, and the associated "performance quadrants". The ramification of such a framework is that target emulsion content for a given RAP could be lowered relative to control materials when utilizing RAs. For example, using a CT Index "tolerance" of 50 units: for the Illinois source, an equivalent CT Index value of 250 occurs at approximately 2.2% emulsion content for the Bio-High emulsion; the same analysis for Mankato results in an equivalent emulsion content of 2.1%. Both would represent a "savings" in terms of emulsion content over the design of 2.5%, without sacrificing performance.

This framework also gives practitioners more insight on how to adjust mix design to meet specifications. For example, RAP sources that are relatively low on asphalt may benefit from a combined approach of use of RAs plus a relatively high add rate of emulsion. For RAP sources with relatively high asphalt (Mankato), the use of an RA plus a slightly reduced emulsion content may be more beneficial. Obviously, the database of materials must be expanded further to adjust the proposed limits, but results are nevertheless promising.

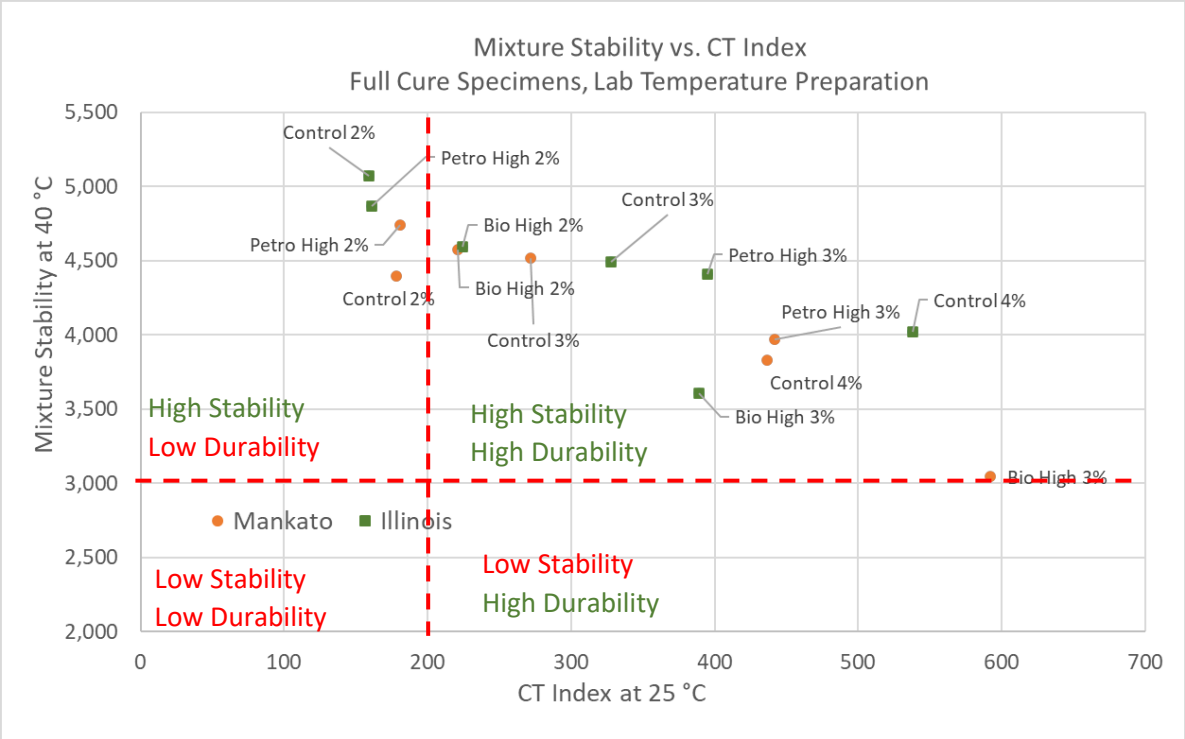


Figure 39. Proposed balanced design framework for emulsion stabilized recycled mixtures

It should be mentioned that the above-outlined framework does not preclude the use of other mixture tests to verify a design emulsion content. For example, in regions where the ASTM D 7196 Raveling Test is used, it is envisioned the raveling test could be used to verify the design emulsion blend, particularly if a reduction in added emulsion is the outcome of the analysis.

Rejuvenating Cold Mix Emulsion Design

An important aspect of implementation of the aforementioned Design Framework is the existence of a rejuvenating emulsion specification and dosage selection tool. Chapter 7 results showed that the intermediate and low temperature rheological properties of the extracted binders were most predictive of the IDEAL-CT performance in the mix. It is observed that the mixture extracted binder low temperature grades of around -23 to -24 (as-extracted with no further binder conditioning) yielded CT Index results in the range of 300. Correlating the results directly to the emulsion residue properties was a less straight forward, as such correlations neglect the significant impact of the RAP binder properties. This is shown in Figure 40.

Making the following key assumptions, some helpful conclusions can be derived:

1. Due to the separation of the emulsion producer from the cold recycling contractor, it would be logistically impractical to adjust RA dosing based on project specific material properties. Instead, it is preferable to design a reasonably conservative and regionally calibrated standard rejuvenating emulsion grade; the CIR designer or contractor can adjust the emulsion dosage as needed to optimize performance, much like is done presently for existing engineered emulsion cold recycling designs.

2. The results show a possible decoupling between the softness of the emulsion residue, and the final cold recycled mix performance. Therefore it is deemed reasonable to conclude that a minimum dosage of RA is necessary to achieve any benefits from the use of such additives.
3. Significant experience exists with the use of rejuvenators in asphalt emulsions used in "rejuvenating scrub seals". Therefore the existing scrub seal rejuvenating emulsion quality specifications can be used as a starting point for cold recycling rejuvenating emulsions.

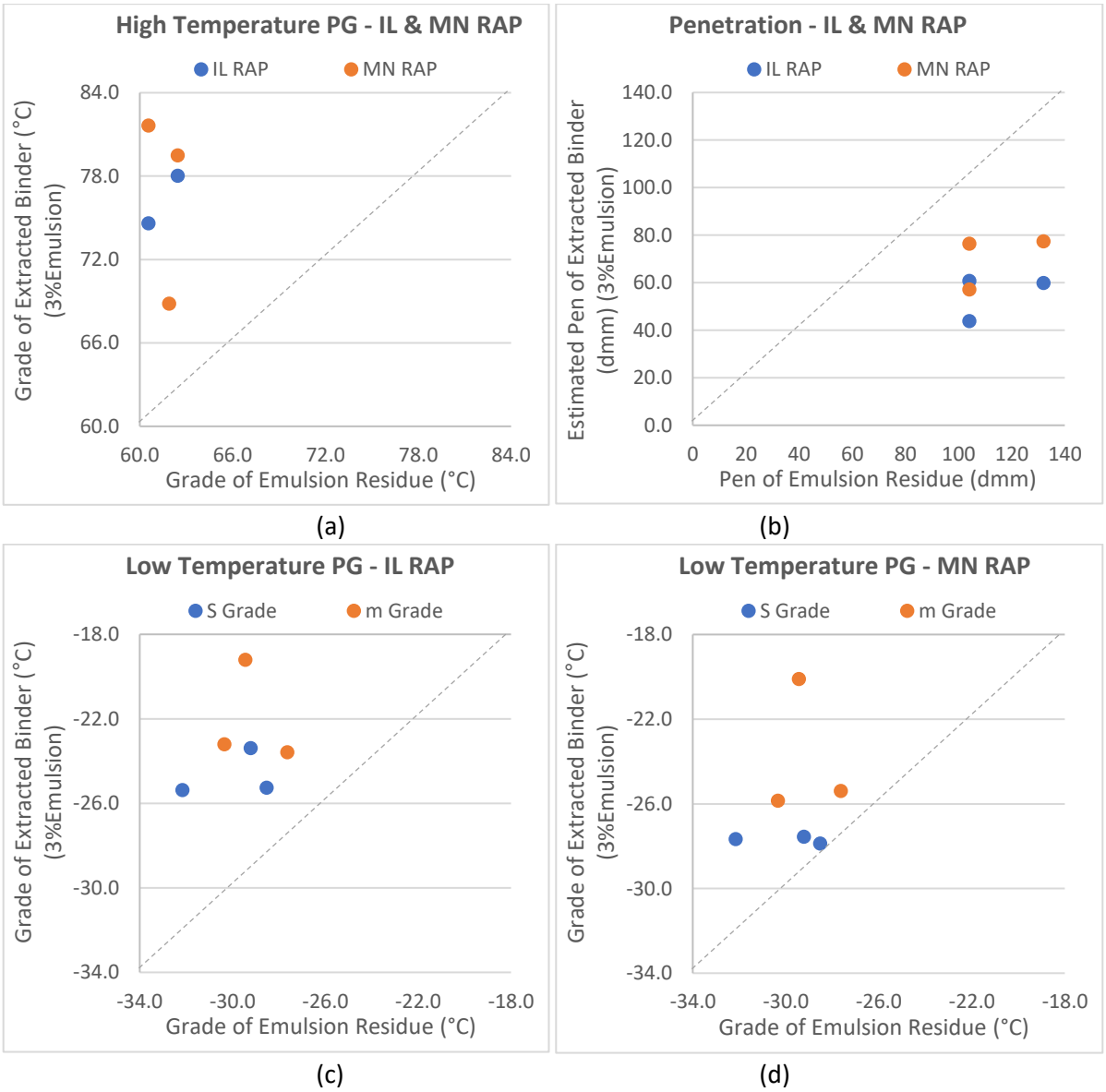


Figure 40 Relationship between emulsion residue and resulting extracted binder PG

It should be noted that Figure 40(b) compares the measured penetration of the residue (all in the range of ~100 ± 10 dmm with that of the estimated penetration of the final extracted binder. The estimation is based on empirical modeling of the mastercurve output, and is only meant for qualitative demonstrative purposes. It is noted that outside of the outlier point, the penetration of both the emulsion base binder and extracted binder varied in a narrow range, and did not provide any meaningful correlation

to mixture performance measures, therefore the following RA dosage discussion is focused on the low temperature PG, as the leading measure that correlated with the IDEAL-CT results.

Based on the above assumptions and discussions, and taking a holistic view of the binder vs. mixture results from the prior chapters, the following emulsion residue design method is recommended:

- **RA Dosage:** RA Dosage be adjusted to achieve a 6°C shift in the low temperature performance grade of the intended base asphalt used in the emulsion production. For example, if a PG64-22 has a continuous low temperature grade of -23°C, sufficient RA dosage be used to achieve a -29°C.
    - For the current study, this would result in about 3.5% of B1, and 8% of P1.
    - It should be re-emphasized that the intent here is not to achieve a specific grade, but to use the grade shift to ensure the required dosage of RA is present in the emulsion to yield a positive impact in the final mix.
- **Base Binder:** the base binder grade, prior to inclusion of the RA impact, needs to be sufficiently stiffer than typical climatically appropriate cold recycling emulsion residue grades to be able to meet the climatically appropriate grade after the inclusion of the impact of the required RA dosage. It is suggested that a one PG shift base binder be used for this purpose. It is anticipated that use of the stiffer binder + RA will allow for an overall reduction in emulsion content in the mix compared to use of the typical softer binder grade.
    - For example, for a climate such as the upper Midwest that typically use PG58-28 emulsion residues for engineered emulsions, a PG64-22 binder may be used as a base in a rejuvenating emulsion.
    - The justification for this step is to obtain the quality benefits from using an RA without over-softening the emulsion residue.
- **Emulsion Quality Specification:** No changes recommended to current practice for cold recycling Engineered Emulsions, other than addition of the language around the base binder selection to accommodate the RA dosage, as described earlier.
- **RA Specification:** Recommend using the most recent version of Table 2 proposed in August 2019 by the Emulsion Task Force for use in the AASHTO "Standard Specification for Materials for Emulsified Asphalt Scrub Seal", as shown below:

Table 16—Tests on Rejuvenating Agent

	Method	Min	Max
Tests on Rejuvenating Agent			
Viscosity, 60°C (140°F), CST	T 201	15	300
Flash Point, °C (°F)	T 48	182 (360)	
Saturates, %	ASTM D4124[a]		30
Tests on TFO or RTFO Residue			
Mass Change, %	T 179 or T 240		6.5
Viscosity Ratio	T 179 or T 240		3

a IP-469 is an acceptable replacement for measurement of % saturates.

The framework described in this section is meant as presentation of the reasoning behind the proposed implementation process and specification modification. These modifications are presented in the form of a suggested specification amendment for use by interested agencies in an appendix of this report.

Chapter 9 – Conclusions and Recommendations for Future Work

Cold-recycling processes such as Cold in Place Recycling (CIR) and Cold-Central Plant Recycling (CCPR) offer opportunities for innovation through the use of recycling additives (RAs). The objective of this project is to evaluate the efficacy of rejuvenating asphalt emulsions in the CIR and/or CCPR process in terms of potential performance benefits relative to existing stabilization options. An experimental matrix was designed to include several of the mix design factors known or thought to control mix performance. Rejuvenating asphalt emulsions containing both Bio-based and petroleum-based RAs were produced and compared to a control engineered emulsion with a proven field history of performance. The concept of utilizing a "Balanced Mix Design" approach is explored to quantify the performance attributes of these materials. Mixture stability at 40 °C and mixture IDEAL CT Index at 25 °C are ultimately selected as the performance tests used in the balanced mix design framework. The following conclusions are made:

- There is a balance between asphalt residue quantity and quality that controls mixture performance:
    - RAs do not appear to negatively impact mixture stability or the mechanism of strength gain with respect to stability during curing for the materials tested in this study;
    - The inclusion of RAs generally improved the CT Index of the cold recycled mixes compared to the use of a similarly graded control emulsion;
    - CT Index specimens with higher total residue are generally more sensitive to the inclusion of RAs and CT Index is sensitive to RAP source, emulsion content, emulsion formulation, and to a lesser extent RA dosage rate.
    - Repeatability of the IDEAL test for cold recycled mixtures tested in accordance with the methods in this report is comparable to published precision estimates from the HMA industry.
- The use of RAs in this study does not appear to influence the compaction behavior of cold recycled mixtures, and compacted air void level is similar to control mixtures.
- There may be interrelated effects of mixture preparation temperature, which effects mixture density, and RA effectiveness. This finding speaks to the need to understand project specific parameters such as local climate at the time of construction.
- The relationship between the emulsion residue high temperature PG and the mixture stability was straight forward, with higher residue grades correlating with slightly higher stability values. The overall impact of the grade on stability seemed to be less important than the impact of the total emulsion content in the mix.
- The emulsion residue properties did not directly correlate with IDEAL-CT results. This is likely due to the importance of RAP binder and emulsion interactions, such as the degree of RAP binder reactivation and reincorporation through rejuvenation or other mechanisms on the final IDEAL-CT results. Better correlations could be derived through use of the extracted binder results.
- The Low Temperature m-value of the extracted binder had the best correlation to the IDEAL-CT performance of the mix (both CT-Index and Post-peak Slope), followed by the Intermediate Temperature PG and the Glover-Rowe Parameter. The correlations were stronger for the IDEAL-CT post peak slope compared to the CT-Index. IDEAL CT results did not correlate well to Delta Tc.
- Findings support the concept of using a "Balanced Mix Design" approach for cold recycled mixtures. Suggested performance limits for 95 mm nominal height, 150 mm diameter, fully cured specimens are:

- Mixture Stability at 40 °C: 3,000 lbs, minimum at design emulsion content
- IDEAL CT Index at 25 °C: 200, minimum at design emulsion content
- Other mix design tests as specified by the owner (such as raveling and moisture conditioning) should be run for verification purposes.

Recommendations for Implementation of the Findings, and Future Work

A framework for mixture design and RA dosage was proposed as part of this study. To aid rapid implementation of the results, the recommendations were also written in the form of a specification amendment document, included in the appendix of this report. For this purpose, the MnDOT CIR design procedure framework was utilized, although it is envisioned similar revisions could be made to many local design frameworks. The specification recommendations were written in a manner to be inclusive of the possibility of use in various climates, or with different types of RAs, beyond those used in this study.

The authors recommend that these recommendations be used as the basis of trial production sections during which certain aspects of the document can be vetted and further refined for potential full implementation in the future. It is also recommended that emulsion suppliers review and attempt the trial implementation of the RA dosage methodology and specification in their practice, and potentially using various RAs in the market. The feedback can be valuable in further refining recommendations if necessary.

The RAs used in this study were first blended into the base asphalt before the emulsification process. It is understood that this method of incorporation may not be practical for all emulsion suppliers and for all projects. Limited work conducted by the authors but beyond the scope of this study suggests that there exist at least two other modes of RA incorporation that could prove more feasible on certain projects and may offer more flexibility in the design process; both methods require the emulsion producer to create an oil-in-water emulsion of the RA using the same emulsion chemistry used for the base asphalt emulsion and targeting a similar residue content (usually around 60% residue). To utilize the RA, the emulsion supplier or contractor may:

- Blend the two emulsions at a ratio that results in the desired RA application rate and use the blended emulsion in the standard process, or
- Dose the RAP with the oil-emulsion prior to dosing the RAP with the asphalt emulsion. The dosage rates of the two emulsions will be different and can be designed to meet project performance goals. This method may be better suited for CCPR processes, but in theory could be used in CIR processes as well.

The performance limits proposed are based on a testing matrix of approximately 25 mixture combinations; nevertheless, the testing database needs to be expanded to further validate limits for a wider range of in-service materials and RA incorporation methods described above. It is recommended that the CT Index value be a “report only” value for the trial emulsion contents during the design process consisting of the testing of at least three 95 mm ITS specimens. This analysis can be run in addition to existing design tests or by analyzing the dry sample ITS curves for CT Index. Many existing ITS load frames have the capability to utilize built in or aftermarket data capture software at relatively low expense to the contractor/designer.

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Appendix A: Special Provision for Mix Design Utilizing Rejuvenating Engineered Asphalt Emulsion

Note: Specification adapted from 2020 MnDOT Grading and Base Manual Section 5-692.291 "CIR Mix Design Procedure". Redlined items are those recommended by the authors.

x-xxx.xxx CIR Mix Design Procedure Utilizing Rejuvenating Asphalt Emulsion

A. General

- If cement is used, the recommended amount is 1 part cement to 3 parts residual asphalt by weight, but no more than ½% of cement.
- Emulsions are recommended for urban projects.
- For low temperature crack resistance, stability and strength, it is recommended to add crushed rock, in lieu of cement or other mineral stabilizers. Design using cement, if stability and strength cannot be obtained with bituminous material and crushed rock.
- ~~The recommended PG grade of foam asphalt is 49-34.~~
- The emulsion is a rejuvenating engineered emulsion meeting the requirements of section B.2
- Use as a first estimate for emulsion of starting amount of emulsion of 3.0% ~~and 2.0% for foaming~~.
- Aim for a minimum 25% crushing, 40% retained on #4 sieve & a minimum of 42 for FAA.

B.1 RAP Evaluation

Performing an ignition test (MnDOT Lab Manual 1853) then perform:

- a washed gradation (MnDOT lab Manual 1201, 1202 and 1203),
- sand equivalent (MnDOT Lab Manual 1215) and an
- Fine aggregate angularity (MnDOT Lab Manual 1206).

B.2 Rejuvenating Emulsion

The engineered emulsion base binder and recycling additive (RA) should be selected as to accommodate the minimum required RA dosage in such a way that the final engineered emulsion grade is regionally appropriate. The rejuvenating emulsion shall meet the emulsion quality specification supplied by the agency for engineered emulsion.

The RA can be emulsified in water to utilize as a separate component in recycling process or blended with the engineered asphalt emulsion. These methods allow the designer a wider range of flexibility in the design process to meet performance requirements. Alternatively, the RA can be blended into the base binder prior to emulsification; if following this method, the following emulsion design and RA dosage selection protocol is recommended:

- **Base Binder:** Select a base binder approximately one PG stiffer than the standard climatically appropriate emulsion residue grade.
    - Note 1: For a climate typically using a PG58-28 emulsion residue for engineered emulsions, a PG64-22 binder should be used as a base in a rejuvenating emulsion. After addition of the RA the final grade will be similar to that typically used in the climatic region.
    - Note 2: It is anticipated that use of the stiffer binder + RA will allow for an overall reduction in emulsion content in the mix compared to use of the typical softer binder grade.
- **RA Dosage:** Dosage be adjusted to achieve a 6˚C shift in the low temperature performance grade of the base asphalt.
    - Note 3: For example, if a PG64-22 has a continuous low temperature grade of -23˚C, sufficient RA dosage be used to achieve a -29˚C.
    - Note 4: For estimating purposes many bio-based RAs the impact on low temperature PG is around 1.8˚C per 1% by weight. Therefore a 6 degree shift would require approximately 6 / 1.8 = 3.3% wt. of RA. For petroleum-based RAs, the impact on low temperature PG may vary in a larger range of around 0.5-1˚C per 1% by weight.
- **RA Specification:** The RAs shall meet the properties shown below. Conformance to these properties will be confirmed by the RA supplier.

Table xx —Tests on Rejuvenating Agent

	Method	Min	Max
Tests on Rejuvenating Agent			
Viscosity, 60°C (140°F), CST	T 201	15	300
Flash Point, °C (°F)	T 48	182 (360)	
Saturates, %	ASTM D4124[a]		30
Tests on TFO or RTFO Residue			
Mass Change, %	T 179 or T 240		6.5
Viscosity Ratio	T 179 or T 240		3

a IP-469 is an acceptable replacement for measurement of % saturates.

C.1 Specific Gravity, Air Voids, Marshall and Moisture Susceptibility

Perform the mix design on crushed millings. Use ASTM C117 and C136 (dried at no greater than 40°C [104°F]) to determine the gradation of the crushed millings. Meet the requirements of the Table xx.

Table xx Requirements for Emulsion CIR Mix Design

Property	Criteria	Purpose
Compaction effort, Superpave Gyratory Compactor, 1.16° internal angle, 600 kPa stress, gyrations	30 gyrations	Density Indicator
Density, ASTM D 2726 or equivalent	Report	Compaction Indicator
Gradation for Design Millings, ASTM C117	Report	--
Marshall Stability*, ASTM D 1559 Part 5, 40°C Follow ASTM D 5581 for 150 mm, 40°C	1,250 lbs. min. (100 mm specimen) 3,000 lbs. min. (150 mm specimen)	Stability Indicator
Retained stability based on cured stability * £	70 % min.	Ability to withstand moisture damage
IDEAL CT-Index, ASTM D 8225, 25 °C* 95 mm nominal height, 150 mm diameter	200 min	Durability (cracking) Indicator
Indirect Tensile Test, AASHTO T 322, Modified	Report	Thermal Cracking
Raveling Test, Method Attached, Ambient, Modified	2% max.	Raveling Resistance
*Cured stability and CT Index determined on 60°C curing to constant weight (<72 hours). Retained stability determined after 23-hour water soak at 25°C followed by 40°C soak for one hour. £ The Tensile Strength Ratio/Retained Stability Ratio may be reduced to 0.60, provided the moisture condition indirect tensile strength or conditioned Marshall stability exceeds the minimum dry strength/stability requirement.		

Prepare: samples with a sample splitter, otherwise dry, screen and recombine millings to the target gradation. Suggested screens include: 1/2 inch, 3/8 inch, No. 4, No. 8, No. 30 and pan. Scalp oversize with a 1.0 inch screen when using 100 mm diameter compaction molds.

Mixing: Use enough material to prepare specimens ~~2.4 to 2.6 inch tall~~ of the appropriate height for testing. Use ASTM D2041 to determine the size for Rice specific gravity.

Number of specimens: ~~Four~~ Seven each for the two lower emulsion contents and ~~six~~ nine for the highest emulsion content for a total of ~~fourteen~~ 23. Two for each emulsion content will be used for long-term stability testing, and two each for moisture testing. Three specimens at each emulsion content will be used for IDEAL CT Index. Additionally, two specimens using the highest emulsion content are required for the Rice specific gravity test; back calculate for the lower emulsion contents.

Recommended emulsion contents: 1.5%, 2.0%, 2.5%, 3.0%, 3.5%, 4.0%. Choose three emulsion contents that bracket the estimated recommended emulsion content.

Add moisture that is expected to be added at the milling head, typically 1.5 to 2.5 percent.

If any additives are in the mixture, introduce the additives in a similar manner that they will be added during field production.

Mix test specimens with a mechanical bucket mixer. First mix the CIR RAP millings thoroughly with water, then mix with emulsion. Mix at ambient temperature. Mix one specimen at a time. Do not exceed 60 seconds for a mixing time.

Compaction: Compact specimens immediately after mixing. Place paper disks on the top and bottom of the specimen before compaction.

Compact specimens with a Superpave gyratory compactor (SGC) in either a 100 or 150 mm mold at 1.16° internal angle, 600 kPa ram pressure, and 30 gyrations. Do not heat the mold.

Curing after compaction: Extrude specimens from molds immediately after compaction, and carefully remove paper disks.

Place specimens in a forced draft oven with ventilation on sides and top at 140°F. Place each specimen in a small container to account for material loss from the specimens.

Dry specimens for Rice specific gravity to constant weight (less than 0.05% weight loss in 2 hours). Take care not to over-dry the specimens.

Cure compacted specimens to a constant weight but no more than 48 hours and no less than 16 hours. Constant weight is defined as less than 0.05% change in weight in 2 hours. After curing, cool specimens at ambient temperature a minimum of 12 hours and a maximum of 24 hours.

Measurements: Determine bulk specific gravity (density) of each compacted (cured and cooled) specimen according to ASTM D2726; however, record the mass of the specimen in water (measurement C) after one minute submersion.

Determine specimen heights according to ASTM D3549, or from the SGC readout.

Determine Rice (maximum theoretical) specific gravity, ASTM D2041, except as noted in this procedure, and do not break any agglomerates which will not easily reduce with a flexible spatula. It is normally necessary to perform the supplemental dry-back procedure to adjust for uncoated particles.

Determine air voids at each emulsion content.

Determine corrected Marshall Stability by ASTM D1559 at 40°C [104°F] after 2 hour temperature conditioning in a forced draft oven. Perform this testing at the same time as the moisture conditioned specimens are tested.

Determine corrected IDEAL CT Index by ASTM D 8225 at 25°C [77°F] after 2 hour temperature conditioning in a forced draft oven. Results are corrected to the actual sample height as prescribed in ASTM D 8225. Report CT Index as the average of three test results.

Moisture Susceptibility: Perform the same conditioning and volumetric measurements on moisture-conditioned specimens as on other specimens. Vacuum saturate to 55 to 75 percent, soak in a 77°F [25°C] water bath for 23 hours, followed by a one hour soak at 104°F [40°C]. Determine corrected Marshall Stability. The average moisture conditioned specimen strength divided by the average dry specimen strength is referred to as retained stability.

C.2 Thermal Cracking - Emulsion

Emulsion Content Selection: Select the minimum emulsion content that satisfies the requirements of Table 22.

Report: Report the following information: Gradation of RAP; amount and gradation of virgin aggregate or additional RAP, if any; recommended water content range as a percentage of dry RAP; optimum emulsion content as a percentage of dry RAP and corresponding density, air void level, and absorbed water; Marshall Stability and retained stability at recommended moisture and emulsion contents, raveling %, and thermal cracking initiation temperature. Include the emulsion designation, company name, plant location, and residue content.

Note: Procedure for critical cold temperature selection

Choose specification temperature using FHWA LTPPBind software (Version 2.1) and the weather station closest to the project. The required temperature for the specification is the coldest temperature at the top of the CIR layer in the pavement structure. Use 98 percent reliability.

C.3 Indirect tensile testing (IDT) Procedure - Emulsion:

Perform testing in accordance to AASHTO T 322 with the following exceptions:

a. Make specimens using the medium gradation 150 mm [6 inches] in diameter and at least 115 mm [4.5 inches] in height and compacted to air voids +/- 1 percent of design air voids at the design emulsion content. A trial specimen is suggested for this. Cure test specimens at 60ºC for no less than 48 hours and no more than 72 hours. Check specimen mass every 2 hours after 48-hour cure to check with compliance of no more than 0.05% change in mass in 2 hours. After curing, cut two specimens from each compacted specimen to 50 mm [2 inches] in height. Perform bulk specific gravity after cutting.

b. Two specimens are the minimum required at each of three temperatures.

c. Select two temperatures at 10ºC intervals that bracket the required specification. For example, if the required specification temperature is –25ºC, then select testing temperatures of –20ºC and –30ºC. A temperature of –10ºC or –40ºC should then be selected as the third required temperature.

d. Perform the tensile strength test on each specimen directly after and at the same temperature as the tensile creep test.

e. Use an environmental chamber capable of temperatures down to –40°C.

f. The critical cracking temperature is defined as the intersection of the calculated pavement thermal stress curve (derived from the creep data) and the tensile strength line (the line connecting the results of the average tensile strength at the two temperatures).

C.4 Raveling Test Procedure on Recycled Asphalt Specimens - Emulsion

For the raveling test use a modified A-120 Hobart mixer with an abrasion head (including hose) as used in the Wet Track Abrasion of Slurry Surfaces Test (ISSA TB-100). The rotation speed for the raveling test is not modified from ISSA TB-100. Remove the ring weight from the abrasion head. The weight of the abrasion head and hose in contact with the specimen should be 600 +/- 15g. Secure the prepared sample

under the abrasion head, and center for accurate result, allowing for free movement vertically of the abrasion head. The device used for securing and centering the sample must allow a minimum of 10 mm of the sample to be available for abrasion. Modify the Hobart mixer to allow the sample to fit properly for abrasion. The modification may be accomplished by adjusting the abrasion head height, or the height of the secured sample. A Raveling Test Adapter can be purchased through Precision Machine and Welding, Salina, KS, 1-877-876-9537. Reference the Hobart Model number A-120 when ordering. The C-100 and N-50 Models are not acceptable for this test procedure due to differences in size and speed of rotation.

a. Split out two recycled asphalt samples from the medium gradation, or field sample, to a quantity of 2700 g. The 2700 g is an approximate weight to give 70 +/- 5 mm of height after compaction.

b. Place the recycled asphalt sample in a container for mixing.

c. Add field or design moisture contents to each of the samples and mix for 60 seconds.

d. Add the design emulsion content to each of the samples and mix for 60 seconds.

e. Immediately place the samples into a 150 mm gyratory compaction mold and compact to 30 gyrations. If the sample height is not 70 +/- 5 mm, adjust the recycled asphalt weight.

f. After compaction, remove the samples from the compaction mold and place on a flat pan to cure at ambient lab temperature 65 – 75ºF for 4 hours +/- 5 minutes.

g. Weigh the specimen after curing, and just prior to testing.

h. Place the specimens on the raveling test apparatus. Take care that the specimen is centered and well supported. The area of the hose in contact with the specimen should not have been previously used. It is allowable to rotate the hose to an unworn section for testing. The abrasion head (with hose) shall be free to move vertically downward a minimum of 5 mm, if abrasion allows.

i. Abrade the samples for 15 minutes and immediately weigh them.

j. Determine the % raveling loss as follows: ((Wt. Prior to test – Wt. After abrasion)/Wt. Prior to test) * 100.

k. Report the average of the two specimens as the % raveling loss. There should not be a difference of 0.5% raveling loss between the two test specimens for proper precision. A difference of >0.5% will require the test to be repeated. If both of the samples have a Raveling Loss of >10% the numbers shall be averaged and the precision rule will be waived.

Note: Omit Steps b, c and d, if field mix samples are taken.