RESEARCH & DEVELOPMENT

Improving the Design of RAP and RAS Mixtures

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16. Abstract

The objectives of this project are to: (1) elucidate recycled binder contribution in reclaimed asphalt pavement (RAP) and recycled asphalt shingle (RAS) asphalt mixtures as a function of material and laboratory fabrication variables; (2) develop improved procedures for the design of RAP and RAS mixtures; and (3) develop a plan for long-term monitoring of field sections to validate the refined mixture design procedure. The recycled binder contribution in 10 laboratory produced asphalt mixtures prepared with four different recycled material sources were evaluated using a tracer-based microscopy procedure. Tracer-based microscopy investigations indicate that recycled material agglomerations exist in asphalt mixtures that prohibit complete recycled binder availability in RAP and RAS materials. The fatigue fracture surfaces of the asphalt mixtures do not contain recycled asphalt material (RAM) agglomerations, suggesting that the fracture initiates and propagates around the agglomerations. These findings suggest that the agglomerations can be considered black rocks for the purposes of mixture design. Recycled binder variability were observed among the four different RAP sources evaluated with values spanning approximately 50 to 90 percent. The recycled binder availability inferred for RAS sources was notably lower, spanning zero to 30 percent for the two sources evaluated. It was found that the RAP and virgin aggregate preheating procedure can impact the recycled binder contribution in laboratory-fabricated asphalt mixture samples and thus, it is recommended that the NCDOT specify the material preheating procedure to minimize mixture variability imparted by the laboratory fabrication procedure. The virgin binder may impact recycled binder contribution in an asphalt mixtures somewhat but additives were found to have only marginal impacts on recycled binder contribution. Findings of tracer-based microscopy analysis of asphalt mixtures informed the development of a practical method to determine recycled binder availability from RAP using sieve analysis and propose changes to volumetric mixture design procedures. Proposed changes to mixture design procedures include considering the unavailable recycled binder bound within agglomerations as part of the bulk aggregate. Additionally, the use of the RAM gradation (i.e., black curve) is proposed to better reflect the gradation of RAM in a mix compared to the recovered aggregate (i.e., white curve) given that agglomerates may act as 'black rocks'. Three of the 'control' NCDOT approved mixture designs were redesigned according to the proposed changes to volumetric mixture design procedures established through this project. The proposed revisions were found to improve asphalt mixture cracking performance without substantially impairing rutting performance. It is recommended that the laboratory findings be verified using plant-produced mixtures. A plan for long-term field validation of the proposed mixture design procedures was developed. This study was limited to the evaluation of laboratory-mixed, laboratory compacted samples.

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

EXECUTIVE SUMMARY

Reclaimed asphalt pavement (RAP) and recycled asphalt shingles (RAS) are commonly used in asphalt mixtures in North Carolina. Reclaimed and recycled asphalt materials reduce the use of virgin aggregate and binder required in the production of asphalt mixtures, yielding economic savings and environmental benefits. RAP binders are generally oxidized from time in service and thus, more susceptible to cracking than virgin binders. RAS binders are intentionally air blown and thus, more stiff and brittle than typical paving asphalts. Consequently, there is generally concern that high recycled asphalt material (RAM) content mixtures will be prone to cracking without appropriate measures to select the appropriate virgin binder grade and perform volumetric mixture design. A poor understanding of the extent to which the recycled binder acts as 'black rock' as opposed to mobilizing and blending with virgin asphalt precludes reliable mixture design procedures for RAP and RAS mixtures. The objectives of this project are to: (1) elucidate recycled binder contribution in RAP and RAS mixtures as a function of material and laboratory fabrication variables; (2) develop improved procedures for the design of RAP and RAS mixtures; and (3) develop a plan for long-term monitoring of field sections to validate the refined volumetric mixture design procedure.

The recycled binder contribution in 10 laboratory produced asphalt mixtures prepared with four different recycled material sources were evaluated using a tracer-based microscopy procedure. The mixtures evaluated encompasses four RAP sources and two RAS sources. A subset of the acquired mixtures were used to study the effects of laboratory fabrication variables and binder variables (i.e., virgin binder source, additives, and recycled binder age level) on recycled binder contribution. Findings of tracer-based microscopy analysis of asphalt mixtures informed the development of a practical method to determine recycled binder availability from RAP using sieve analysis and propose changes to volumetric mixture design procedures. Three of the 'control' NCDOT approved mixture designs were redesigned according to the proposed changes to volumetric mixture design procedures established through this project. The rutting and cracking performance of the 'control' versus redesigned mixtures was evaluated. A plan for long-term field validation of the proposed volumetric mixture design procedures was developed.

Tracer-based microscopy investigations indicate that recycled material agglomerations exist in asphalt mixtures that prohibit complete recycled binder availability in RAP and RAS materials. The fatigue fracture surfaces of the asphalt mixtures do not contain RAM agglomerations, suggesting that the fracture initiates and propagates around the agglomerations. These findings suggest that the agglomerations can be considered black rocks for the purposes of volumetric mixture design. The degree of blending was not found to vary appreciably among the asphalt mixtures evaluated.

The RAP binder contribution in asphalt mixtures do not exhibit clear trends with respect to the high-temperature grade of the RAP binder or asphalt mixture RAP content based on tracer-based microscopy investigations. However, differences in recycled binder variability were observed among the four different RAP sources evaluated with values spanning approximately 50 to 90 percent. The recycled binder availability inferred for RAS sources was notably lower, spanning zero to 30 percent for the two sources evaluated. The RAS source with the lower availability also had a higher high-temperature performance grade. It is recommended that the recycled binder availability of additional RAS sources be measured using tracer-based microscopy of asphalt mixtures to gain an improved understanding of RAS binder availability and its variation among sources. In the interim, an assumed RAS binder availability of 30 percent (i.e., the maximum value of the two sources evaluated) is recommended.

It was found that the RAP and virgin aggregate preheating procedure can impact the recycled binder contribution in laboratory-fabricated asphalt mixture samples and thus, it is recommended that the NCDOT specify the material preheating procedure to minimize mixture variability imparted by the laboratory fabrication procedure. The virgin binder may impact recycled binder contribution in an asphalt mixtures somewhat but additives were generally not found to change recycled binder contribution significantly.

The comparison of the gradation of RAP and recovered RAP aggregate provides a measure of the extent of agglomeration that exists within asphalt mixtures and in turn recycled binder availability. Tracer-based microscopy measurements were generally in good agreement with the estimations of recycled binder availability derived from the sieve analysis procedure developed in this study. The sieve analysis method requires only equipment found in a basic asphalt mixture testing laboratory as it requires neither extraction nor recovery of the asphalt binder, nor asphalt binder testing. It is recommended that the NCDOT consider implementing the sieve analysis procedure to estimate source-specific RAP binder availability for volumetric mixture design. An assumed RAP binder availability of 60 percent is recommended based on the collective results of this study if source-specific RAP binder availability is unknown.

The collective findings of tracer-based microscopy and sieve analysis were used to propose several potential changes to asphalt volumetric mixture design procedures in light of partial recycled binder availability. First, the unavailable recycled binder bound within agglomerations should be considered as part of the bulk aggregate. This change has implications to the calculation of the VMA, VFA, DP of asphalt mixtures. Additionally, the use of the RAM gradation (i.e., black curve) is proposed to better reflect the gradation of RAM in a mix compared to the recovered aggregate (i.e., white curve) given that agglomerates may act as 'black rocks'. The cracking performance improved significantly for three NCDOT approved 'control' mixtures redesigned on the basis of measured recycled binder availability to achieve an available VMA equal to the intended VMA specified in the corresponding control mixture. The redesigned mixtures contained higher virgin asphalt contents than the respective control mixtures. The permanent deformation was higher in the redesigned mixes compared to the control mixes. However, the rutting performance of the redesigned mixtures was still satisfactory, falling well below the maximum allowable APA rut depth specified by the NCDOT. Thus, the methods used to redesign mixes containing RAM proposed in this study may serve as a means to improve mixture cracking performance without substantially impairing rutting performance. This study was limited to the evaluation of laboratory-mixed, laboratory-compacted asphalt mixtures and the applicability of the findings to plant-produced asphalt mixtures merits investigation. It is also recommended that future research be dedicated to understanding the impacts of the partial recycled binder availability on the selection of an appropriate virgin binder grade and maximum permitted RBRs since considering availability lowers the effective RBR in the mixture.

1. INTRODUCTION

1.1. Overview

1.1.1. Introduction

Reclaimed asphalt pavement (RAP) and recycled asphalt shingles (RAS) are commonly used in asphalt mixtures in North Carolina. Reclaimed and recycled asphalt materials reduce the use of virgin aggregate and binder required in the production of asphalt mixtures, yielding economic savings and environmental benefits. RAP binders are generally oxidized from time in service and thus, more susceptible to cracking than virgin binders. RAS binders are intentionally air blown and thus, more stiff and brittle than typical paving asphalts. Consequently, there is generally concern that high recycled asphalt material (RAM) content mixtures will be prone to cracking without appropriate measures to select the appropriate virgin binder grade and perform volumetric mixture design. A poor understanding of the extent to which the recycled binder acts as ‘black rock’ as opposed to mobilizing and blending with virgin asphalt precludes reliable mixture design procedures for RAP and RAS mixtures.

1.1.2. Research Need Definition

There is uncertainty in the validity of the current volumetric design and the associated performance of high RAM content mixtures. The volumetric mixture design procedure currently employed by the NCDOT assumes 100 percent of the recycled binder is released from RAM and is incorporated into the virgin binder matrix. However, it is generally accepted that only a portion of the recycled binder is incorporated into the virgin binder and therefore, contributes to the mix. Consequently, the current NCDOT procedures may lead to an underestimation of the effective binder content, yielding mixtures with high cracking susceptibility. These effects may be negligible at low RAM contents but significant effects at higher contents. Also, a softer virgin asphalt than required by the climatic conditions is used in RAS and high RAP content mixtures to compensate for the stiff and brittle recycled binder. However, if only a portion of the recycled binder blends with the virgin binder, the selection of a softer virgin asphalt may lead to the production of a mixture that is more prone to rutting. Therefore, the appropriate RAP and RAS content thresholds to adjust the virgin binder grade are unclear without a solid understanding of the distribution of recycled binders in the mix. Given the use of higher RAM content mixtures in practice, an in-depth study is needed to develop an understanding of recycled binder contribution to inform the improved design of RAP and RAS mixtures.

1.1.3. Research Objectives

The objectives of this project are to:

- Elucidate recycled binder contribution in RAP and RAS mixtures as a function of material and laboratory fabrication variables;
- Develop improved procedures for the design of RAP and RAS mixtures; and
- Develop a plan for long-term monitoring of field sections to validate the refined volumetric mixture design procedure.

1.2. Summary of the Literature

A comprehensive review of the literature pertaining to this project is presented in Appendix A. A summary of most relevant components of this review is presented below.

1.2.1. Terminology

The literature contains several terms to describe the distribution of recycled binder in RAM sources and asphalt mixtures. Herein, the term recycled binder availability is considered an inherent property of a given RAM that reflects the proportion of the total recycled binder that is mobilized under typical production conditions in the absence of recycling agents. The definition of recycled binder availability herein is analogous to the term degree of activity in the literature (Lo Presti et al. 2020, Abdelaziz et al. 2021). In contrast, the term recycled binder contribution is considered an asphalt mixture property that reflects the proportion of the total recycled binder contained within the virgin binder matrix due to the recycled binder availability as well as the interaction between RAM and virgin materials, any additives, and production conditions. The term degree of blending is also considered a mixture property that measures the distribution of the available recycled binder within the virgin binder matrix (Kaseer et al. 2019).

1.2.2. RAP and RAS Considerations in Mixture Design

The majority of volumetric mixture design procedures implemented by state highway agencies, including those implemented by the NCDOT, are founded on Superpave volumetric mixture design (AASHTO R 35-17 and AASHTO M 323-17). Superpave mixture design was originally developed for virgin mixtures. The current AASHTO R 35-17 and M 323-17 standards include guidelines for incorporating recycled materials that assume 100 percent availability and are largely based on the recommendations from National Cooperative Highway Research Program (NCHRP) Projects 09-12 (McDaniel et al. 2000) and 09-43 (West et al. 2013). Many agencies have expressed concern that Superpave volumetric mixture design procedures assume complete availability with unknown implications on long-term performance (Copeland 2011). The inaccurate assumption of complete recycled binder availability has consequences, notably leading to a lower effective binder content, and therefore, lower VMA than what may be calculated. Consequently, the mixtures designed under current procedures may have insufficient virgin asphalt and lack durability. In addition, the current Superpave mix design and many agency requirements specify adjustment to a softer virgin binder performance grade when the amount of recycled materials in the mixture exceeds certain thresholds, expressed in terms of total mixture content or recycled binder replacement. The NCDOT requires a softer binder grade whenever a mix contains RAS or the recycled binder replacement ratio (RBR) exceeds 30 percent. These grade adjustment procedures were established under the assumption of 100 percent recycled binder availability and while it has been demonstrated to improve cracking resistance (McDaniel et al. 2000, West 2013), may yield softer blended binder grades than intended if only part of the recycled binder is available.

A survey of state agencies conducted in 2019 indicates that 9 out of 38 respondents assume partial availability in their mixture design procedures (Epps Martin et al. 2020[a], Abdelaziz et al. 2021). Given the lack of an accepted method to quantify recycled binder availability from RAM, these nine agencies currently use a single RAP recycled binder availability value and a single (often distinct) RAS recycled binder availability value, irrespective of the source. They adjust their volumetric mixture design procedures by either reducing credit given to recycled materials when calculating the total binder content of the mix or making an ad hoc adjustment to the virgin binder content after performing volumetric mixture design (Epps Martin et al. 2020[a]). Ad hoc adjustments to increase the asphalt content of the mixture after volumetric mixture design may improve cracking resistance but simultaneously compromise rutting susceptibility.

Balanced mix design (BMD) procedures integrate cracking and rutting tests into the mixture design process to ensure adequate performance is achieved. BMD offers a means to alleviate concerns associated with uncertainty in the effects of the assumed recycled binder availability since performance is directly quantified (Zhou et al. 2011). Four BMD approaches are outlined in AASHTO PP 105-20. Approach A constitutes the simplest and most conservative approach where performance testing is used to verify if the volumetric mixture design yields adequate performance. Approach D constitutes the most complex approach where the mix optimization is solely based on performance measures with no volumetric property requirements. The majority of implemented BMD procedures follow Approach A (NAPA 2021). Furthermore, estimates of volumetric properties typically guide establishment of the trial mixtures in the other BMD approaches. Consequently, an improved understanding of recycled binder availability may enable the design of mixtures with higher recycled contents that meet performance requirements.

A consistent laboratory procedure for handling recycled materials when preparing asphalt mixture samples for mix design and performance testing does not presently exist. Some agencies lack any specific guidance for recycled material handling while the specifications that exist vary. MDSHA (2014) specifies conditioning of RAP at 60°C for a maximum of 4 hours, and then combining it with superheated virgin aggregate to achieve the desired mixing temperature when the two are mixed. TxDOT (2016) specifies conditioning RAP at the mixing temperature for a minimum amount of time. In contrast, NYDOT (2019) attempts to limit RAP heating by specifying that RAP is dried immediately before use, batched hot, and heated at the mixing temperature for no more than one hour. RAS preheating practices also vary. The former AASHTO PP 53-09 advised adding the RAS at ambient temperature to the virgin aggregates heated slightly above the mixing temperature. TxDOT (2016) specifies heating of RAS in the same manner as RAP. Practices for mixing and compaction temperature can also vary. AASHTO M 323-17 specifies selection of mixing and compaction temperatures based on the virgin binder viscosity whereas TxDOT (2016) specifies selection based on the intended blended binder grade.

1.2.3. Measurement of Recycled Binder Contribution and Degree of Blending in Asphalt Mixtures

Several different methods have been employed to study the distribution of virgin and recycled binders within asphalt mixtures. Researchers have used measurements of the mechanical properties of asphalt mixtures to infer differences in recycled binder contribution and blending. Some of these studies have relied on attributing differences in the mechanical properties of asphalt mixture produced with the same constituent materials under different conditions (e.g., silo storage time) to differences in blending (e.g., Jacques et al. 2016, Wen and Zhang 2016). Other studies have compared the measured dynamic modulus of a mixture to that predicted from the Hirsch model coupled with the extracted and recovered asphalt binder properties, postulating that discrepancies between the model predictions and measured values can be attributed to differences in blending (Bonaquist 2007, Booshehrian et al. 2013). A limitation of the aforementioned approaches is that variation in recycled binder contribution and blending may not be the only sources of differences in the properties of the asphalt mixtures produced using varying conditions. Different production conditions can yield differences in the oxidation levels of the binders and can also impact workability of the mixture, which may contribute to differences in mechanical properties. Furthermore, uncertainty in the Hirsch model and other available models to predict the dynamic modulus of an asphalt mixture based on the constituent binder properties and volumetric compositions can be substantial (Sakhaeifar et al. 2015).

Other studies have employed microscopy techniques to evaluate the distribution of virgin and recycled binders within asphalt mixtures more directly. In conventional asphalt mixtures, virgin and recycled binders cannot be distinguished using optical or compositionally-based microscopy techniques. To overcome this challenge, several studies have used clear virgin binders (Nguyen 2009, Navaro et al. 2012, Farris 2016, Wu et al. 2018). A larger set of studies have incorporated a titanium dioxide (TiO_2) tracer into the virgin asphalt binder to distinguish it from recycled binder both visually and compositionally using Energy Dispersive X-ray Spectroscopy (EDS) Scanning Electron Microscopy (SEM) (Lee et al. 1983, Rinaldini et al. 2014, Bressi et al. 2015, Castorena et al. 2016, Jiang et al. 2018, Abdalfattah et al. 2021, Pape and Castorena 2021). TiO_2 is white and turns the virgin binder brown, which allows for visually identifying unavailable binder that appears black (e.g., Figure 1 (a)). EDS can also identify unavailable recycled binder using elemental maps. Titanium is not naturally present in asphalt whereas sulfur is present in all binders. Thus, regions with sulfur but no titanium indicate unavailable recycled binder (e.g., region below the line in Figure 1 (b)). EDS-SEM can also quantify the concentration of recycled binder within local regions of the virgin binder matrix of a mix, an in turn, calculate the recycled binder contribution and degree of blending in the mix (Jiang et al. 2018, Pape and Castorena 2021).

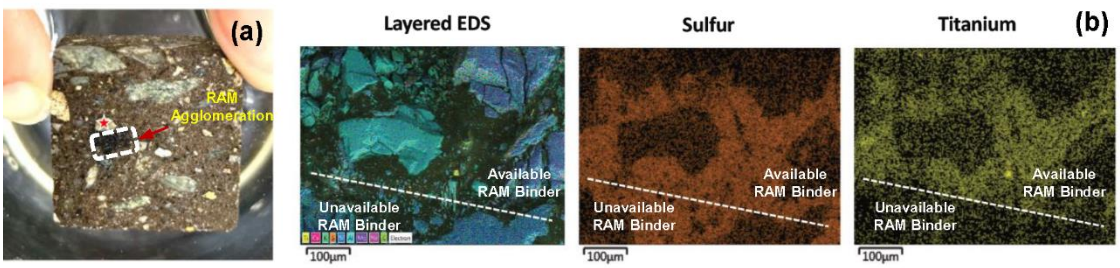

Figure 1. Example of RAP sample fabricated with titanium dioxide added to the virgin binder viewed (a) optically and (b) using EDS-SEM.

1.2.4. Measurement of Recycled Binder Availability from RAP

Recently, several methods to quantify the recycled binder availability from RAP have been proposed that utilize readily available equipment. These approaches may enable quantification of source-specific recycled binder availability within mixture design procedures. NCHRP 09-58 recently developed a size exclusion method to determine recycled binder availability (Kaseer et al. 2019, Epps Martin et al. 2020[b]). The NCHRP 09-58 method quantifies recycled binder availability based on an aggregate size exclusion method using comparative virgin and RAP mixtures. The virgin mixture is prepared using four aggregate sizes and virgin binder. The recycled mixture is prepared in the same way but with No. 4 size RAP aggregates in place of the virgin aggregates of the same size. Fabricated loose mixtures are sieved and the binder content of the No. 4 sieve-size materials are measured via ignition oven. The results are used to calculate a RAP binder availability factor (BAF). However, the use of a single size of RAP particles does not allow for assessing the impacts of RAP agglomerations that occurs over a range of particle sizes. The method also requires extensive ignition oven testing (No. 4 size RAP, No. 4 size particles sieved from the virgin mix, and No. 4 size particles from the RAP mix).

RILEM TC 264 TG 5 proposed an alternative procedure to quantify recycled binder availability that utilizes 100 percent RAP mixtures (without the addition of virgin binder) (Menegusso Pires et al. 2021). RAP is conditioned for four hours at various temperatures spanning from 70°C to 190°C, compacted, and subjected to indirect tensile strength (ITS) testing. RAP specimens with higher ITS are assumed to have higher recycled binder availability. Correspondingly, the ratio between the measured ITS at the temperature of interest and a maximum ITS assumed to coincide with 100 percent availability is reported as the degree of activity (DoA). The RILEM procedure was recently evaluated using a wide range of RAP materials from the U.S. (Abdelaziz et al. 2021, Sobieski et al. 2021). Both studies suggested the method could be used to identify the production temperature to yield maximum availability in a given RAP source. However, the studies recognized there is considerable uncertainty in defining the maximum ITS for a given RAP source given that complete availability is unlikely at any production temperature. Also, differences in ITS of a given RAP as a function of conditioning temperature can arise from sources other than availability (e.g., oxidative age level differences), potentially compromising the use of ITS ratios as a measure of availability.

1.2.5. Summary of Knowledge Gaps and Applications

The literature highlights the need for considering recycled binder availability within asphalt volumetric mixture design procedures due to its implications on the effective binder content and consequently VMA of mixtures. The majority of specifications today rely on the assumption of complete recycled binder contribution, presumably due to the lack of an accepted method to quantify recycled binder availability from RAM sources. Several candidate methods for quantifying RAP availability exist in the literature but each has limitations and has not been calibrated against recycled binder contribution measurements within asphalt mixtures. Considerably less attention has been dedicated to the quantification of recycled binder availability from RAS. There is a lack of consensus on the appropriate procedure for the laboratory fabrication of RAP and RAS mixtures. Different laboratory fabrication procedures may yield different recycled binder contribution and thus, impact the design and evaluation of RAP and RAS mixtures. Consequently, there is a need for an in-depth study to elucidate how material and fabrication variables affect recycled binder contribution in asphalt mixtures to improve the virgin binder grade selection, laboratory mixture fabrication, and the volumetric design of RAP and RAS mixtures. Tracer-based microscopy offers a research tool to quantify recycled binder contribution in asphalt mixtures. Measurements of recycled binder contribution in asphalt mixtures serve as a reference to evaluate and calibrate a simple, implementable method to quantify recycled binder availability.

1.3. Organization of the Report

This report is composed of eight primary sections and two appendices. Section 1 presents the needs, objectives, and summarizes the most relevant literature (see Appendix A for the full literature review). Section 2 describes the research methodology, including the materials evaluated, methods to elucidate recycled binder contribution and blending in asphalt mixtures and recycled binder availability directly from RAP, and experiments conducted to evaluate the impacts of incorporating recycled binder availability into volumetric mixture design procedures. Section 3 presents the results and findings of the experiments. Section 4 summarizes the proposed changes to volumetric mixture design procedures based on the research findings. Section 5 presents the plan for long-term monitoring of field sections to validate the proposed revisions to volumetric mixture design procedures. Section 6 presents the conclusions and recommendations for future research. Section 7 lists the references cited in the main body of the report. Appendices A provides the detailed literature review. Appendix B summarizes experiments conducted in an effort to quantify the rate of diffusion between recycled and virgin binders. Appendix C provides a detailed example of the calculation of asphalt mixture volumetric properties when accounting for recycled binder availability in accordance with the proposed revisions to asphalt volumetric mixture design procedures.

2. METHODOLOGY

2.1. Mixture Designs Evaluated

Table 1 details the component materials from four suppliers corresponding to 2018 9.5-mm Nominal Maximum Aggregate Size (NMAS) surface mixture designs approved by the NCDOT that were evaluated in this study. The suppliers are designated A through D to preserve anonymity. Three NCDOT approved mixture designs were evaluated from each of Sources B and C. The three mixture designs from a given source contained the same virgin and RAP stockpiles but differed in terms of recycled material content and whether RAS was included in the case of Source B. Three mixture designs corresponding to the Source A materials were also evaluated. The A25/4 mixture design was approved by the NCDOT. The research team prepared the A29 and A45 mixture designs in the laboratory in accordance with requirements for RS9.5C mixtures using the same RAP and virgin material stockpiles included in the A25/4 mixture. The mix design variations from Sources A through C were included to evaluate the impacts of RAP content and recycled material source on recycled binder contribution. A single mixture design was obtained and evaluated from Source D. A subset of the acquired mixtures were used to study the effects of laboratory fabrication variables and binder variables (i.e., virgin binder source, additives, and recycled binder age level) on recycled binder contribution. Findings of tracer-based microscopy analysis of asphalt mixtures informed the development of a practical method to determine recycled binder availability from RAP using sieve analysis and propose changes to volumetric mixture design procedures. Three of the 'control' NCDOT approved mixture designs were redesigned according to the proposed changes to volumetric mixture design procedures established through this project, which are also indicated in Table 1. The rutting and cracking performance of the 'control' versus redesigned mixtures was evaluated. All asphalt mixture samples evaluated were laboratory-mixed and laboratory-compacted.

Table 1. Summary of the Mixtures Evaluated

Source	A			B			C			D
Mixture ID	A25/4	A29	A45	B21	B30	B15/5	C40	C30	C15	D30
NCDOT Designation	RS9.5C	--	--	RS9.5B	RS9.5C	RS9.5B	RS9.5B	RS9.5B	RS9.5B	RS9.5B
RAP (%)	25	29	45	21	30	15	40	30	15	30
RAS (%)	4	0	0	0	0	5	0	0	0	0
RBR (%)	29	18	28	16	22	30	36	27	13	24
Virgin PG	58-28	58-28	58-28	64-22	64-22	58-28	58-28	58-28	64-22	64-22
Mix Redesign	✓					✓	✓			

2.2. Recycled Material Stockpile Characterization

All RAP and RAS materials were spread in pans and dried in a forced-draft oven at a warm temperature (60°C) until constant mass before characterization or use in asphalt mixtures. The binder content of each RAM source was measured by ignition oven, following AASHTO T 308. RAP and RAS binders were extracted and recovered following ASTM D2172 and ASTM D5404, then subjected to high-temperature grading following AASHTO T 315 and AASHTO M 320. The theoretical maximum specific gravity was measured following AASHTO T 209. The effective specific gravity (G_{se}) was calculated from the theoretical maximum specific gravity of the RAM materials, using the measured binder content an assumed binder specific gravity of 1.02. In addition, sieve analysis was conducted to obtain the gradation of both the RAM (commonly referred to as the ‘black curve’) and recovered RAM aggregate (commonly referred to as the ‘white curve’). To accomplish this, dried RAM samples were washed according to AASHTO T 11-20. The washed samples were then dried, and subsequently subjected to sieve analysis according to AASHTO T 27-20. The RAM was collected from each sieve and ignited according to AASHTO T 308-18. The recovered aggregate was collected and a washed sieve analysis was performed, incorporating the dust lost during the first washing.

The RAM asphalt contents and high-temperature grades are detailed in Table 2. The selected sources encompass a range in recycled binder properties and asphalt contents. All sources contained siliceous, crushed aggregate.

Table 2. RAM Stockpile Properties

Mix	Material	RAM binder content (%)	Continuous High PG (°C)
A	RAP	4.0	96.9
	RAS	19.0	194
B	RAP	5.0	92.9
	RAS	22.0	143
C	RAP	5.7	91.0
D	RAP	4.8	102.8

2.3. Measurement of Recycled Binder Contribution in Asphalt Mixtures

2.3.1. Mixture Conditions Evaluated

Tracer-based microscopy was conducted to evaluate recycled binder contribution in asphalt mixtures. Tracer-based microscopy was used to evaluate the effects of the following variables on recycled binder contribution in asphalt mixtures:

- RAM type, source, and content
- Bulk specimen versus fatigue fracture surface
- Laboratory fabrication procedure
- Binder considerations, including the virgin binder source, inclusion of an antistrip additive, inclusion of recycling agents, inclusion of an extender, and the age level of the RAP

Table 3 summarizes the specimens fabricated for tracer-based microscopy analysis. The different rows below the mixture designations detail the different specimen fabrication procedures and material variables evaluated. Further details on the mixture fabrication procedures are provided in subsequent sections. Specimens of each study mixture were fabricated using what is referred to as the ‘local contractor fabrication’ procedure to study the effects of material variables on recycled binder contribution. As previously discussed, guidance on the preheating and mixing of RAP with virgin materials is lacking in current specifications. The most commonly employed procedure locally coincides with the ‘local contractor fabrication’ procedure, which was identified through personal communication with local laboratories. In the ‘local contractor fabrication’ procedure, the virgin aggregate was preheated to the target mixing temperature plus 10°C and the virgin binder was preheated to the mixing temperature. The preheated virgin aggregate was removed from the oven and combined with ambient temperature, dried recycled materials. The virgin aggregate and recycled materials were agitated and returned to the virgin aggregate oven for 45 minutes. Subsequently, the virgin aggregate-recycled material blend was removed from the oven and mixed with the virgin binder. All mixtures were short-term aged according to AASHTO R 30 prior to compaction. All microscopy specimens were extracted from gyratory-compacted samples via sawing to generate ‘bulk’ specimens representative of the bulk mixture with the exception of specimens indicated in the ‘fracture surface’ row of Table 3. The ‘fracture surface’ specimens correspond to the fatigue fracture surface of an asphalt mixture specimen generated by conducting laboratory fatigue testing of an asphalt mixture sample to failure. These specimens were used to study the distribution of recycled and virgin binders along with fracture surface of asphalt mixtures and compare findings to the bulk mixture. Additional details pertaining to the fabrication of bulk and fracture surface specimens are provided in Sections 2.3.3 and 2.3.4.

To study the effects of binder variables on recycled binder contribution in asphalt mixtures, select mixtures were prepared using an alternate virgin binder, with the addition of an antistrip additive, with the addition of recycling agents, and/or with the addition of an extender. The alternate virgin binder is a PG 64-22 whereas the specified mixture included a PG 58-28 virgin binder. The PG 64-22 binder was also used to prepare the mixtures with recycling agents. The recycling agents are designated RA1 and RA2 and the extender is designated E in Table 3 to preserve supplier anonymity. In addition, the Source C RAP was aged in an oven at 95°C for 4 days to simulate a harsher aging state prior to fabricating a C40 mixture; this specimen was compared to an analogous C40 mixture sample prepared without prior aging of the RAP to evaluate the impact of RAP age level on recycled binder contribution. All specimens prepared to evaluate binder variables were fabricated using the ‘local contractor fabrication’ procedure.

Select mixtures were also produced using alternative material preheating procedures to evaluate the effects of laboratory fabrication variables on recycled binder contribution. The alternative procedures evaluated are detailed in Table 4. The NCHRP 09-12 procedure coincides with the recommendations by McDaniel et al. (2000) and involves preheating the RAM at 110°C for two hours and then combining with virgin aggregate conditioned slightly higher than the mixing temperature. Virgin binder preheated to the mixing temperature is subsequently added. The Superheat procedure was included to better mimic plant preheating procedures and involves superheating the virgin aggregate and combining with ambient temperature RAP; subsequently, virgin binder conditioned to the mixing temperature is added.

Table 3. Summary of Tracer-based Microscopy Specimens Evaluated

Source	A			B			C			D
Mixture	**A25/4**	**B29**	**B45**	**B21**	**B30**	**B15/5**	**C40**	**C30**	**C15**	**D30**
Local Contractor	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Fracture surface[1]	✓				✓	✓	✓	✓		
NCHRP 9-12 fabrication	✓			✓			✓			✓
Superheat fabrication – Aggregate Temp = 240ºC	✓			✓			✓			✓
Superheat fabrication – Aggregate Temp = 200ºC				✓						
Aged RAP[1]							✓			
Alternate Binder[1]							✓			
Virgin Binder + Antistrip[1]							✓			
Alternate Binder + RA1[1]	✓						✓			
Alternate Binder + RA2[1]	✓									
Alternate Binder + E[1]							✓			

[1] Fabricated using the local contractor fabrication method.

Table 4. Material Preheating Procedures

Method	Virgin Aggregate Temperature	Recycled Material Preheating
Local contractor	Mixing Temperature + 10°C	Add to preheated aggregate and condition at mixing Temperature + 10°C for 45 min
NCHRP 09-12	Mixing Temperature + 10°C	Precondition at 110°C for two hours
Superheat	Either 240°C or 200°C	None

2.3.2. Preparation of Tracer-modified Virgin Binder and Binder Samples for EDS-SEM Analysis

Tracer-modified virgin binders were prepared by mixing with titanium dioxide tracer micro particles using high shear mixing; the average tracer particle size was 0.2 microns in accordance with the recommendations by Pape and Castorena (2021). The tracer was added at a rate of 10 percent of the virgin binder mass, which equates to less than five percent by volume of virgin binder. A small portion of the tracer-modified virgin binder was transferred into 25-mm diameter silicone molds; the remainder was used to prepare asphalt mixture samples. Binders from the RAP and RAS sources were extracted and recovered according to ASTM D2172 and ASTM

D5404. Recovered binders were also placed in silicone molds. Both recycled and virgin binder samples were stored in a freezer prior to testing. EDS analysis of the binder samples was used to measure the titanium concentration of the virgin binder and sulfur contents of the virgin and recycled binders, which is required for inference of the recycled binder contribution using mixture microscopy measurements.

2.3.3. Bulk Mixture Specimen Fabrication

To prepare bulk specimens, laboratory-mixed loose mixture samples were prepared and short-term aged according to AASHTO R 30. Loose mix samples were compacted with a Superpave gyratory compactor using the design number of gyrations, with a target height of 115 mm. The gyratory samples were sawn to produce 25x25x12 mm prisms for EDS analysis, following the same procedure as Castorena et al. (2016). The specimens were polished using the same procedure as the fracture surface specimens, as detailed in Pape and Castorena (2021).

2.3.4. Fatigue Fracture Specimen Fabrication

To observe and evaluate the distribution of recycled and virgin materials along the fracture surface of asphalt mixtures, small specimen geometry asphalt mixture performance test specimens were fabricated from Superpave gyratory-compacted samples made with tracer-modified virgin binder, following AASHTO PP 99-19. Figure 2 depicts the process used to prepare specimens for fracture surface analysis. Asphalt Mixture Performance Tester (AMPT) cyclic fatigue testing was conducted following AASHTO TP 133-19, and the sample was completely separated into two halves after testing, as shown in Figure 2 (a). The specimen halves were sawn off the loading platens and gauge points were removed, as shown in Figure 2 (b). Then, the specimen halves were embedded in clear epoxy resin to stabilize the failure surface, as shown in Figure 2 (c). After the resin cured, the failure surface was sliced parallel to the axis of the specimen to develop flat cross-sectional samples for EDS analysis, as shown in Figure 2 (d). The flat samples were then polished and prepared for EDS analysis, following the procedure for mixture specimen preparation procedure outlined in Pape and Castorena (2021). Analysis was conducted along the interface between the epoxy resin and the fracture surface.

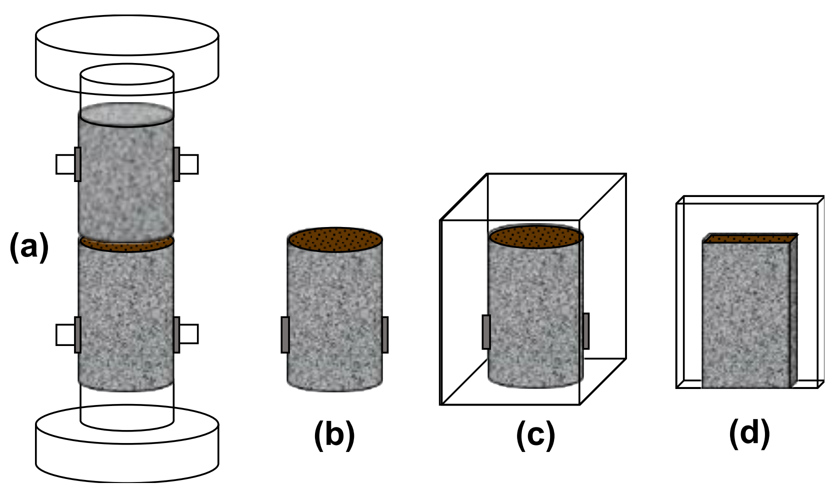

Figure 2. Process to prepare EDS specimen from an AMPT cyclic fatigue test specimen.

2.3.5. Tracer-based Microcopy Analysis

Microscopy was conducted in a Hitachi S3200N VPSEM outfitted with an Oxford X-Max silicon drift detector. Using a variable pressure SEM (VPSEM) eliminates the need for sample coating, thus increasing efficiency. EDS mapping of elemental composition was conducted at least 10 sites for each mixture, as was recommended in Pape and Castorena (2021).

2.3.6. Interpretation of the Results

2.3.6.1. Visual Interpretation using Optical Inspection and EDS Maps

Optical observations and backscattered electron images were used to make inferences about the composition of bulk specimens and fracture surfaces and guide selection of the locations for quantitative EDS analysis. Titanium dioxide is white and turns the virgin binder brown, allowing for visual identification of unavailable recycled binder, which appears black. SEM analysis yields backscattered electron images, which depict sample compositional contrast based on relative atomic weight. In backscattered electron images, asphalt binder appears darker than aggregate due its constituents having lower atomic weight. However, electron images cannot distinguish virgin and recycled binders within a mixture. Electron images were used to select areas rich in asphalt binder for EDS analysis. EDS analysis generates maps that depict the presence and absence of elements of interest, which can give visual feedback about the composition of an area under investigation. Titanium is not naturally present in asphalt whereas sulfur exists in all asphalt binders. Thus, regions with sulfur but no titanium signify unavailable recycled binder. Elemental maps obtained using EDS also can identify unavailable binder at smaller length scales and without the complexity of interference of aggregate color contrast that exists when making optical inferences.

Figure 3 shows an illustrative schematic of optical, SEM, and EDS images of an asphalt mixture specimen prepared with tracer-modified virgin binder; markers are placed on certain aggregates to call attention to specific regions. The backscattered electron and optical images both delineate binder from aggregate; aggregates appear grey in both. Aggregate 1 is embedded in a region of unavailable recycled binder as evident by the optical image where the binder is visually black and EDS maps which show sulfur is present near the Aggregate 1 but titanium is absent. Partial mixing with the virgin binder (visually brown) is observed near Aggregate 2 based on the gradient in color and titanium intensity in the vicinity of Aggregate 2. Aggregate 3 is coated in unblended recycled binder based on the black film surrounding the particle in the optical image and lack of titanium where sulfur is present. Aggregate 4 is contained in an agglomeration of adhered recycled aggregates containing unavailable recycled binder as evident by the black binder surrounding the particles based on the black binder surrounding the adjacent aggregates in the optical image and presence of sulfur but no titanium in the same region.

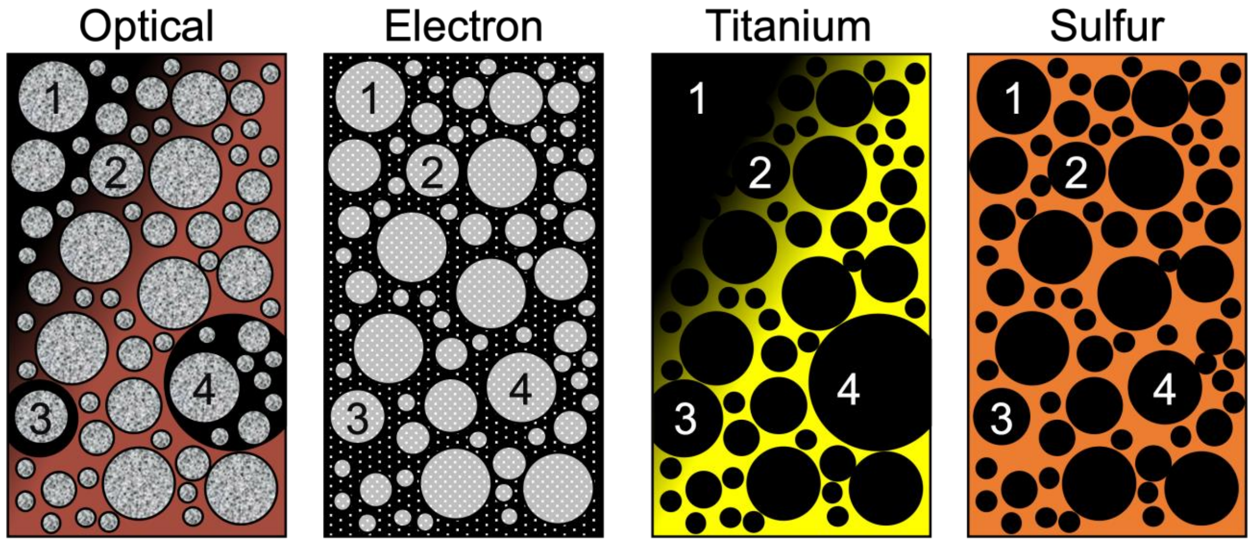

Figure 3. Illustration of optical appearance compared to SEM imaging and EDS maps of asphalt mixture.

In this study, optical observations and EDS maps were used to identify the presence of recycled material agglomerations. The optical image is not available while specimens are in the SEM, so the backscattered electron image was used to guide the selection of sites for EDS analysis. Fracture surface analysis was conducted along the boundary between epoxy and the edge of the specimen. Bulk specimen imaging was conducted in areas with visibly high amounts of asphalt binder.

2.3.6.2. Quantitative Assessment of Recycled Binder Contribution

In addition to generating maps of elemental composition, EDS was used to quantify the relative mass concentrations of elements present on localized sample areas of asphalt binders and mixtures identified using backscattered electron images. Equation (1) is used to quantify the local recycled binder contribution in asphalt mixtures using EDS measurements, which requires: (1) local measurements of titanium (Ti) and sulfur (S) contents in an asphalt mixture sample, (2) measurements of the sulfur contents of the constituent binders, (3) the titanium concentration of the tracer-modified virgin binder, and (4) the proportions of virgin and recycled binders in the mix. Equation (1) is a modified version of the blending ratio equation originally proposed by Jiang et al. (2018).

$$\text{RBC (\%)} = \left(\frac{Virgin_{Ti:S}}{Mix_{Ti:S}} - 1\right) \times \frac{AC - RAP_{AC} - RAS_{AC}}{RAP_{AC} + RAS_{AC}} \times \frac{S_V}{S_R} \times 100\% \qquad (1)$$

where: Recycled Binder Contribution (%) = actual recycled binder concentration within the virgin binder matrix divided by the concentration expected under the condition of complete availability; $Virgin_{Ti:S}$ = titanium to sulfur ratio in the virgin binder; $Mix_{Ti:S}$ = titanium to sulfur ratio of mix sample in the area of interest; AC = mix total asphalt content; RAP_{AC} = RAP binder content of the mix (i.e., mass of RAP binder/total mass of mix); RAS_{AC} = RAS binder content of the mix; S_V = sulfur content of the virgin binder; and S_R = sulfur content of the recycled binder blend.

Equation (1) assesses the local recycled binder contribution by assessing the amount of recycled binder present in the image using the Ti:S ratios, then comparing it to the theoretical level of the perfect availability and blending scenario calculated using mixture volumetric parameters. A local binder contribution value of 50 percent indicates that the concentration of recycled binder in the location of interest is half of expected value based on the proportion of recycled to total binder in the mix (i.e., the condition corresponding to complete availability and blending). This normalization based on the total proportion of recycled binder to virgin binder in the mix facilitates comparisons between mixtures with differing recycled material contents, recycled binder contents and recycled material types.

The average local recycled binder contribution result of a given mixture is reported as the overall mixture recycled binder contribution. Variation in local binder contribution measurements within a given mixture provides a measure of the degree of blending.

The tracer-based microscopy recycled binder contribution results of mixtures containing both RAP and RAS include the combined contributions from the RAP and RAS. The overall mixture recycled binder contribution, expressed in terms of the individual contributions from the RAP and RAS availabilities, is shown in Equation (2). Herein, the RAP and RAS binder contents in the mix and the RAP binder availability were input in Equation (2) and the RAS binder availability value (i.e., $Availability_{RAS}$) that yields the recycled binder contribution value from tracer-based microscopy was calculated and reported as the RAS binder availability.

$$\text{Recycled Binder Contribution}(\%) = \frac{RAP_{AC} \times Availability_{RAP} + RAS_{AC} \times Availability_{RAS}}{RAP_{AC} + RAS_{AC}} \times 100\% \qquad (2)$$

where RAP_{AC} = RAP binder content of the mix (i.e., mass of RAP binder/total mass of mix); RAS_{AC} = RAS binder content of the mix; $Availability_{RAP}$ = RAP binder availability; and $Availability_{RAS}$ = RAS binder availability.

2.3.6.2. Statistical Analysis

Statistical tests of difference and equivalence were used to assess the significance of experimental factors on recycled binder contribution result in asphalt mixtures. To assess the significance of the factors, the mean differences in tracer-based microscopy recycled binder contribution results were compared.

Two-sample t-tests were used to identify statistically significant differences. Statistical tests of equivalence tests were also conducted to enable practical considerations. The hypotheses used in difference and equivalence statistical tests are summarized in Table 5. Equivalence tests identify whether or not mean differences fall outside of a maximum acceptable difference given practical considerations (Δ) using Two One-Sided t-Tests (TOST). TOST consists of two, one-sided t-test to assess whether or not the difference between two means falls below a lower limit ($-\Delta_L$) and/or exceeds an upper limit (Δ_U, where $\Delta_U + \Delta_L = \Delta$) given a defined confidence level. If either condition falls outside of the limit, the two samples are considered not equivalent. The definition of practically acceptable differences was guided by the sensitivity of the calculated VMA to the assumed recycled binder availability. The sensitivity of the mixture VMA to the assumed availability was evaluated for all study mixtures. The C40 mixture exhibited the greatest sensitivity to the assumed availability. The maximum Δ value that would not result in a difference in the inferred VMA that falls outside of established quality control limits (i.e., a one percent difference in VMA) was found to be 20 percent. Therefore, a mean recycled binder contribution difference of 20 percent was used as the Δ in equivalence tests. A confidence level of 90 percent was used for the statistical tests of difference whereas an 80 percent confidence level (coinciding with a 90 percent confidence level for each one-sided t-test) was used for the equivalence tests. Results were considered equivalent if equivalence tests indicated the results were equivalent and difference tests indicated the results were not different.

Table 5. Null (*H_0*) and Alternate (*H_1*) Hypotheses in Difference and Equivalence Statistical Tests

Difference Test	Equivalence Test
$H_0 : \mu_1 \neq \mu_2$: The mean results are different	$H_0 :\| \mu_1 - \mu_2 \| > \Delta$: The difference in mean results is greater than the equivalence acceptance criterion
$H_1 : \mu_1 = \mu_2$: The mean results are the same	$H_1 :\| \mu_1 - \mu_2 \| \leq \Delta$: The mean difference is less than or equal to the equivalence acceptance criterion

2.4. Measurement of RAP Recycled Binder Availability using Sieve Analysis and Ignition Oven Testing

The tracer-based microscopy results of laboratory-produced asphalt mixtures (discussed in detail within Section 3) indicate that agglomerations of adhered RAM particles are the primary inhibitor of recycled binder availability in asphalt mixtures. It is hypothesized that the majority of agglomerations are present in the RAM prior to asphalt mixture production. Assuming that RAM agglomerations that do not break down due to the agitation of sieving will also persist in the asphalt mixture, it follows that the difference in the particle size distribution of RAM and recovered RAM aggregate provides a measure of the extent of agglomeration, and therefore, recycled binder availability. Accordingly, a practical method to determine recycled binder availability from RAP was developed.

Within the method, the proportion of RAP binder that is bound within agglomerations is estimated using the gradation of recovered RAP aggregate (termed the ‘white curve’), gradation of the RAP itself (termed the ‘black curve’), RAP aggregate specific gravity, and RAP binder content. To obtain the required gradations, dried RAP samples were washed according to AASHTO T 11-20. The washed samples were then dried, and subsequently subjected to sieve analysis according to AASHTO T 27-20. The RAP was collected from each sieve and ignited according to AASHTO T 308-18. The recovered aggregate was collected and a washed sieve analysis was performed, incorporating the dust lost during the first washing.

Sieve analysis of RAP demonstrates minimal aggregate passing the No. 200 sieve, suggesting that all RAP aggregates maintain a mastic coating. Peripheral mastic coatings in the RAP are assumed to be available to contact and blend with virgin asphalt whereas the mastic bound within RAP agglomerations is considered unavailable, as shown schematically in Figure 4.

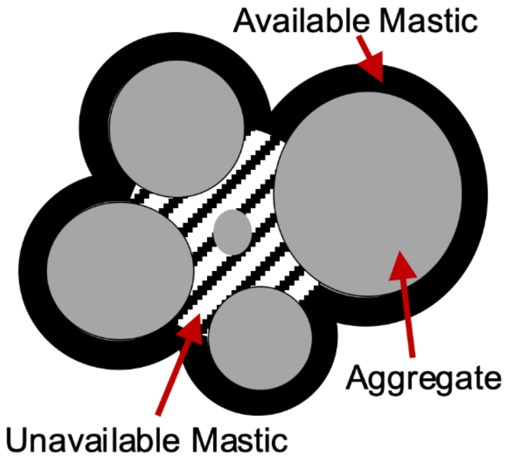

Figure 4. RAP agglomeration.

Correspondingly, the RAP gradation measurements, asphalt content, and effective specific gravity results are used to calculate the recycled binder availability using several steps. First, the total volume of mastic in a sample of RAP containing 100 g of aggregate is calculated using Equation (3).

$$V_{mastic} = V_b + V_{filler} = \frac{P_b\left(1+\frac{P_b}{(100-P_b)}\right)}{G_b} + \frac{P_{200}}{G_{se}} \quad (3)$$

Where: V_{mastic} = volume of mastic in mix with 100 g of aggregate (cm^3); V_b = binder volume (cm^3); V_{filler} = volume of filler (cm^3); P_b = total binder content; G_b = binder specific gravity; G_{se} = effective aggregate specific gravity; and P200 = percent passing the No. 200 (0.075 mm) sieve for the recovered aggregate.

Next, the average mastic film thickness in the RAP, t, is calculated via optimization to minimize the absolute difference between the volume of mastic calculated using Equation (4) and the total known volume of mastic calculated using Equation (3). Equation (4) computes the volume of mastic in the RAP by assuming spherical aggregate particles are coated in a concentric shell of mastic with uniform thickness equal to t. Underwood and Kim (2013) showed that Equation (4) reasonably reflects the distribution of mastic within asphalt mixtures based on microscopic measurements of the mastic film thickness within asphalt mixture samples.

$$V_{mastic} = \sum N_i \times V_i = \sum \frac{P_{i+1}-P_i}{G_{se}\times\rho_{water}\times \pi/6 \times\left((d_{i+1}+d_i)/2\right)^3} \times \frac{\pi}{6}\left[\left(\frac{d_{i+1}+d_i}{2}+2t\right)^3 - \left(\frac{d_{i+1}+d_i}{2}\right)^3\right] \quad (4)$$

where: V_{mastic} = volume of mastic in mix with 100 g of aggregate (cm^3); N_i = number of particles of size i; V_i = volume of mastic coating aggregate of size i (cm^3); P_i = recovered aggregate percent passing sieve size i; and d_i = sieve size (mm).

Subsequently, the RAP gradation and calculated t are used to calculate the volume of peripheral (i.e., available) mastic coating the RAP particles using Equation (5). Equation (5) resembles Equation (4) but utilizes the RAP gradation instead of the recovered aggregate gradation. Also, an adjustment to the particle diameter corresponding to each sieve size is made to account for the peripheral mastic film present on the RAP particles (i.e., particle size = $d_i - 2t$).

$$V_{available\ mastic} = \sum N_i \times V_i = \sum \frac{RP_{i+1} - RP_i}{G_{se} \times \rho_{water} \times \pi/6 \times \left(\left(d_{i+1} + d_i \right)/2 - 2t \right)^3} \times \frac{\pi}{6} \left[\left(\frac{d_{i+1} + d_i}{2} \right)^3 - \left(\frac{d_{i+1} + d_i}{2} - 2t \right)^3 \right] \qquad (5)$$

where: $V_{available\ mastic}$ = volume of available mastic in mix with 100 g of aggregate (cm^3); and RP_i = RAP percent passing sieve size i.

Lastly, the RAP binder availability is calculated using Equation (6). The filler content of the mastic is assumed to be consistent within the available and unavailable mastic. Thus, the ratio of available to total mastic volume provides the RAP binder availability.

$$Availability = \frac{V_{available\ mastic}}{V_{mastic}} \times 100\% = \frac{V_{available\ binder}}{V_{binder}} \times 100\% \qquad (6)$$

where: $V_{available\ binder}$ = volume of available binder in the mastic; and V_{binder} = volume of total binder in the mastic.

Other researchers have also suggested that differences between black and white curves may be related to recycled binder availability (Al-Qadi et al. 2009, Roque et al. 2015, Xu et al. 2019, Zhu et al. 2020, Abdelaziz et al. 2021, Sobieski et al. 2021), but have not formalized this hypothesis into a method and/or validated their method against recycled binder contribution measurements.

2.4.1. Assumptions and Discussion

Equation (4) assumes spherical aggregate particles are coated in a concentric shell of mastic that has uniform thickness, which does not fully reflect the complex composition of asphalt mixtures. However, Equation (4) may still allow for reasonably accurate estimation of the proportion of recycled binder bound in agglomerations. Also, asphalt mastic film thickness calculations circumvent the major source of uncertainty in asphalt binder film thickness calculations, which is the unknown gradation of the material passing the No. 200 sieve, which in turn constitutes the aggregate fraction with the highest specific surface area (Radivosky 2003).

The asphalt mastic is generally considered the combination of effective binder and mineral filler (Underwood and Kim 2013). If this definition is maintained, then it follows that the bulk RAP aggregate specific gravity is used instead of the effective specific gravity within Equation (4) and Equation (5) and that the volume of total mastic for a 100 g sample of aggregate is calculated using the effective rather than total asphalt binder content. RAP aggregate bulk specific gravity is required for current volumetric mixture design procedures specified in AASHTO R 35 and AASHTO M 332. However, there is presently uncertainty in the bulk specific gravity values reported for RAP materials. Intuitively, the G_{sb} of RAP aggregate should be easy to measure by recovering the RAP aggregate using the ignition oven or solvent extraction. However, solvent extraction can leave trace amounts of residual binder on the aggregates which confounds the results (Copeland 2011). In addition, aggregate recovered after conducting ignition oven testing has been found to produce unreliable results, which may be due to the high dust content of processed RAP that can coat the coarser fine aggregates and pose challenges with G_{sb} testing (Copeland 2011). Consequently, the majority of agencies use measurements of RAP theoretical maximum specific gravity (G_{mm}) obtained using AASHTO T 209 rather than relying on measurements of recovered aggregate bulk specific gravity (Copeland 2011). When combined with the asphalt content of the RAP, G_{mm} measurements can be used to calculate the RAP aggregate effective specific gravity (G_{se}). Agencies use the calculated G_{se} combined with an assumed absorption value based on typical local values to estimate the G_{sb} of RAP aggregate (Copeland 2011). Given the uncertainty in RAP aggregate G_{sb} measurements, and in turn, effective asphalt contents, the sensitivity of the aforementioned scheme to calculate RAP binder availability to the assumed absorption of the RAP aggregate merits evaluation. Consequently, two procedures were applied to estimate the RAP binder availability using sieve analysis to evaluate the maximum expected sensitivity to the chosen absorption:

1. Using the RAP aggregate G_{se} and correspondingly, assumed absorption of zero (i.e., assumption that the mastic includes all binder within the mix).
2. Using the measured G_{se} combined with an assumed absorption of three percent, constituting a value near the upper limit expected in practice, to obtain G_{sb} and P_{be} for use in place of G_{se} and P_b in the above equations.

Note that the RAP sources evaluated in this study were all sourced from locations that contain aggregate with low absorption so scenario one more closely reflects the G_{sb} of these sources.

The sieve analysis method established herein was not found to be directly applicable to RAS, which has a much higher P200 content than RAP and thus, likely negating the assumption that all particles maintain a mastic coating. In RAS materials, agglomerates of P200 material alone are likely.

2.5. Measurement of Recycled Binder Diffusion

Past studies have suggested that diffusion is an important component of blending in RAM mixtures and correspondingly, several studies have proposed various Dynamic Shear Rheometer (DSR)-based procedures to measure diffusion between recycled and virgin binders but noted experimental challenges. DSR-based experiments were tried quantify the rate of diffusion between two binders using the DSR. In these experiments, aged and virgin binder wafers were conditioned in contact. Oscillatory loading was applied to monitor the time-dependent response of the wafer system and infer blending. Experiments where samples were conditioned within the DSR and external to the DSR were tried. When samples were conditioned within the DSR at hot-mix asphalt production temperatures, poor stress waveform quality precluded the application of oscillatory loading in the DSR using sufficiently low strain amplitudes to prevent mechanical mixing. The use of relatively large, 50-mm diameter, samples did not alleviate the data quality limitations. When samples were conditioned outside of the DSR, mechanical mixing was induced when the samples were transferred from the conditioning chamber to the DSR, which compromised measurements. In the absence of mechanical mixing, time-dependent blending between binders specimens conditioned in contact in the DSR at 120°C was not observed, suggesting diffusion was minimal. Based on the aforementioned observations, the diffusion rate between RAP and virgin binders could not be quantified using DSR experiments. Based on the lack of observed diffusion in the absence of mechanical mixing, it is inferred that mechanical mixing rather than diffusion is dominant in asphalt mixtures, which supports the findings that agglomerations of adhered RAM particles drive recycled binder contribution in asphalt mixtures rather than partial diffusion of binders in contact. Details pertaining to the diffusion experiments and results are presented in Appendix B.

2.6. Redesign of Current NCDOT Asphalt Mixtures on the Basis of Availability

The collective findings of the tracer-based microscopy investigations and sieve analysis informed several proposed changes to volumetric mixture design proposed that are discussed in detail within Section 3. Two of the proposed changes include: (1) the use of the RAM gradation (i.e., black curve) rather than the recovered aggregate gradation (i.e., white curve) when designing the aggregate gradation of an asphalt mixture and (2) including the unavailable recycled binder as part of the bulk aggregate volume. Including the unavailable recycled binder in the bulk aggregate volume instead of the binder phase lowers the calculated VMA for a given asphalt mixture. This implies that current designs assuming 100 percent availability may yield lower actual VMAs than the current calculations suggest. Considering availability also impacts the calculation of the voids filled with asphalt (VFA) and dust-to-binder ratio (DR).

The impacts of these changes were assessed by redesigning three of the NCDOT approved 'control' mixtures that were designed on the basis of availability in light of these proposed changes. The redesigns were prepared to achieve the intended effective binder content, and therefore VMA, specified in the control mixture design when accounting for availability. The redesigned mixtures contained the same total amount of RAM materials as a proportion of the total aggregate content as the corresponding control mixture. When redesigning the control mixtures, the original RAP and RAS stockpile proportions and virgin binder in the control mix were maintained and the virgin stockpile proportions were adjusted in an effort to achieve an available VMA equal to the specified VMA (and thus, intended effective binder content) in control mixture. The specified VMA corresponds to the VMA calculated assuming 100 percent availability whereas the available VMA corresponds to the VMA calculated when considering the unavailable recycled binder as part of the bulk aggregate volume. The RAM availabilities were inferred from the results of previous tasks. The revised gradation met NCDOT gradation specifications when using the gradation of the RAM rather than the recovered aggregate. Samples were prepared at four asphalt contents using the refined virgin stockpile proportions to determine the asphalt content that yields four percent air voids at the design compaction level according to NCDOT specifications (NCDOT 2020) was selected as the design asphalt content. The volumetric properties of the design mixture were calculated according to current specifications that assume complete availability and based on the measured availability.

2.7. Comparative Performance Testing of Current NCDOT versus Redesigned Asphalt Mixture Performance

The cracking and rutting performance of the control and redesigned mixtures were measured to assess the impacts of considering partial availability in volumetric mixture design procedures. The consideration of partial recycled binder availability in mix design is expected to yield a higher virgin binder content, which is expected to improve the cracking performance but may increase rutting susceptibility.

Indirect Tension Asphalt Cracking Tests (IDEAL-CT) were used to determine the cracking tolerance index (CT_{index}) of the control and redesigned mixtures according to ASTM D8225. The test consists of subjecting the specimens to an indirect tensile, monotonic displacement rate until failure, with the vertical load and displacement recorded during the entire test duration. For each mixture design, four specimens were fabricated for IDEAL-CT testing using the gyratory compactor to achieve a test specimen diameter of 150 mm and height of 62 mm. The air void contents for all of the fabricated specimens were within 7 ± 0.2 percent.

The Asphalt Pavement Analyzer (APA) was used to measure the rutting susceptibility of the control and redesigned mixtures according to AASHTO T 340-10. The test consists of applying repetitive loads to asphalt mix specimens through pressurized hoses via moving wheels. The tests were conducted at 64°C and the results are reported as the average rut depth after 8,000 wheel load cycles. Six gyratory-compacted specimens that were 150 mm in diameter and 75 mm tall were fabricated and subjected to testing for each mix design. All test specimens had air void contents within 4 ± 0.3 percent air voids, which fall within the NCDOT requirements (NCDOT 2020). The 'local contractor fabrication' procedure was followed when preheating the component materials prior to mixing. In addition, the loose mixtures were subjected to short-term aging for 4 hours at 135°C according to AASHTO R 30-02 recommendations for mechanical property testing prior to compaction.

3. RESULTS

3.1. Recycled Binder Contribution in Asphalt Mixtures

3.1.1. Comparison of the fatigue fracture and bulk specimen surfaces of asphalt mixtures

Visual inspection and tracer-based microscopy indicated the presence of agglomerations of adhered RAM particles within sawn surfaces of asphalt mixtures (referred to as bulk specimens herein). As discussed in Section 2, the addition of the titanium dioxide tracer to the virgin binder to distinguish it from the recycled binder turns the virgin binder brown. Consequently, regions in asphalt mixtures containing only virgin binder or a combination of recycled and virgin binders appear brown. This color differential from the black RAM binder allows for occasional optical visualization of recycled material agglomerations in asphalt mixture specimens. A specimen of the A45 mixture is shown in Figure 5. From the photograph, it is visually apparent that the large triangular aggregate (containing the inserted white flag) is surrounded by a region containing black (i.e., recycled) binder, indicating a RAP agglomeration containing fine aggregates.

Microscopy images of elemental composition were obtained using EDS in the region adjacent to the point of the flag, with the edge of the triangular aggregate shown in the lower right corner of the corresponding microscopy images, as marked by additional flags. The three microscopy images each show a different view of the same area. The electron image shows compositional contrast, with heavier elements reflecting more electrons and thus appearing brighter. In asphalt mixture, this results in bright aggregates and dark binder, mimicking what would likely be seen in a greyscale photograph of asphalt mixture. The sulfur (S) image is bright in regions with asphalt binder, as both virgin and recycled asphalt binders contains sulfur but aggregates generally do not. Within binder films, the titanium image is only bright in regions where virgin binder is present; however, in this specific mixture sample, it is evident that some titanium is naturally present in the large aggregate itself based on the relatively large bright yellow spots near the lower right corner of the titanium image. From visual comparison of the titanium and sulfur images, it is apparent that the titanium and sulfur images only match in the upper left corner of the image, which indicates that virgin binder (or a combination of virgin and recycled binder) is only present in that region. Along the edge of the triangular aggregate, there is a clear sulfur signal with no corresponding titanium signal, which indicates that only RAP binder is present. The microscopy images thus, confirm visual observations of the RAP agglomeration. Other, smaller agglomerations are also visually evident in the photograph in Figure 5. Similar findings exist in RAS mixtures.

Microscopy investigations conducted within the virgin binder matrix in this study reveal variation in local recycled binder contribution values within a given mixture, suggesting incomplete blending. Past studies have also noted observations of heterogeneous concentrations of recycled binder within the virgin binder matrix of asphalt mixtures (Castorena et al. 2016, Jiang et al. 2018, Abdalfattah et al. 2021). However, past studies and microscopy analyses herein do not reveal a clear trend in local recycled binder contribution values with increasing distance from RAM aggregate particles. The lack of a clear gradient in RAP binder contribution suggests that incomplete diffusion is not the primary inhibitor of recycled binder availability and blending as suggested in several past studies (Navaro et al. 2012, Booshehrian et al. 2013, Farris 2016). Based on these observations, is inferred that RAP agglomerations are the primary inhibitor of recycled binder availability in asphalt mixtures.

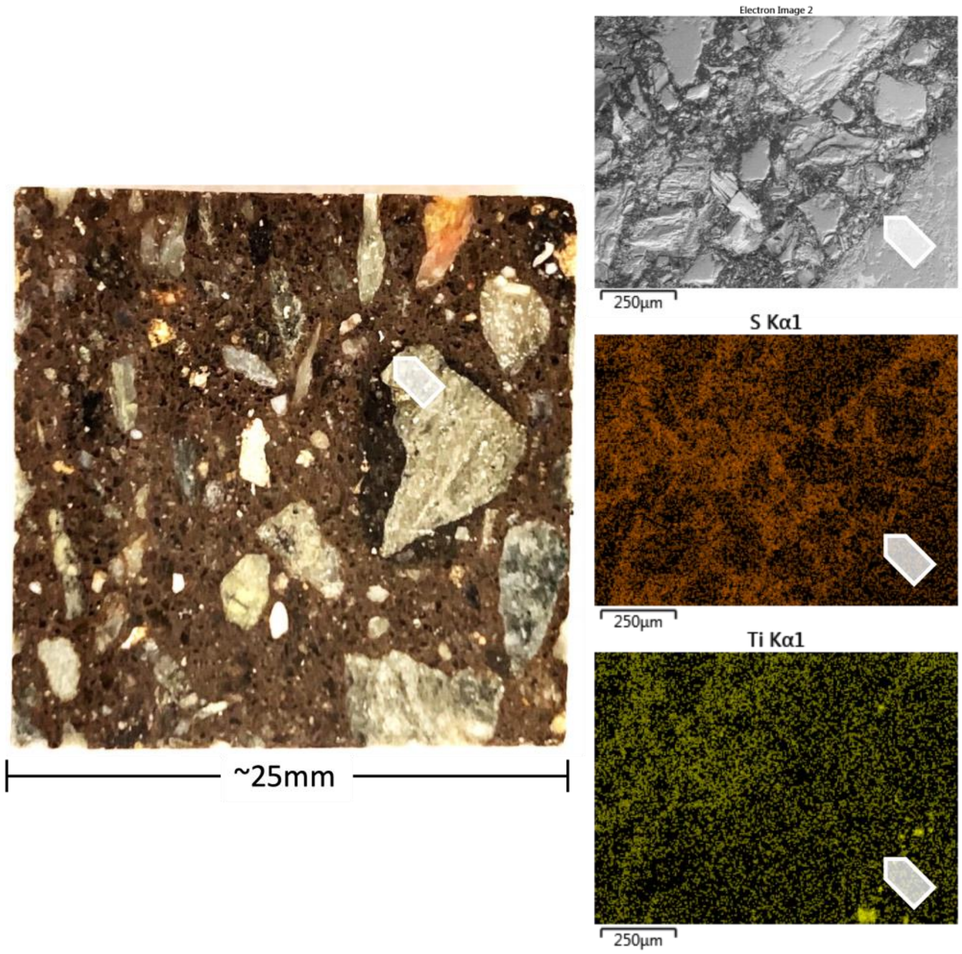

Figure 5. Visualization of RAP agglomerations in asphalt mixture.

The fatigue fracture surfaces and sawn surfaces were compared for select study mixtures to understand the role of recycled material agglomerations on the cracking resistance of asphalt mixtures and guide the locations for quantitative tracer-based microscopy of bulk specimens. Each fracture surface was visually analyzed to assess whether recycled material agglomerations were acting as fracture initiation sites. The fracture surfaces were embedded in epoxy resin, as illustrated in Figure 2 (c). Figure 6 shows two views of the AMPT fatigue specimen made from mixture A25/4. The fracture surface is shown in Figure 6 (a), and appears almost entirely brown, with some shadows cast by the irregular nature of the surface. This indicates the fracture occurred almost entirely through binder that contained titanium (i.e., contained virgin binder and possibly also recycled binder). Note that the bubbles are contained within the epoxy surrounding the specimen. Figure 6 (b) shows the sawn surface where the fracture specimen was removed from the loading platen. This plane includes several black recycled material agglomerations, indicating that recycled material agglomerations were present in the specimen and thus, fracture in the AMPT test appears to have occurred around the agglomerations rather than through them. Similar observations were made in the other mixtures evaluated.

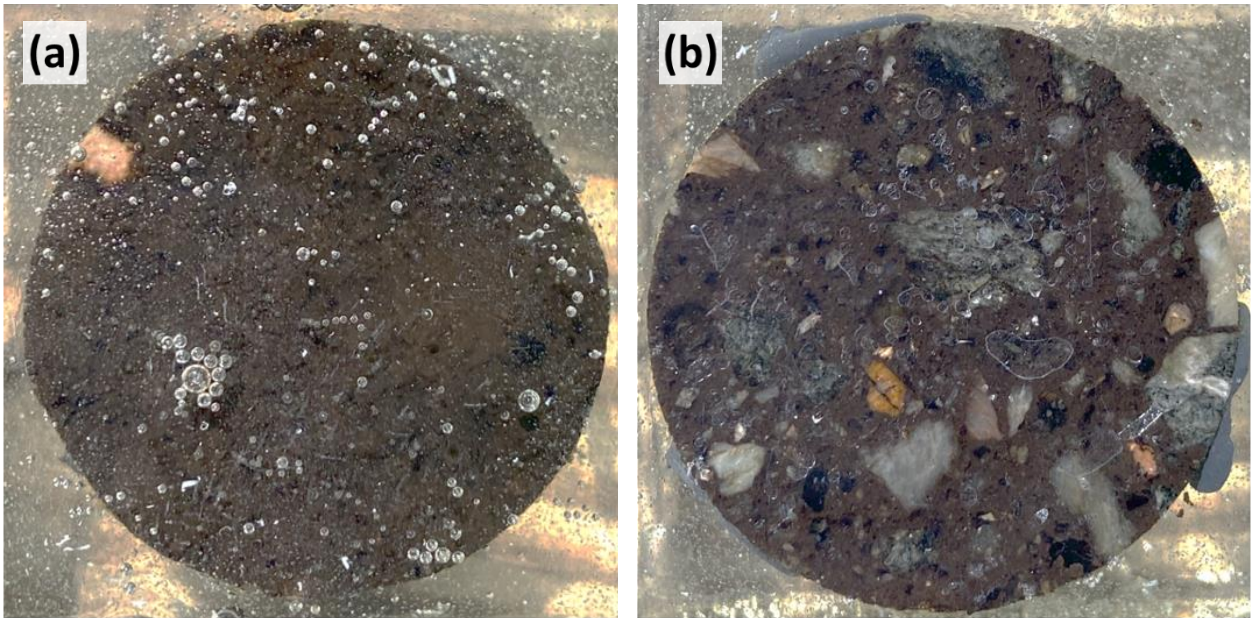

Figure 6. Photographs of AMPT cyclic fatigue specimen, mixture A25/4, embedded in epoxy resin to observe (a) the fracture surface and (b) a sawn surface.

The AMPT cyclic fatigue fractured specimens were sliced and prepared for EDS analysis, as illustrated in Figure 2 (d). In the backscattered electron images used to guide the microscopy, the epoxy resin stabilizing the fracture surface appeared a smooth gray color, similar to asphalt binder but containing no fine aggregate or dust. To evaluate the fracture surface itself, images were taken which contain both asphalt mixture and the stabilizing epoxy resin, with the area of asphalt along the interface being considered the fracture surface. At least 14 EDS maps were generated for each fracture surface investigated. For brevity, only a limited number are presented here. Figure 7 shows three EDS maps of the A25/4 mixture fracture surface. The elemental image headings include Kα1, indicating that the image is based on identification of the characteristic X-ray emitted when an electron returns to the K electron shell of the given element. The magnification level was selected to allow for imaging as much area as possible while maintaining accurate EDS results. The epoxy is evident by the greenish-grey regions with limited color contrast in the layered EDS images shown on the left that contain no titanium (Ti) or sulfur (S) based on the right two images. The first and second rows of maps (Sites 1 and 4) show that the areas containing titanium and sulfur visually coincide, indicating that virgin binder is present in all binder films and that no recycled material agglomerations exist along the fracture surface despite being visually evident in the bulk mixture. Site 5 was specifically selected for inclusion as it shows evidence of virgin binder present in all binder films near the fracture surface, as illustrated by the appearance of the mixture inside the boxes marked Spectrum 2, 3, and 4 where patterns of titanium and sulfur match. However, there is a recycled material agglomerations containing unavailable asphalt just inside the fracture surface, as can be seen in the box marked Spectrum 1 where sulfur (and thus, binder presence is evident) but the titanium signal is weak (indicating that virgin binder is absent).

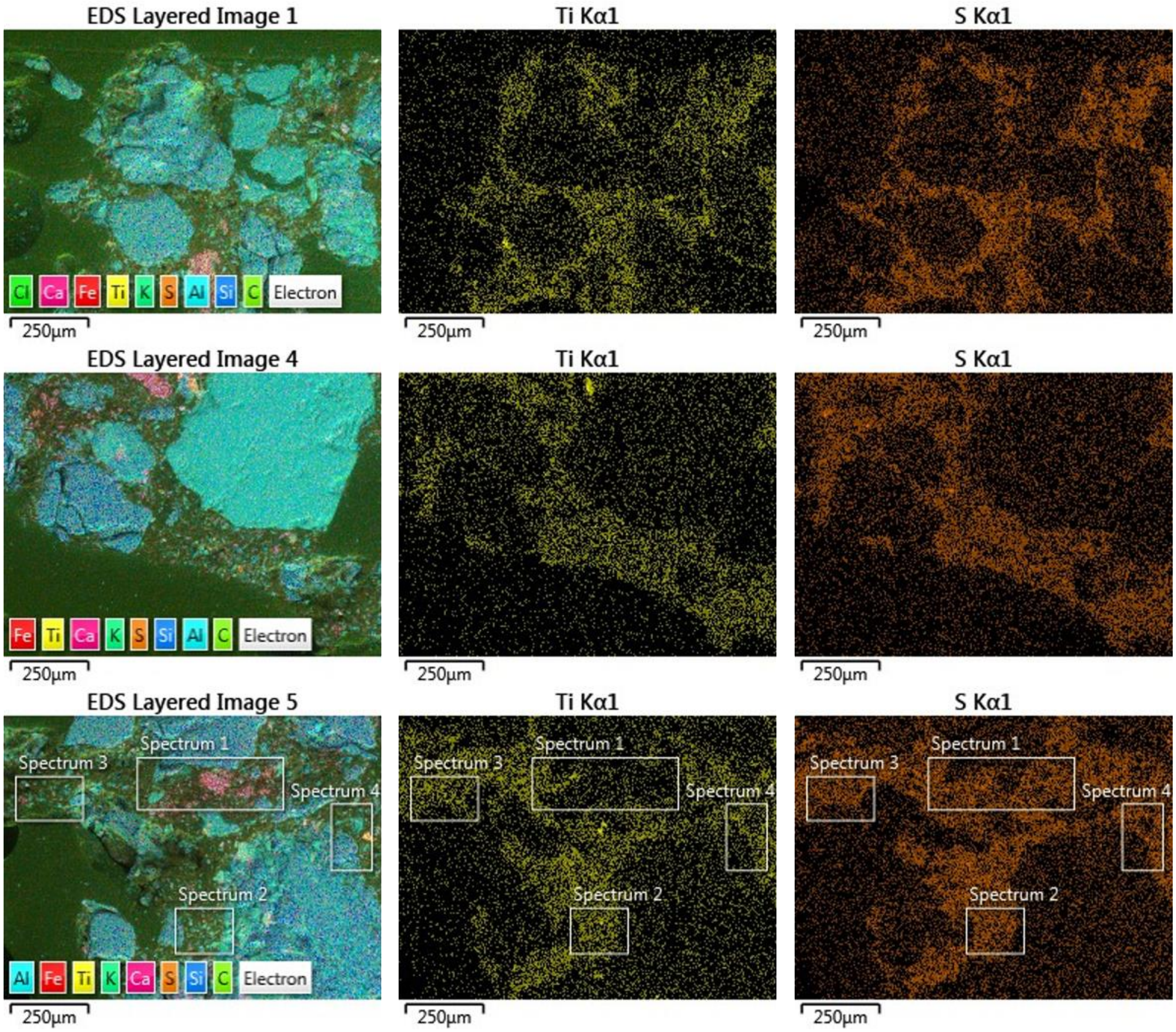

Figure 7. EDS visualization of the A25/4 asphalt mixture fracture surface.

Figure 8 shows example EDS maps of obtained along the fracture surfaces of mixtures prepared with recycled material sources B and C. A single, representative image was selected for each mixture for brevity. All the titanium and sulfur maps appear visually similar, thus demonstrating the presence of virgin binder in all binder films near the fracture surfaces of asphalt mixtures. The clear lack of recycled material agglomerations along the fracture surface indicates that cracks propagate through the matrix containing the combination of available recycled binder and virgin binder, suggesting that RAP agglomerations act as black rocks. The most critical sites for fracture and thus quantitative EDS analysis are the areas with uniform tracer dispersion, away from recycled material agglomerations since this is where all the fatigue cracks occurred.

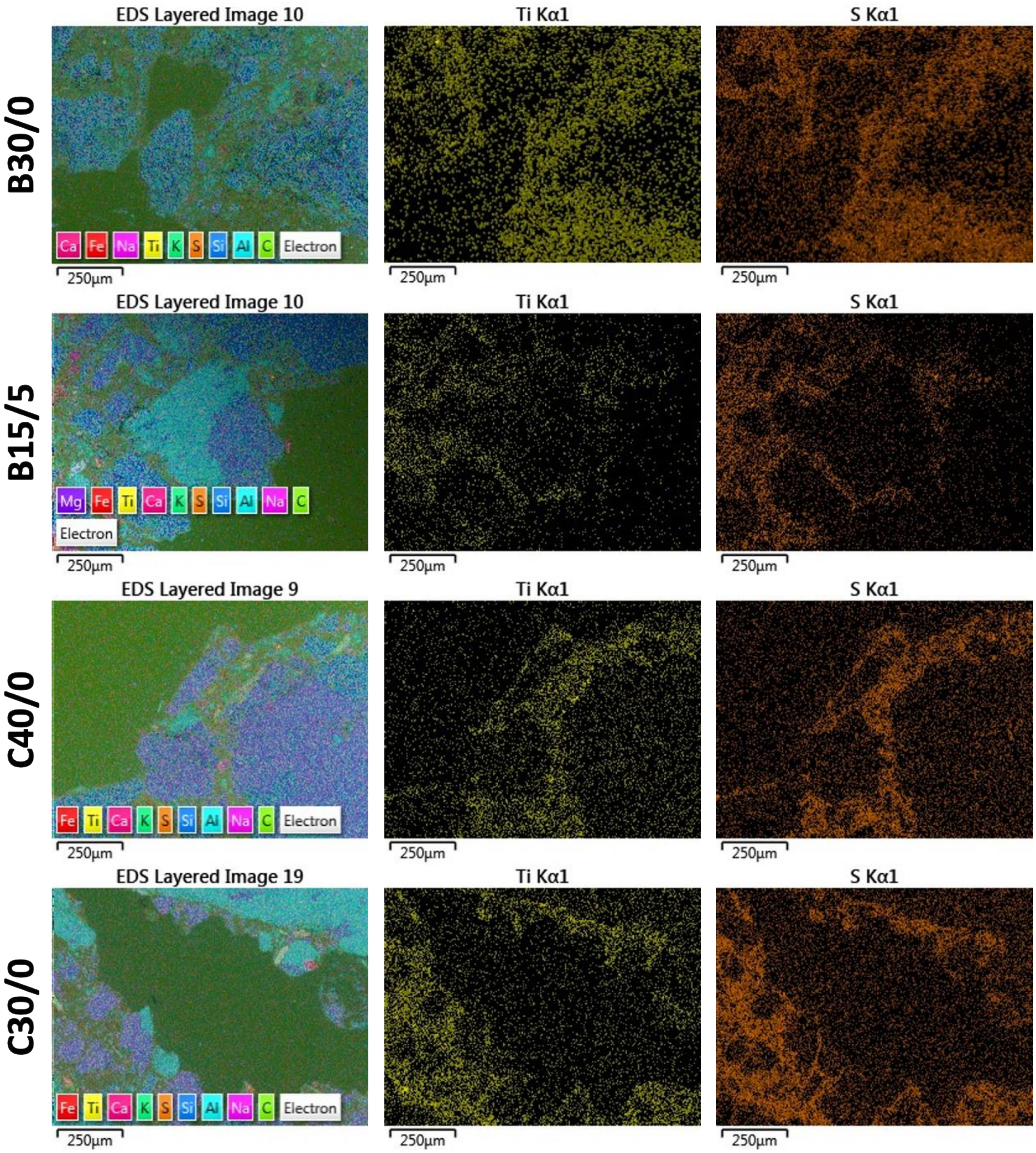

Figure 8. EDS visualization of the source B and C asphalt mixture fracture surfaces.

While evaluating fracture surfaces provides valuable information on the composition of the material at the fracture surface, it is both time- and resource-intensive to prepare and test cyclic fatigue test specimens and then prepare and test the fracture surfaces with this methodology. To expedite sample preparation for EDS analysis, it would be preferable to work with a bulk gyratory specimen. Thus, comparison specimens were fabricated from separate gyratory specimens to assess the viability using bulk specimens instead of fracture surface specimens. Based on the observation that fatigue cracks propagate around recycled material agglomerations that act as ‘black rocks’, bulk specimen EDS analysis for local recycled binder contribution measurements focused on areas with binder films present that did not contain recycled material agglomerations based on comparison of titanium and sulfur maps. Given that there was not a predefined area to visualize along, imaging was conducted in a switchback pattern on the sample (to avoid multiple images in the same area) until preliminary assessment yielded ten sites without artifacts (i.e., highway paint which contains titanium, aggregate containing titanium, and/or recycled material agglomerations).

Figure 9 shows the comparison of the fracture surface and bulk mixture specimen microscopy recycled binder contribution results. The height of the bars indicates the average local recycled binder contribution and the error bars correspond to the standard error of the measurements, which is an indicator of the degree of blending. The results of statistical tests comparing the mean recycled binder contribution of the fracture surface and bulk specimen surface of a given mixture are presented in Table 6. For Sources A and B, the use of bulk mixture specimens shows no significant difference from the results along the fracture surface, indicating that bulk specimen measurements can be used in lieu of fatigue fractured specimens. However, the mixtures from Source C were unexpected. It was expected that the use of the same RAP source should yield similar average recycled binder contribution values given that agglomerations are the primary inhibitor of availability. In addition, while the bulk and fracture specimens for the C40 mixture show no significant difference, the C30 specimens show a significant difference in average recycled binder contribution values, and neither corresponded with the results of the C40 specimens. To investigate this discrepancy further, additional imaging of both C-source fracture specimens was conducted along the cut edge where the specimen was sawn off the platen. These showed identical average values of 76 percent, indicating that the results from C40 are likely to be accurate. The very low contribution along the fracture surface in the C30 specimen is hypothesized to be a result of non-uniform blending of the binders, resulting in fracture through the softest binder matrix with the least recycled binder and highlights the potential impacts of heterogeneous and variable composition of asphalt mixtures on failure. Considering the C30 fracture surface result appears to be an outlier, the results suggest that careful imaging of bulk specimens, avoiding confounding artifacts, allows for adequate characterization of mixtures without conducting fracture tests.

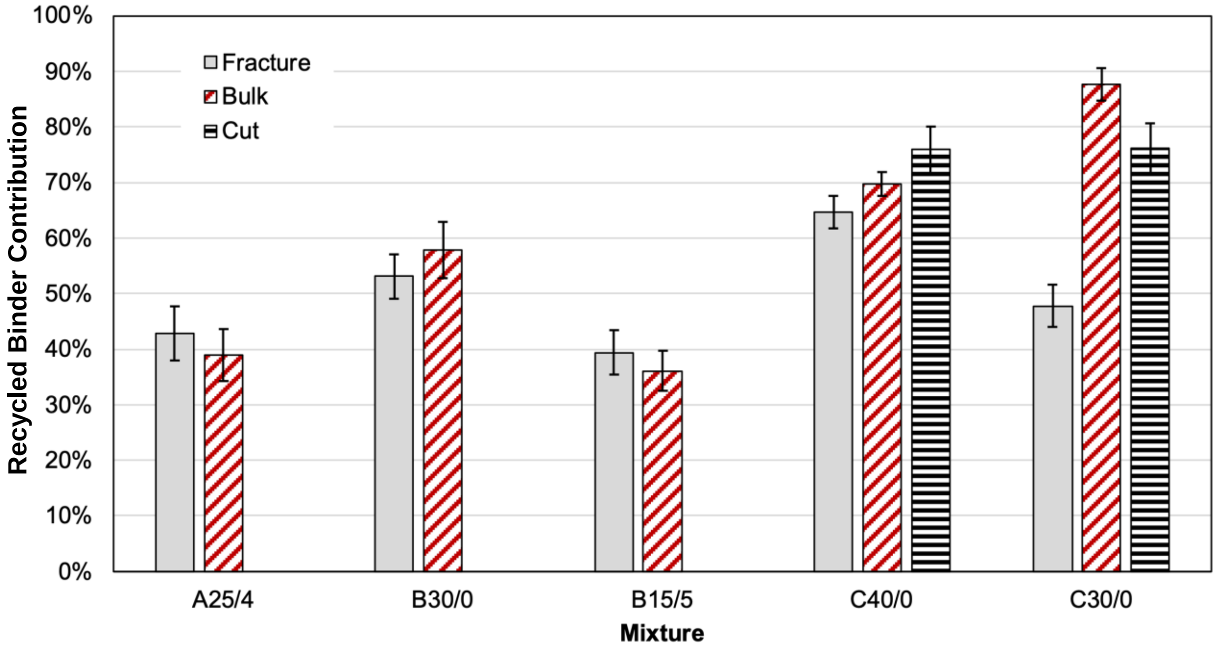

Figure 9. Comparison of fracture specimen and bulk mixture recycled binder contribution.

Table 6. Results of Statistical Tests Comparing Fracture Surface vs. Bulk Specimen Microscopy Recycled Binder Contribution Results

Mix	Statistical Test Result
A25/4	Equivalent
B30	Equivalent
B15/5	Equivalent
C40	Equivalent
C30	Different

The error bars in Figure 9 indicate that the variability in local recycled binder contribution values among the mixtures and specimen types evaluated are comparable. However, the recycled binder contribution of the different mixtures varies among the different mixtures, suggesting that recycled binder availability varies among RAM type (i.e., RAP vs. RAS) and to a lesser extent among sources, which was further evaluated using additional analysis of bulk specimens.

3.1.2. Effect of RAM Source and Content on Recycled Binder Contribution

Figure 10 shows the tracer-based microscopy recycled binder contribution results of the collective mixtures given in Table 1 when material preheated followed the local contractor procedure. The results shown in Figure 10 by preparing bulk specimens and obtaining EDS measurements at a minimum of 10 locations, guided by the findings from comparing the fracture surface and bulk specimen results discussed above. The A RAS and B RAS values shown in Figure 10 correspond to the inferred RAS binder contribution according to Equation (2), which integrates the collective results of the RAP and RAP/RAS mixtures obtain from the given source.

The results in Figure 10 indicate recycled binder contributions values between approximately 50 and 90 percent for the RAP mixtures evaluated. These results further highlight that the assumption of 100 percent availability is erroneous. Furthermore, the microscopy results suggest that different RAP sources can yield different recycled binder contribution values. For Sources B, C, and D, the mixtures evaluated, with the exception of C30, indicate similar recycled binder contribution results near 60 percent. In contrast, the Mixture A29 and A45 results indicate the RAP form Source A had notably higher contribution than the other sources. The recycled binder contribution results of the difference RAP mixtures do not exhibit clear trends with respect to the RAP binder grade as evident by comparing trends in Figure 10 to the high-temperature grades reported in Table 2. RAP Source A yields the highest recycled binder contribution but has an intermediate high-temperature grade compared to the other sources evaluated.

With the exception of Mixture C30, which appears to be an outlier, the results suggest only marginal differences in the recycled binder contribution in mixtures prepared using the same RAP source and no clear trend with respect to RAP content. If agglomerations form during mixing, it is expected that the extent agglomerations would increase as the RAP content (and thus opportunity for agglomeration) increases; therefore, these results suggest that RAP agglomerations are pre-existing and do not form upon mixing.

Comparison of the recycled binder contribution results of the RAP/RAS mixtures and RAP only mixtures prepared from Sources A and B indicate that RAS has lower recycled binder contribution than RAP. Correspondingly, the inferred RAS binder contribution is notably lower than the than the RAP binder contribution (based on the results of the RAP only mixtures), which matches expectations since RAS is purposefully oxidized during production through heat and air blowing to achieve the desired characteristics for roofing, which are much harder than paving asphalts. The inferred RAS binder contributions are zero and 30 percent for Sources A and B, respectively. Source A had a notably higher high-temperature grade (194°C) than Source B (143°C), as shown in Table 2, suggesting that the RAS binder grade may influence recycled binder availability.

Note that the recycled binder availabilities currently assumed by state agencies that consider partial recycled binder availability in their mix design procedures vary from 60 to 100 percent for RAP and 60 to 85 percent for RAS (Abdelaziz et al. 2021). The results herein suggest that these assumptions may be erroneously high in some instances, particularly for RAS.

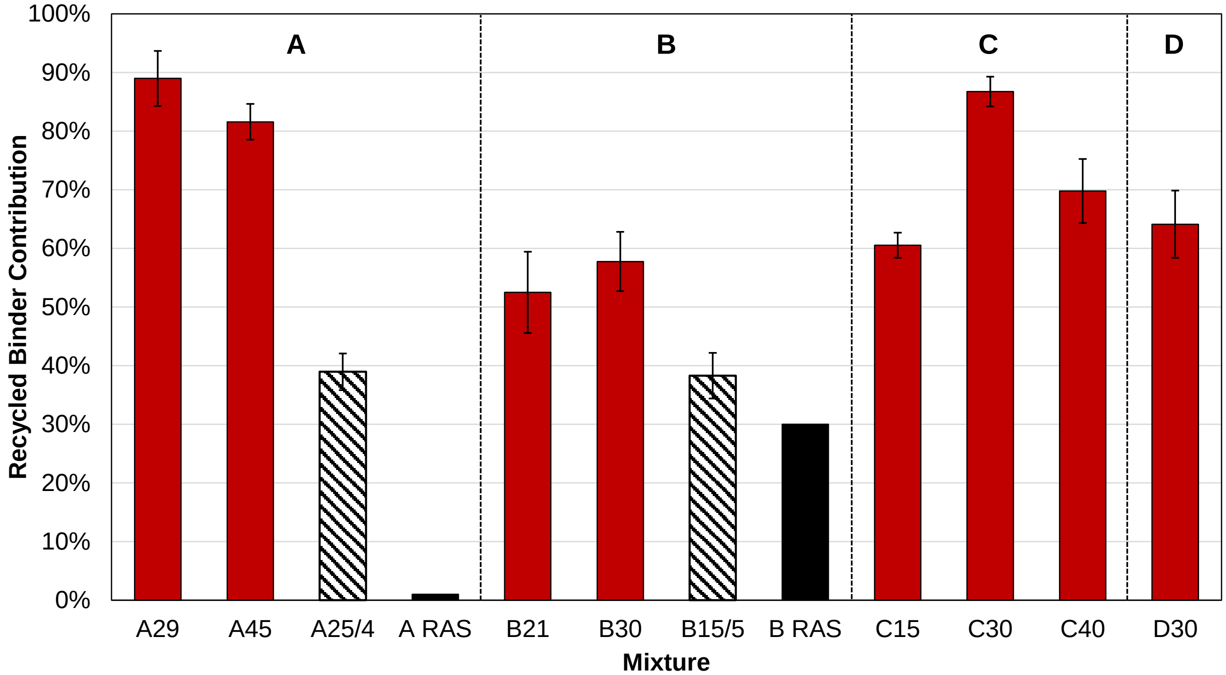

Figure 10. Recycled binder contribution results of the control mixtures fabricated using the local contractor procedure.

3.1.3. Effect of Lab Production Variables on Recycled Binder Contribution

The effect of the laboratory material preheating procedure on the recycled binder contribution of select study mixtures are presented in Figure 11. The preheating procedures are detailed in Table 4. Table 7 shows the results of the statistical tests comparing the average recycled binder contribution result obtained from different preheating procedures for a given mixture. Equivalent results in Table 7 indicate the pair of conditions were deemed both equivalent and not different from statistical tests whereas different results indicate the pair of conditions were deemed both not equivalent and different. Cases deemed not equivalent but also not different are indicated as not different in Table 7.

The results demonstrate minimal sensitivity in the recycled binder contribution of the C40 mixture to the production procedure; all conditions were deemed equivalent based on statistical tests. The results of the other mixtures do suggest that the recycled binder contribution can vary with the preheating and therefore, mixture production procedures. For the RAP only mixtures, the local contractor and NCHRP 09-12 procedures yield equivalent recycled binder contribution for a given mixture. A difference is noted for the A25/4 mixture; the reason for the higher difference in the RAP/RAS mix is unknown. Perhaps it is due to the inclusion of RAS. The superheat procedure using a virgin aggregate temperature of 240°C yielded the most distinct results for Mixtures A25/4 and B21. For the C40 mixture, the combination of ambient temperature RAP with 240°C virgin aggregate is expected to yield a RAP temperature of 155°C according to the weighted average temperature of the virgin aggregate-RAP mixture; this is very close to the target mixing temperature. In contrast, the virgin aggregate superheat temperature of 240°C likely yielded excessive RAM temperatures in the other mixtures that had lower recycled material content. For example, a virgin aggregate superheat temperature of 240°C is expected to yield an excessive RAP temperature of 195°C in the B21 mixture (with only 21 percent RAP). Production temperatures for hot-mix asphalt do not typically exceed 170°C (Sobieski et al. 2021). When the virgin aggregate superheat temperature was reduced to 200°C to yield a RAP temperature of approximately 155°C, the recycled binder contribution in the B21 mixture became more similar but not statistically equivalent to the local contractor and NCHRP 09-12 preheating procedures. The results of the D30 mixture, with higher RAP content than the B21 mixture but less than that of the C40 mixture, were not statistically different but also not deemed statistically equivalent when the superheat condition was used compared to the NCHRP 09-12 and local contractor procedures.

The NCHRP 09-12 procedure is expected to yield a RAM temperature that is lower than the mixing temperature since RAM conditioned to 110°C is combined with virgin aggregate heated only slightly above the target mixing temperature. The local contractor procedure will not yield a RAP temperature that exceeds the mixing temperature since the RAP is conditioned for 45 min at a temperature only slightly above the target mixing temperature. Thus, RAM temperatures may also differ among the local contractor and NCHRP 09-12 procedures but the effect on the recycled binder contribution is much less pronounced than when RAM temperatures that greatly exceed the mixing temperature are induced. The collective results suggest that excessive RAM temperatures may lead to increased recycled binder contribution but those that yield temperatures relatively close to the mixing temperature have comparatively marginal impacts.

It is recommended that the NCDOT standardize material preheating procedures for RAM mixtures to improve the consistency of laboratory-produced mixtures. Future research is needed to better understand the temperature of RAM during plant production and corresponding recycled binder contribution in plant-produced asphalt mixtures.

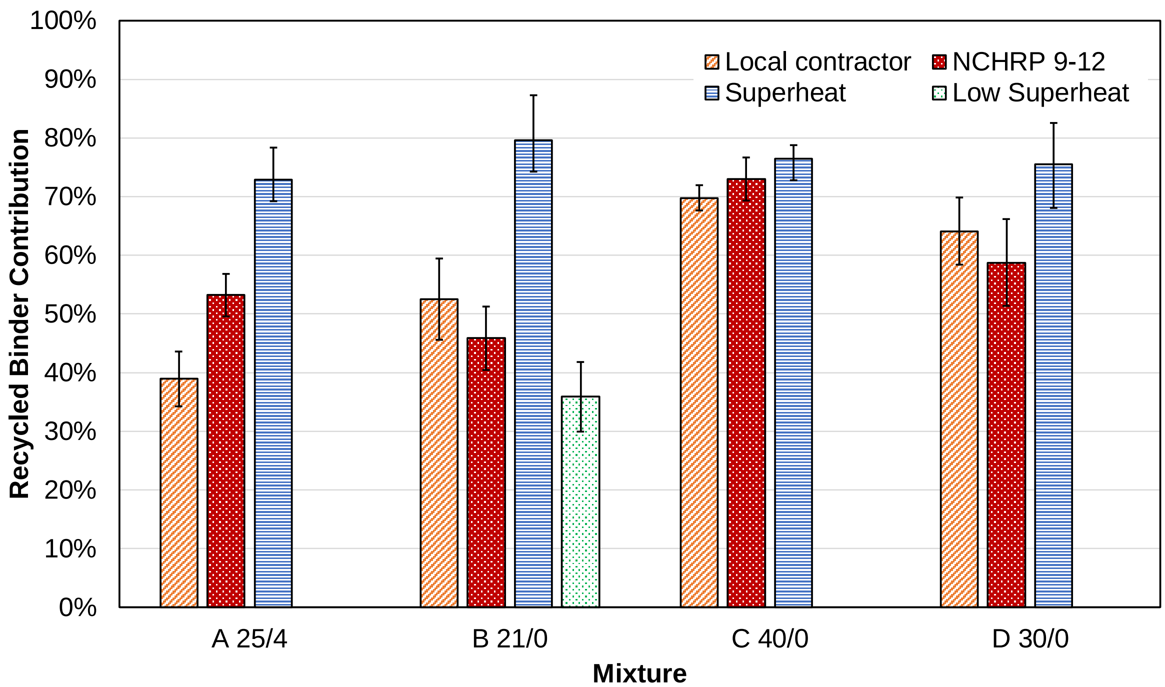

Figure 11. Effect of the laboratory material preheating procedure on recycled binder contribution.

Table 7. Statistical Test Results of the Effects of Laboratory Preheating Procedures on Recycled Binder Contribution

Preheating Procedure Comparison	Mix			
	A25/4	B21	C40	D30
Local contractor vs. NCHRP 09-12	Different	Equivalent	Equivalent	Equivalent
Local contractor vs. Superheat 240ºC	Different	Different	Equivalent	Not Different
Local contractor vs. Superheat 200ºC	--	Different	--	--
NCHRP 09-12 vs. Superheat 240ºC	Different	Different	Equivalent	Not Different
NCHRP 09-12 vs. Superheat 200ºC	--	Not Different	--	--
Superheat 240ºC vs. Superheat 200ºC	--	Different	--	--

3.1.4. Effect of the Virgin Binder, RAP Age Level, and Additives on Recycled Binder Contribution

Figure 12 shows the effects of binder variables on recycled binder contribution in the C40 mixture.

Figure 13 shows the effects of two recycling agents on the recycled binder contribution in the A25/4 mixture. Table 10 shows the results of statistical tests comparing different pairs of recycled binder contribution results. All results shown in this section were prepared using the local contractor preheating method.

The control C40 mixture included a PG 58-28 virgin binder whereas the alternate binder mixture was prepared with the same constituent materials but a PG 64-22 virgin binder. The aged RAP mixture was prepared using the same component materials and proportions as the control C40 mixture but prior to sample fabrication, the RAP was aged in an oven at 95°C for 4 days to simulate a harsher aging state. The anti-strip mixture is identical to the control C40 mixture with the exception that an anti-strip additive was added. The results in Figure 12 and Table 10 indicate that the alternate virgin binder yielded a higher recycled binder contribution than the control mixture. However, the harsher RAP age level and incorporation of an anti-strip additive resulted in equivalent recycled binder contribution to the control C40 mixture. It is unclear why the PG 64-22 virgin binder yielded higher recycled binder contribution compared to the PG 58-28. The C40 mixtures with RA1 and the extender were prepared using the alternate binder. The results of the RA1 mixture and extender are equivalent to the C40 alternate binder result and thus, the additives had no effect on the recycled binder contribution.

The A25/4 control mixture was prepared using a PG 58-28 virgin binder. The A25/4 mixtures with RA1 and RA2 were prepared using the same virgin aggregate and RAM as the control mixture but with a PG 64-22 virgin binder and recycling agent. The A25/4 mixture with RA1 mixture yielded a higher (and statistically different) recycled binder contribution than the control mixture based on

Figure 13 and Table 10. The recycled binder contribution of the A25/4 mixture with RA2 was also higher than the control mixture but considered statistically equivalent. The increase in recycled binder contribution in the RA1 and RA2 mixtures could have been caused by the difference in virgin binder and/or the RA. It is speculated that it was caused by the virgin binder rather than the RA because the PG 64-22 virgin binder yielded an increase in recycled binder contribution in the C40 mixture.

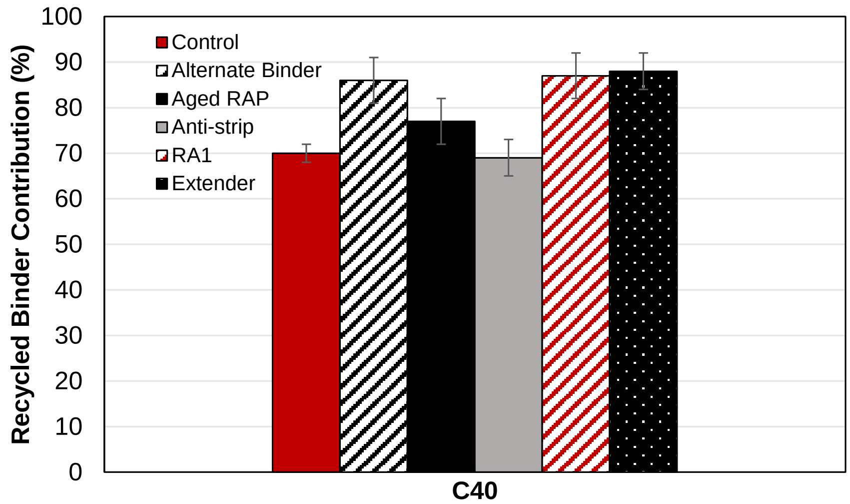

Figure 12. Effect of binder variables on the recycled binder contribution in the C40 mixture.

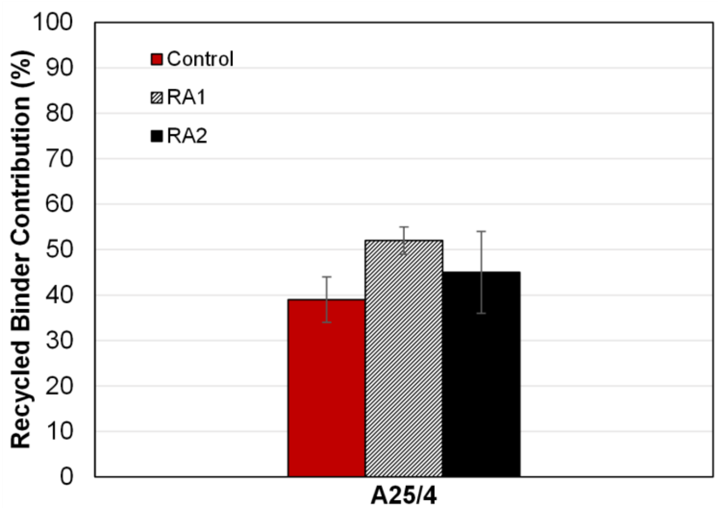

Figure 13. Effects of binder variables on the recycled binder contribution in the A25/4 mixture.

Table 8. Statistical Test Results of the Effects of Binder Variables on Recycled Binder Contribution

Mixture	Comparison	Statistical Test Result
C40	Control vs. Alternate Binder	Different
	Control vs. Aged RAP	Equivalent
	Control vs. Antistrip	Equivalent
	Alternate Binder vs. RA1	Equivalent
	Alternate Binder vs. Extender	Equivalent
A25/4	Control vs. RA1	Different
	Control vs. RA2	Equivalent
	RA1 vs. RA2	Equivalent

3.2. RAP Recycled Binder Availability Results from Sieve Analysis

3.2.1. Sieve Analysis Results

The findings from tracer-based microscopy that suggest agglomerations of adhered RAM are the primary inhibitor of recycled binder availability guided the development of a practical method to determine recycled binder availability from RAP. It is hypothesized that the majority of agglomerations are present in the RAP prior to asphalt mixture production based on the limited sensitivity of recycled binder contribution recycled to RAM content and to preheating procedures that do not induce excessive RAP temperature. Accordingly, the difference in the particle size distribution of RAP and recovered RAP aggregate provides a measure of the extent of agglomeration, and therefore, recycled binder availability. Note that this study is limited to laboratory-produced asphalt mixtures and findings should be verified in the future using plant-produced asphalt mixtures.

Washed sieve analysis was conducted on a sample of RAP, which was then ignited using the ignition oven to remove the asphalt binder and separate aggregates. The recovered aggregate was collected and subjected to washed sieve analysis again. The results are plotted in Figure 14, with Figure 14 (a) showing the results of the RAP, while Figure 14 (b) shows the results of the recovered aggregate. The overall shapes indicate that the RAP is much coarser than the recovered aggregate, especially at the finest sieves. Notably, the RAP exhibits minimal material passing the No. 200 sieve; this observation was the primary basis for establishing the three-step procedure described above to estimate the RAP binder availability based on peripheral versus total mastic contained within the RAP.

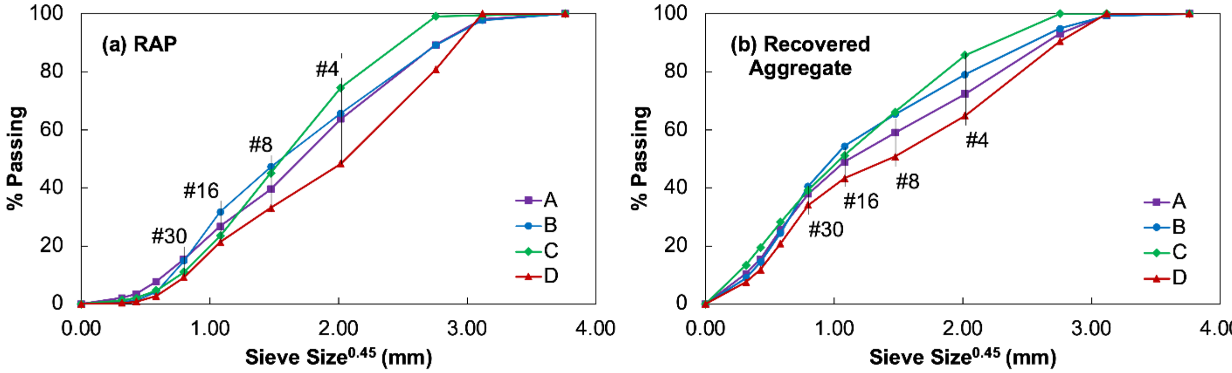

Figure 14. Gradation curves for (a) RAP and (b) recovered aggregate.

Figure 15 shows the individual percent retained on different sieve sizes was investigated to better visualize the impact of agglomeration on different aggregate sizes. Each RAP source show distinct trends, with all showing sizeable discrepancies at the finest sieve sizes. RAP A shows slightly less discrepancy between the RAP and recovered aggregate, as compared to the other three sources.

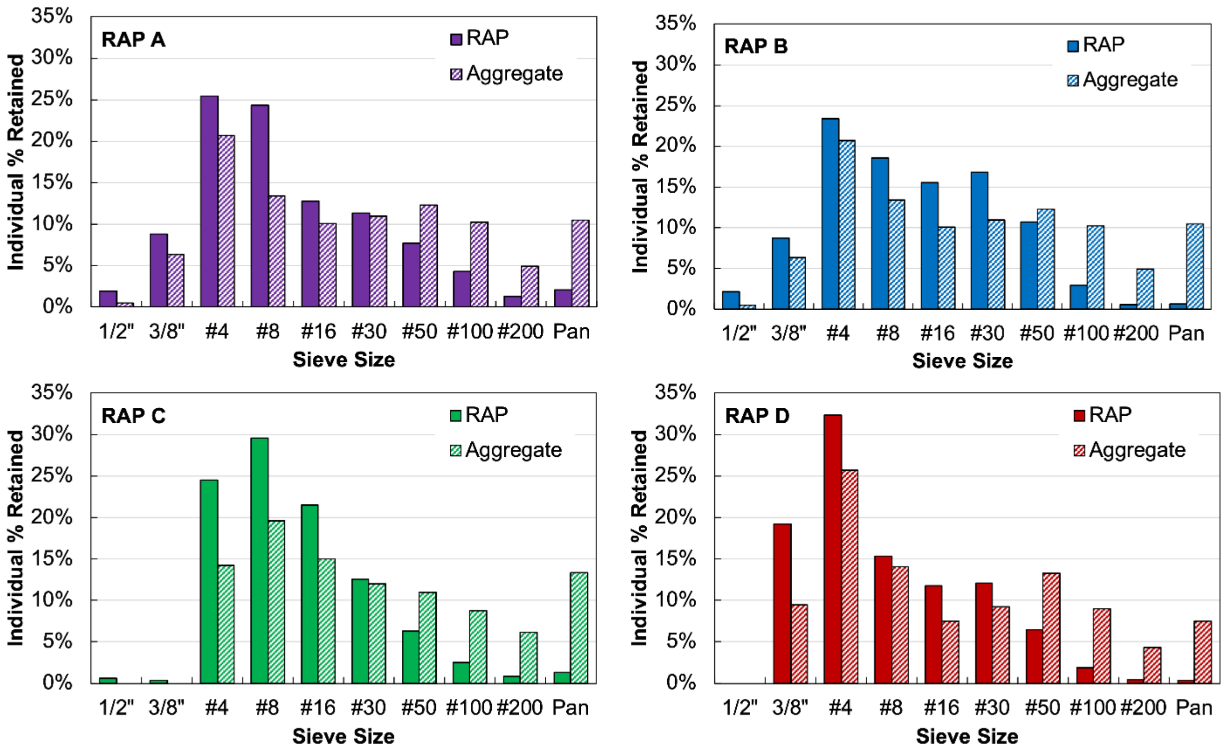

Figure 15. RAP and recovered aggregate gradation comparison.

Table 9 shows the mastic film thickness (t) and recycled binder availability values estimated from sieve analysis according to the procedure described in Section 2.4 under two extreme assumptions for asphalt binder absorption (P_{ba}): zero absorption (i.e., $G_{sb} = G_{se}$) and three percent absorption. The specific gravity and binder content input parameters for each RAP source under the two assumed absorption scenarios are also provided in Table 9.

The results show that sieve analysis method for calculating recycled binder availability identifies differences among RAP sources, ranging from approximately 50 percent in Source D to approximately 80 percent in Source A. Furthermore, the results show that while the mastic film thickness value is sensitive to the assumed absorption, the calculated recycled binder availability values do not vary considerably with the assumed absorption value. The maximum difference in the resultant availability resulting from a three percent difference in assumed absorption is six percent, which would result in negligible differences in terms of the inferred RBR and effective binder content given the typical RAP contents in use. These results indicate that uncertainty in the asphalt absorption characteristics of RAP aggregate and corresponding RAP aggregate bulk specific gravity values do not contribute to concerning uncertainty in recycled binder availability estimates from sieve analyses under the proposed framework. Based on these results, it is suggested that the effective aggregate specific gravity and total binder content are used due to uncertainty associated with defining the RAP aggregate absorption.

Table 9. Comparison of Availability Results Obtained using Differing Assumptions of Binder Absorption

RAP	Method	G_{sb}	P_{ba} (%)	P_b (%)	P_{be} (%)	*t* (micron)	Availability
A	G_{se}	2.727	0.0	5.3	5.3	23.6	83%
	P_{ba} = 3%	2.474	3.0	5.3	1.1	16.7	77%
B	G_{se}	2.670	0.0	5.3	5.3	19.6	57%
	P_{ba} = 3%	2.476	3.0	5.3	2.4	14.0	56%
C	G_{se}	2.761	0.0	5.7	5.7	25.8	63%
	P_{ba} = 3%	2.553	3.0	5.7	2.8	19.4	60%
D	G_{se}	2.720	0.0	4.8	4.8	21.5	51%
	P_{ba} = 3%	2.519	3.0	4.8	1.9	13.5	49%

Figure 16 shows the comparison between recycled binder availability results from sieve analysis and tracer-based microscopy measurements of recycled binder contribution in mixtures prepared with the four RAP sources. All tracer-based microscopy results shown coincide with the local contractor fabrication procedure. Table 10 shows the statistical test results comparing the mean recycled binder contribution measurements with the recycled binder availability result from sieve analysis for a given mixture. Equivalent results in Table 10 indicate the results were deemed both equivalent and not different from statistical tests whereas different results indicate the results were deemed both not equivalent and different. Cases deemed not equivalent but also not different are indicated as not different.

The results of the sieve analysis method are generally close to those obtained from tracer-based microscopy and are deemed equivalent with the exception of Mixture C30 (deemed different) and C40 (deemed not equivalent but also not different). The agreement between sieve analysis and microscopy results suggests that the breakdown of agglomerations of RAP during asphalt mixture production is minimal and that the sieve analysis offers a practical tool to estimate RAP binder availability.

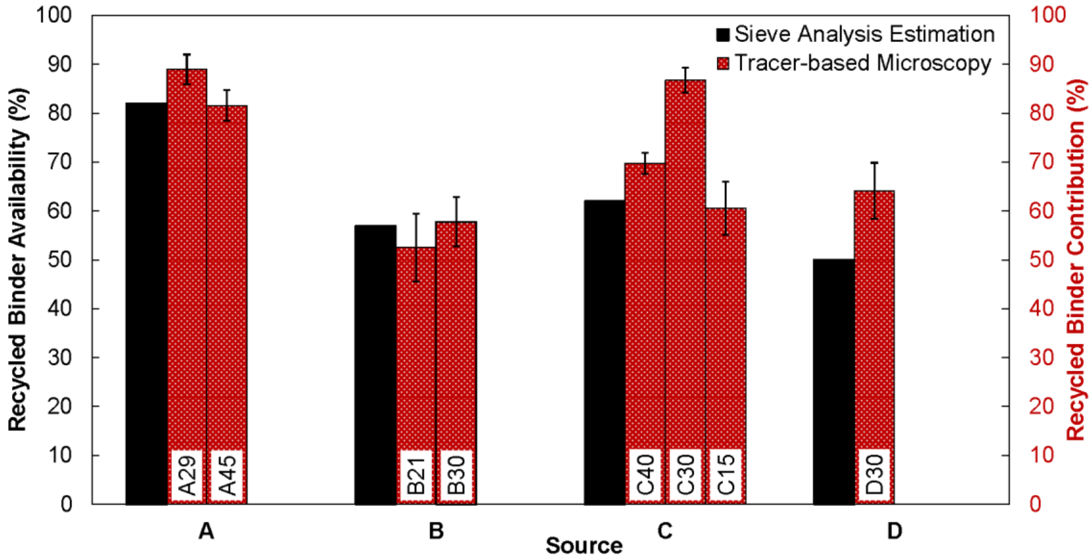

Figure 16. Comparison of recycled binder availability results of sieve analysis and tracer-based microscopy measurements of recycled binder contribution in asphalt mixtures.

Table 10. Statistical Test Results Comparing Recycled Binder Contribution Measurements from Tracer-based Microscopy and Recycled Binder Availability Measurements from Sieve Analysis

Mix	Statistical Test Result
A29	Equivalent
A45	Equivalent
B21	Equivalent
B30	Equivalent
C15	Equivalent
C30	Different
C40	Not Different
D30	Equivalent

3.3. Incorporation of Recycled Binder Availability into Mixture Design

3.3.1. Implications of Recycled Binder Contribution and Availability Findings on Volumetric Mixture Design

Table 11 presents three possible changes to volumetric mixture design in light of the collective findings of recycled binder contribution and availability measurements in the laboratory-produced asphalt mixtures evaluated.

Table 11. Possible Implications of Recycled Binder Availability on Mixture Design

Component	Change
Virgin binder selection	Use the 'effective' rather than total recycled binder replacement ratio
Design of the aggregate structure	Use the recycled material rather than recovered aggregate gradation
Volumetric property inferences	Include unavailable recycled binder in the bulk aggregate volume

First and foremost, unavailable recycled binder should be considered part of the bulk aggregate rather than the binder volume in the asphalt mixture. Consequently, accounting for recycled binder availability lowers the calculated effective binder volume in a given mixture. Accordingly, consideration of recycled binder availability may affect virgin binder selection if one considers the 'effective' recycled binder replacement ratio (RBR), defined in Equation (7), rather than the total RBR. In this study, the virgin binder was kept the same between the control and redesigned mixes to isolate the other variables within the performance comparisons.

$$Effective\ RBR = \frac{V_{RAP\ binder} \times Availability_{RAP} + V_{RAS\ binder} \times Availability_{RAS}}{V_{RAP\ binder} \times Availability_{RAP} + V_{RAS\ binder} \times Availability_{RAS} + V_{virgin\ binder}} \tag{7}$$

where: $V_{RAP\ binder}$ = volume of RAP binder, $V_{RAS\ binder}$ = volume of RAS binder, $Availability_{RAP}$ = RAP recycled binder availability, $Availability_{RAS}$ = RAS recycled binder availability, and $V_{virgin\ binder}$ = volume of virgin binder.

If RAM agglomerations act as 'black rocks' that do not break down during sieving and continue to persist in the mixture, it follows that the use of the RAM gradation (i.e., black curve) rather than the recovered aggregate gradation when designing the aggregate structure may better reflect the state of the RAM in the mix. Using the RAM gradation also affects calculation of the dust-to-binder ratio (DP) (i.e., P200 content/effective binder content) of a given mixture. Note that the characterization of RAM black curves has been used by many researchers as a tool to better understand the material's characteristics and the quality of processing (e.g., Menegusso Pires et al. 2019, Roque et al. 2020, and Zaumanis et al. 2021) but has not been used extensively for aggregate gradation design. Saliani et al. (2019) did evaluate the use of RAP black curves for designing the gradation of asphalt mixes; however, full recycled binder availability was considered in their analysis and therefore, the study did not comprehensively evaluate the implications of agglomerations on volumetric mixture design.

Lastly, including the unavailable recycled binder in the bulk aggregate volume lowers the calculated VMA for a given asphalt mixture. Figure 17 presents a comparison of the compacted asphalt mixture phase diagrams (a) in current specifications that assume complete recycled binder availability and (b) when including unavailable recycled binder within the bulk aggregate volume. The corresponding revised calculation of the bulk aggregate volume (V_{sb}) when considering availability is shown in Equations (8) and (9). In Equation (9), the effective aggregate volume (V_{se}) of RAM is used instead of V_{sb} because it is assumed that the absorbed binder is part of the unavailable binder. Note that calculating the bulk aggregate volume according to Equation (9) negates the need for the bulk specific gravity of the RAP aggregate, which constitutes significant uncertainty (Copeland 2011). The calculated air void content of a compacted mixture is not affected by the recycled binder availability and thus, the effective binder volume, calculated according to Equation (10), decreases when considering partial as opposed to complete availability. The reduction in effective binder volume also implies that current designs assuming 100 percent availability may yield lower actual VMAs (Equation (11)) than the current calculations suggest. Considering availability also impacts the calculation of the voids filled with asphalt (VFA) and DP.

$$V_{unavailable\ RAM\ binder} = V_{RAP\ binder} \times (1 - Availability_{RAP}) + V_{RAS\ binder} \times (1 - Availability_{RAS}) \quad (8)$$

$$V_{sb} = V_{sb\ virgin} + V_{se\ RAP} + V_{se\ RAS} + V_{unavailable\ RAM\ binder} \quad (9)$$

$$V_{be} = V_{mb} - V_{sb} - V_{a} \quad (10)$$

$$VMA = \frac{V_{ma}}{V_{mb}} = \frac{V_{mb} - V_{sb}}{V_{mb}} \times 100\% \quad (11)$$

where: $V_{unavailable\ RAM\ binder}$ = volume of unavailable recycled binder, $V_{RAP\ binder}$ = total volume of RAP binder, $V_{RAS\ binder}$ = total volume of RAS binder, $Availability_{RAP}$ = RAP recycled binder availability, $Availability_{RAS}$ = RAS recycled binder availability, V_{sb} = bulk volume of aggregate calculated on the basis of availability, $V_{se\ RAP}$ = effective RAP aggregate volume, and $V_{se\ RAS}$ = effective RAS aggregate volume, $V_{sb\ virgin}$ = bulk volume of virgin aggregate, V_{be} = effective binder volume in the mix, V_{mb} = total volume of mix, V_{ma} = voids in mineral aggregate volume and *VMA* = percent voids in mineral aggregate.

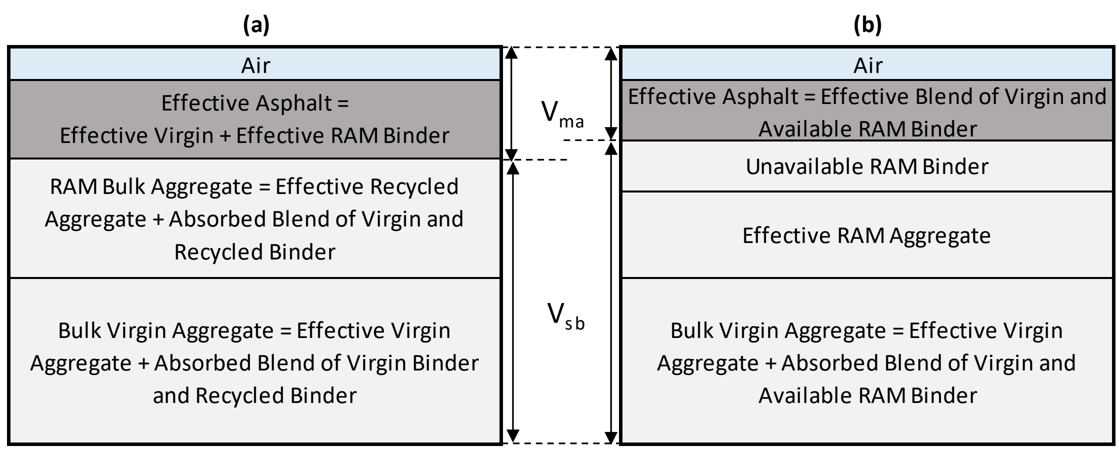

Figure 17. Phase diagrams according to (a) the specification and (b) redesigned mixes.

The ‘specified’ and ‘available’ volumetric properties of the NCDOT-approved mixture designs evaluated in this studies are shown in Table 12 and

Table 13 for the RS9.5B and RS9.5C mixtures, respectively. The specified values correspond to those calculated when assuming complete recycled binder availability and white curves to reflect the RAM aggregate gradation. The available values correspond to the properties calculated when considering the unavailable binder as part of the RAM bulk aggregate bulk, as conveyed by Equations (8) through (11), and using the black curves to reflect the RAM gradation. In addition, the total RBRs and effective RBRs (determined according to Equation (7)) are shown. The RAP binder availabilities used in the calculations were taken to be those from sieve analysis. The RAS binder availabilities were those inferred from the tracer-based microscopy results.

Table 12 and

Table 13 show that considering availability leads to substantial reductions in the calculated VMA, VFA, and DP of asphalt mixtures based on the comparison of specified and available quantities for a given mixture design. The VMA and VFA both decrease due to the inclusion of the unavailable recycled binder in the bulk aggregate volume. The reduction in the effective binder content and decrease in filler content when the RAM gradation is used in place of the recovered aggregate gradation yields a net decrease in the DP. Changes in DP, VMA, and VFA are most notable for the mixtures containing RAS and the RAP only mixtures with contents of 30 percent or more, suggesting considering availability is most important for high RAP content mixtures and mixtures containing RAS. The C15 mixture and B21 mixtures show only marginal differences when availability is considered; for example the available and specified VMAs differ by less than one percent. In contrast the specified versus available VMAs for the other mixtures are up to two percent in the higher RAM content mixtures. The mixtures generally still meet the NCDOT specification criteria when the recycled binder availability with the exception of the DP for several mixtures and the VMA for Mix C. However, the available VMAs do not reflect the typical VMAs in NCDOT approved mixtures, which are consistently higher than the minimum limit. Figure 18 shows box plots depicting the distribution of the VMAs in approved JMFs for RS9.5B and RS9.5C mixtures in NCDOT, which shows that mixtures in North Carolina are consistently designed with VMAs much higher than the specification minimum. Thus, considering availability in volumetric mixture design procedure may result in changes to the composition of high RAM content mixtures in North Carolina.

Comparison of the effective and total RBR values shows that considering availability results in a considerable reduction in the inferred RBR. The NCDOT requires a PG 58-28 virgin binder whenever a mixture contains RBR exceeds 30 percent and in any mixture containing RAS with an RBR that exceeds 20 percent (NCDOT 2020). Otherwise, a PG 64-22 virgin binder is required. While no adjustment to the virgin binder grade was made on the basis of availability herein, these specifications do suggest that the consideration of the effective versus total RBR for could have yielded a different virgin binder grade in many of the mixtures evaluated.

Table 12. Specified and Available Volumetric Properties for RS9.5B Mixes

Mix properties	Mixture ID						Specification limits
	B30	B15/5	C40	C30	C15	D30	
Total binder content (%)	6.7	6.7	6.3	6.3	6.4	6.0	--
Available binder content (%)	6.0	5.4	5.4	5.7	6.1	5.5	--
Virgin binder content (%)	5.2	4.6	4.1	4.6	5.6	4.7	--
Total RBR (%)	22.0	30.6	35.6	26.7	13.1	22.1	--
Effective RBR (%)	12.8	14.3	25.5	18.4	8.6	15.0	--
Specified VMA (%)	18.5	18.2	18.5	17.8	18.2	19.0	Min. 16.0
Available VMA (%)	17.5	**15.9**	17.0	16.7	17.6	18.1	
Specified VFA (%)	78.4	78.0	78.4	77.6	78.0	79.0	70 - 80
Available VFA (%)	77.1	74.8	76.4	76.0	77.3	77.9	
Specified DP	0.95	1.14	1.14	0.89	0.74	0.96	0.6 - 1.4
Available DP	0.66	0.92	**0.58**	**0.43**	0.52	0.67	

Table 13. Specified and available volumetric properties for RS9.5C mixes

Mix properties	Mixture ID		Specification limits
	A25/4	B21	
Total binder content (%)	6.3	6.3	--
Available binder content (%)	5.2	5.8	--
Virgin binder content (%)	4.4	5.3	--
Total RBR (%)	29.4	16.4	--
Effective RBR (%)	15.3	9.3	--
Specified VMA (%)	18.3	17.5	Min. 15.5
Available VMA (%)	16.3	16.8	
Specified VFA (%)	78.1	77.1	65 - 78
Available VFA (%)	75.4	76.1	
Specified DP	0.91	1.03	0.6 - 1.4
Available DP	0.77	0.82	

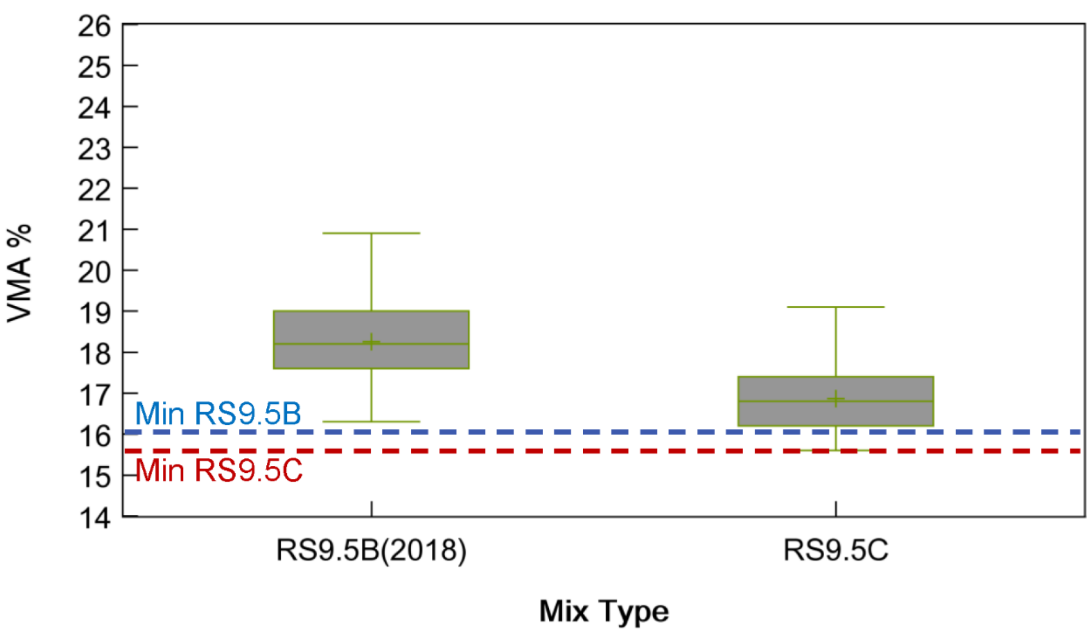

Figure 18. Distribution of VMAs in NCDOT mixtures (Underwood et al. 2021).

3.3.2. Composition of Current NCDOT 'Control' Mixture Designs and Comparative Mixture Designs Prepared on the Basis of Availability

Three of the NCDOT mixture designs evaluated in this study (A25/5, B15/5, and C40) were redesigned on the basis of the proposed changed in Table 11 with the exception of changes to virgin binder selection. The redesigns were prepared to achieve the intended effective binder content, and therefore VMA, specified in the control mixture design when accounting for availability. A given control and corresponding redesigned mixture contained the same virgin aggregate, RAM materials, RAM aggregate stockpile proportions, and virgin binder.

The RAP binder availabilities used when redesigning and evaluating the composition of the mixtures were those determined from sieve analysis. Given that a procedure has not been established to determine recycled binder availability directly from the RAS, the RAS binder availabilities used were those obtained from the tracer-based microscopy results.

Redesigning a given control mixture involved the following steps. The available VMA in the control mixture was calculated using Equations (8), (9), and (11). Based on the difference between the specified VMA (calculated on the basis of 100 percent availability) and the available VMA (calculated according to the definition of V_{sb} in Equation (9)), the virgin aggregate stockpile proportions were adjusted to increase the available VMA to the specified VMA while still meeting the NCDOT's gradation requirements when using the RAM aggregate gradation; these adjustments were guided by the Bailey method (Vavrik et al. 2002). Then, asphalt mixtures were prepared at four asphalt content using the design compaction level given the respective control mixture designation (i.e., RS9.5B or RS9.5C). The results were used to determine the asphalt content that yielded four percent air voids.

The black and white gradations for the three RAP sources used in the control mixtures are presented in Figure 19 and the corresponding, calculated availabilities are presented in Table 14. The black curves exhibit much coarser gradation with minimal amounts of fine materials compared to the white curves, indicating that a substantial portion of the fine particles are agglomerated (as also detailed in Section 3.2). Also, it can be observed that the white curves for the three RAP sources are similar for particle sizes 1.18 mm and smaller, indicating similar fine aggregate gradations with greater distinction in the coarser aggregate sizes. RAP A exhibits the smallest differences between the white and black curves of the three RAP materials evaluated, which translated into a notably higher calculated recycled binder availability of 82 percent compared to 62 percent and 52 percent for RAP C and B, respectively (which is further discussed in Section 3.2).

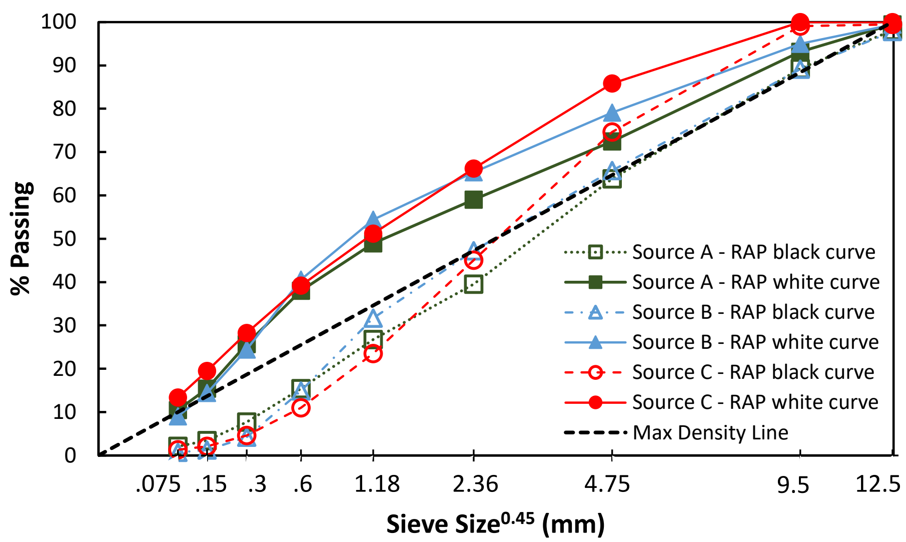

Figure 19. Black and white curves for the three RAP sources.

Table 14 also presents the RAS binder availabilities for Sources A and B, which were inferred from tracer-based microscopy. Tracer-based microscopy results of Mixes A25/4 and B15/5 both yielded overall average recycled binder contributions of approximately 40 percent as presented in Section 3.1.2. The implications of the measured recycled binder availabilities on the total and available binder content of the RAM is shown in Table 14. The availability values listed in Table 14 were used when interpreting the effective RBR and available volumetric properties of the control and redesigned mixtures.

Table 14. Recycled Binder Availabilities

Mix	Material	Total binder content (%)	Availability (%)	Available binder content (%)
C40	RAP	5.7	62	3.5
A25/4	RAP	4.0	82	3.3
	RAS	19.0	0	0.0
B15/5	RAP	5.0	52	2.6
	RAS	22.0	30	6.6

The black and white curves for the two sources of RAS in Figure 20. The extension of the sieve analysis method to determine recycled binder availability developed herein to RAS may prove challenging since Figure 20 shows a smaller difference between the black and white curves for Source A, whereas the microscopy analysis showed a higher availability for Source B. RAS contains a considerably higher P200 content than RAP. Therefore, agglomerates of mastic alone likely exist in RAS. Consequently, availability calculations may require adjustment to consider binder rather than mastic coatings of aggregate particles. This requires an assumed particle size distribution for the P200 material; the feasibility of such an approach merits exploration in future work.

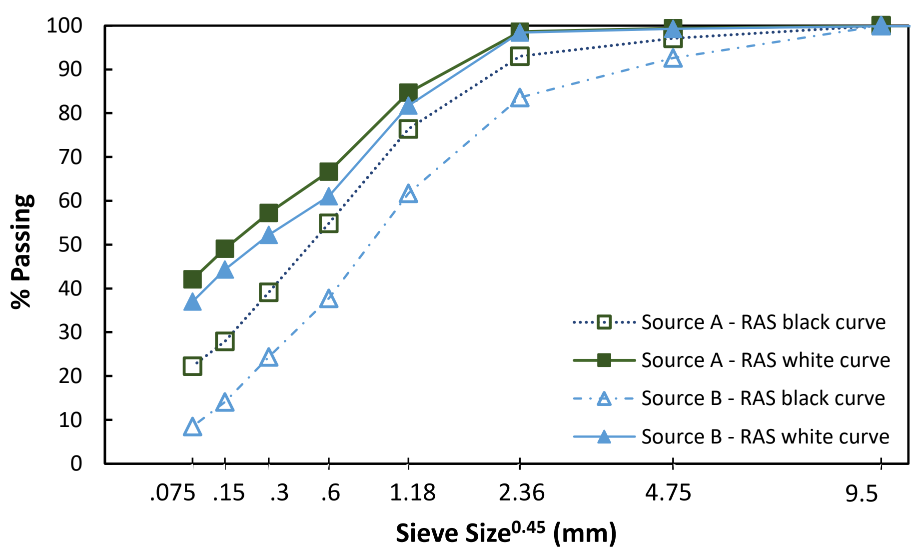

Figure 20. Black and white curves for the two RAS sources.

Figures 21, 22 and 23 show the control and redesigned gradations, conveyed when using both the black and white curves to reflect the RAP and RAS gradations, for Mixes C40, A25/4, and B15/5, respectively. The gradations using the black curves are notably coarser than those using the white curves to reflect the RAM gradation. In some cases, using the black gradation results in control mixtures failing to meet the NCDOT gradation limits at the 0.075 mm and/or 2.36 mm sieves. All of the redesigned gradations meet the NCDOT specifications when using the black curves to reflect the RAM gradation. However, in some cases, the redesigned stockpile proportions fail to meet the current NCDOT specifications when using the RAM white curves. Collectively, these results highlight that using black versus white curves can result in substantial changes to the inferred gradation. The impact of using the RAM black curves instead of the white curves was most pronounced for the Mix C40 since it has the highest RAM stockpile percentage, equal to 40 percent. Figure 21 shows that the redesigned gradation when using the RAP black curve for Mix C40 was established to be close to the corresponding control gradation when using the RAP white curve. Figures 22 and 23 show that Mixes A25/4 and B15/5, which contain both RAP and RAS, were redesigned with a finer gradation to yield a higher VMA by shifting the gradation away from the maximum density line.

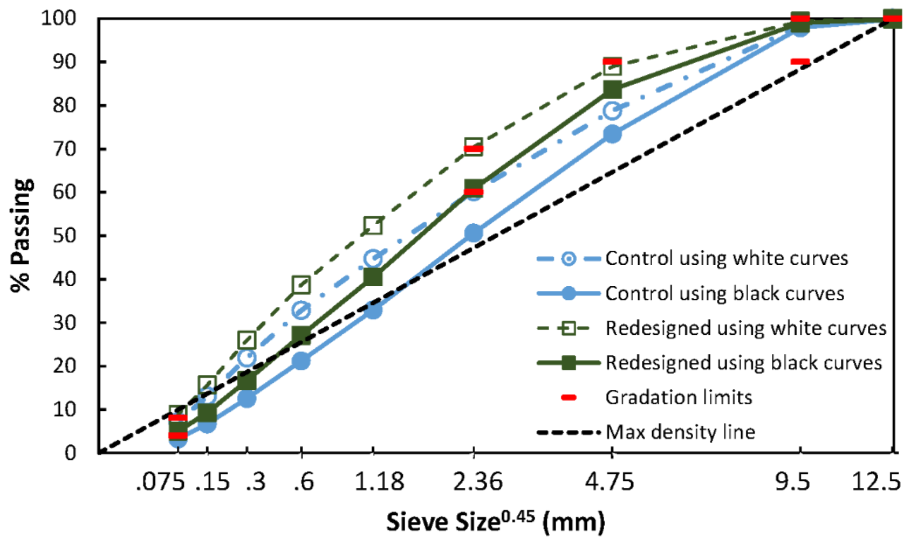

Figure 21. Control and redesigned gradations for Mix C40.

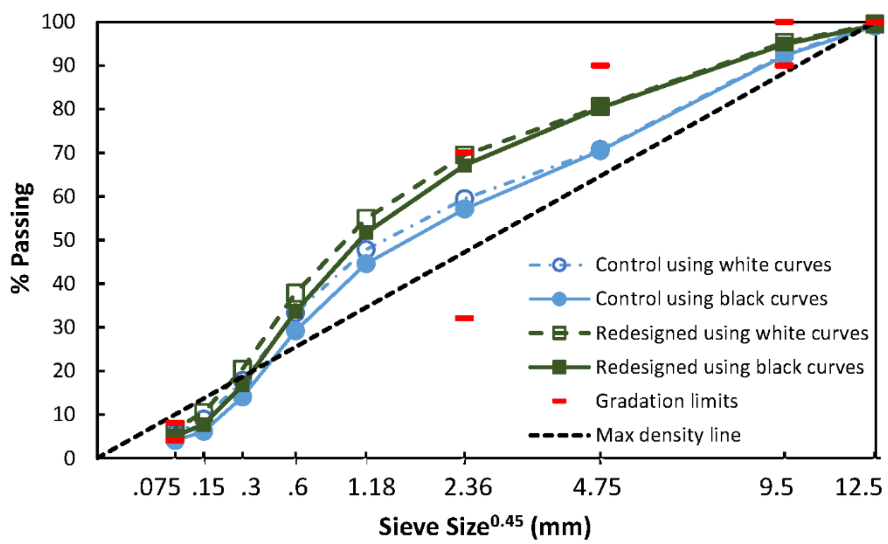

Figure 22. Control and redesigned gradations for Mix A25/4.

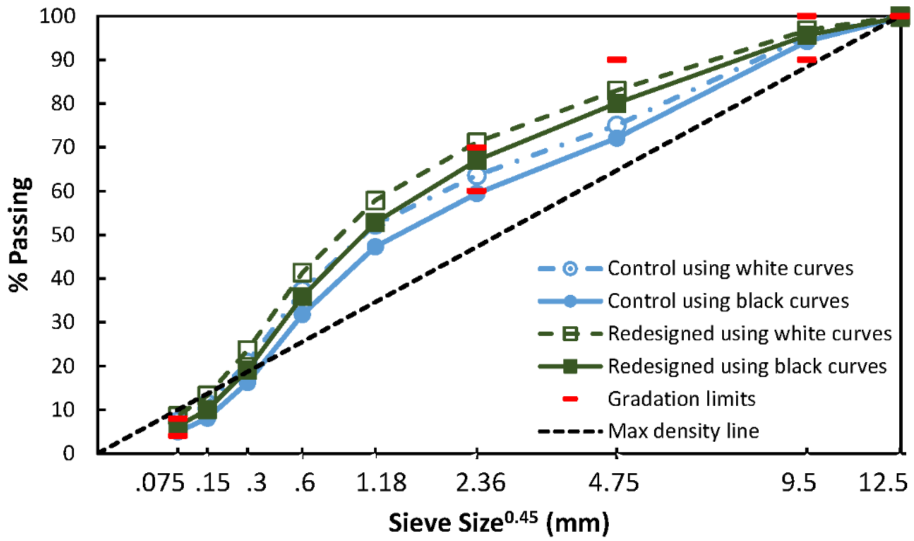

Figure 23. Control and redesigned gradations for Mix B15/5.

The optimum binder content that yields four percent air voids at the design compaction level required by the NCDOT (2020) was determined for each redesigned gradation to complete the mixture redesign. The 'specified' and 'available' volumetric properties of the control and redesigned mixtures were then tabulated. The specified values correspond to those calculated when assuming complete recycled binder availability and white curves to reflect the RAM aggregate gradation. The available values correspond to the properties calculated when considering the unavailable binder as part of the RAM bulk aggregate bulk, as conveyed by Equations (8) through (11), and using the black curves to reflect the RAM gradation. In addition, the total RBRs and effective RBRs (determined according to Equation (1)) were evaluated. Tables 15, 16 and 17 show the collective properties of the control and redesigned Mixes C40, A25/4 and B15/5, respectively. The NCDOT specification limits for each property, where applicable, are also presented. Figure 24 also graphically presents the (a) VMA, (b) VFA, (c) DP and (d) RBR values of the control and redesigned mixes for both the specified and available scenarios.

Tables 15, 16 and 17 show that the available VMAs in the redesigned mixtures are within ± 0.4 percent of the VMA in the corresponding control mixture and thus the redesigned gradations achieved the intended VMA in the control mix when availability is properly accounted for. Recycled binder availability does not affect the calculated air void content of an asphalt mixture. However, the increase in VMA in the redesigned mixtures compared to the control mixtures resulted in higher virgin binder contents to achieve the design air void content, as shown in Tables 15, 16 and 17. For Mix A25/4, a slight increase in the VMA (18.2 to 18.5) caused the available VFA to go slightly over (78.4) the specification limit, since the control mix had the VFA already on the upper limit (78.0). All other available volumetric properties of the redesigned mixtures meet the NCDOT specifications. The DP values of the redesigned mixtures are notably higher than the control mixtures. Moreover, the total and effective RBRs for the redesigned mixtures are lower than the respective control mixtures.

Table 15. Properties for the Control and Redesigned Mix C40

Mix properties	Mix C40		
	Control	**Redesign**	**Specification limits**
Total binder content (%)	6.3	7.0	--
Available binder content (%)	5.4	6.1	--
Virgin binder content (%)	4.0	4.8	--
Total RBR (%)	35.9	31.6	--
Effective RBR (%)	25.8	22.3	
Specified VMA (%)	18.5	19.6	Min. 16.0
Available VMA (%)	17.0	18.1	
Specified VFA (%)	78.4	79.6	70 - 80
Available VFA (%)	76.4	78.0	
Specified DP	1.14	1.29	0.6 - 1.4
Available DP	**0.58**	0.80	

Table 16. Properties for the Control and Redesigned Mix A25/4

Mix properties	Mix A25/4		
	Control	**Redesign**	**Specification limits**
Total binder content (%)	6.3	6.9	--
Available binder content (%)	5.2	6.0	--
Virgin binder content (%)	4.4	5.2	--
Total RBR (%)	29.4	24.4	--
Effective RBR (%)	15.3	13.3	
Specified VMA (%)	18.2	20.2	Min. 15.5
Available VMA (%)	16.4	18.5	
Specified VFA (%)	78.0	**80.2**	65 - 78
Available VFA (%)	75.6	**78.4**	
Specified DP	0.90	0.92	0.6 - 1.4
Available DP	0.74	0.79	

Table 17. Properties for the Control and Redesigned Mix B15/5

Mix properties	Mix B15/5		
	Control	**Redesign**	**Specification limits**
Total binder content (%)	6.7	7.5	--
Available binder content (%)	5.4	6.3	--
Virgin binder content (%)	4.6	5.6	--
Total RBR (%)	30.6	24.5	--
Effective RBR (%)	14.3	11.1	
Specified VMA (%)	18.2	20.0	Min. 16.0
Available VMA (%)	**15.9**	18.0	
Specified VFA (%)	78.0	80.0	70 - 80
Available VFA (%)	74.8	77.8	
Specified DP	1.14	1.20	0.6 - 1.4
Available DP	0.92	1.00	

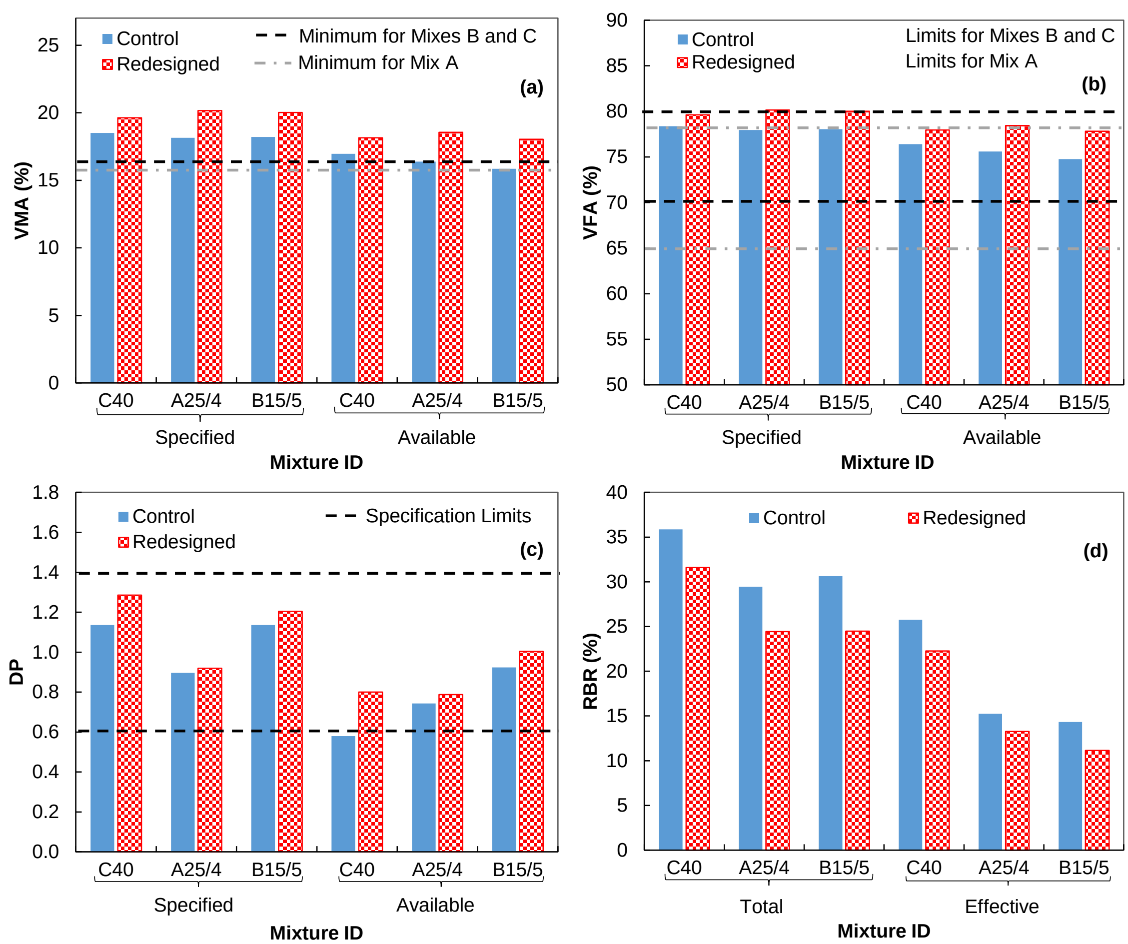

Figure 24. Composition of the control and redesigned mixtures in terms of (a) VMA, (b) VFA, (c) DP and (d) RBR considering the specified values (assumed 100 percent availability) and those recalculated on the basis of the measured availability.

3.3.3. Performance of the Control versus Redesigned Mixtures

The cracking and rutting performances were assessed by means of the CT_{index} and APA rut depth, respectively. Both control and redesigned mixes were tested and the results compared to evaluate the revised mixture design method proposed herein. Figure 25 presents the CT_{index} results of the control and redesigned mixes. Four specimens were tested for each mix and design. The results suggest a significant improvement in the cracking performance for the redesigned mixes compared to the respective control mixtures, especially for the two mixes containing RAP and RAS. The redesigned Mixes A25/4 and B15/5 exhibited CT_{index} that were 60.4 percent and 73.5 percent higher than the respective control mixture. For the Mix C40, which contained only RAP, the CT_{index} of the redesigned mixture was 20.5 percent higher than the corresponding control mix, which is a smaller but still noticeable improvement.

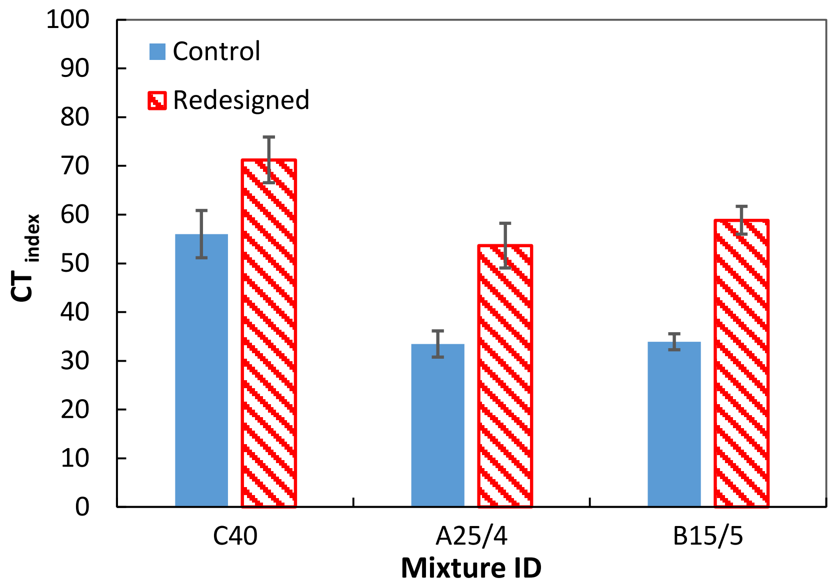

Figure 25. CT_{index} results for the control and redesigned mixes.

The APA rut depth obtained for the control and redesigned mixes are presented in Figure 26, along with the NCDOT specification limits for the two mix types. The results show a slight increase in the permanent deformation susceptibility in the redesigned mixtures compared to the respective control mixtures, which is expected due to the increased virgin binder content. However, all the mixes still fall well below the maximum allowable rut depth. These results suggest adjustment of the virgin binder grade in light of availability considerations may not be required to maintain adequate rutting resistance.

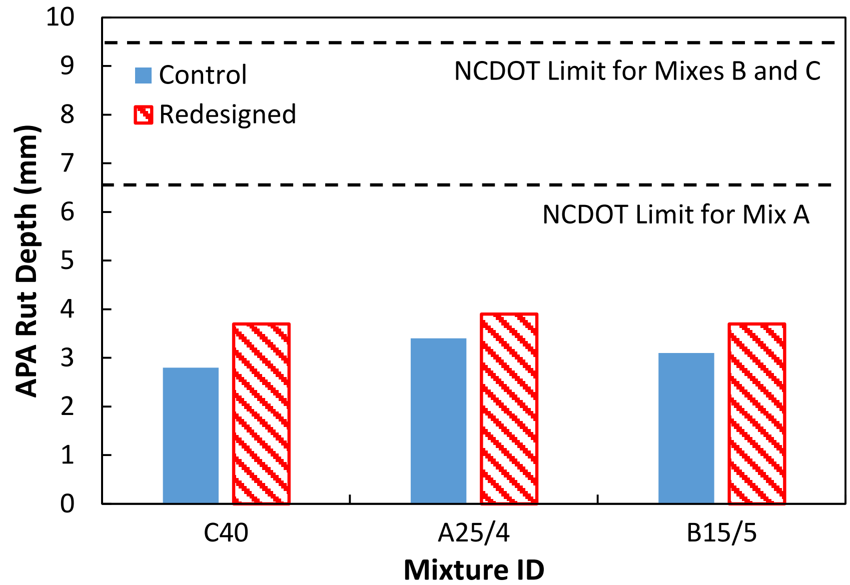

Figure 26. APA rut depth for the control and redesigned mixes.

Cracking and rutting are two major distresses for asphalt mixes and are affected differently by the virgin binder content. Mixes with higher binder content tend to perform better for cracking, and mixes with low binder content tend to perform better for rutting. In other words, for a balanced mix, the cracking performance determines the minimum binder content, while the rutting performance determines the maximum. The redesigns resulted in a higher virgin binder content due to the increased VMA. As a consequence, the cracking performance was improved significantly, and in spite of the increased permanent deformation, the rut depth was still below the critical thresholds. These results suggest that considering recycled binder availability within volumetric mixtures design could result in overall improvements to pavement performance.

4. PROPOSED CHANGES TO VOLUMETRIC MIXTURE DESIGN PROCEDURES

If the laboratory findings of this study are verified to be valid for plant-produced mixtures, the recommended changes to the NCDOT volumetric mixture design procedure are:

- Measure and use the gradation of RAP and RAS materials (i.e., black curves) to design and evaluate aggregate blends containing RAM, including when calculating the DP of an asphalt mixture.
- Quantify source-specific RAP binder availability using the sieve analysis procedure developed through this project, which requires both the RAP and recovered RAP aggregate gradation. An assumed RAP binder availability of 60 percent is recommended based on the collective results of this study if source-specific RAP binder availability is unknown.
- Assume the recycled binder availability of RAS equals 30 percent since this is the maximum RAS binder availability observed from the two sources evaluated in this study and an implementable procedure to quantify RAS availability does not exist. It is recommended that the NCDOT refine this assumption in the future based on tracer-based microscopy investigations of asphalt mixtures prepared with additional RAS sources.
- Standardize RAM and virgin aggregate preheating procedures for the laboratory fabrication of asphalt mixtures to minimize variability in recycled binder contribution imparted by different material preheating practices.
- Consider the unavailable recycled binder in asphalt mixtures as part of the bulk aggregate volume according to the calculation procedure outlined in this study. This negates the need for the bulk specific gravity of the RAM aggregate for volumetric mixture design, which constitutes considerable uncertainty.
- It is recommended that the NCDOT also consider the ‘effective’ RBR, discrediting unavailable recycled binder, when assessing asphalt mixtures with respect to RAM content. The effective RBR is lower than the total RBR in the case of partial recycled binder availability, which suggests a potential need for revising the maximum RBR limits permitted in RAM mixtures on the basis of the effective RBR. Consideration of the effective RBR may also warrant reconsideration of virgin binder selection for high RAM content mixtures. The implications of the effective RBR on performance were not directly evaluated in this study. Therefore, future research to investigate the consequences of using the effective RBR rather than total RBR for virgin binder selection and defining maximum permissible RAM contents is warranted prior to implementing changes to specifications pertaining to RBR.

5. Long-term Monitoring Plan to Validate the Revised Mixture Design Procedure

Laboratory and field measures are proposed to validate the proposed revisions to volumetric mixture design procedures. Validation of the research products should include the production of a plant-produced asphalt mixture with incorporation of a titanium dioxide-modified virgin binder. The titanium dioxide is a fine powder that can be mixed with the virgin binder like other powder additives. The plant-produced loose mixture could be compacted in the laboratory and subjected to microscopy to determine the recycled binder contribution. In addition, the component aggregate and binder will be acquired from the plant and used to fabricate laboratory samples for microscopy analysis to compare the recycled binder contribution of laboratory- and plant-produced asphalt mixtures.

It is recommended that laboratory validation include comparative sieve analysis and tracer-based microscopy analysis of mixtures prepared with additional RAP sources to validate the procedure and better understand variation in the recycled binder availability of RAP sources that encompass a broad range in stockpiling and processing characteristics within the state. Tracer-based microscopy of asphalt mixtures prepared with additional RAS sources is also recommended to better understand variation in RAS binder availability and assess whether or not the assumption of an assumed availability across different sources is appropriate.

Subsequently, field validation of the revised mixture design procedure is recommended. Four field sites are proposed in each region of North Carolina (i.e., coastal, piedmont, and mountains): two RS9.5B sites and two RS9.5C sites. Evaluating three regions is suggested since the originating material streams might differ as well as the contractor practices. In reality, it may be sufficient to select a range of contractors who are known to handle recycled materials differently. One of each of the two RS9.5B and RS9.5C sites in each region should include RAP only mixtures and the other should include both RAP and RAS. It is suggested that all the selected mixtures have a minimum RBR of 0.3. Control sections containing a surface mixture designed according to the NCDOT's current practices and comparative sections designed using the same component materials and recycled material stockpile proportions as the control mixture but designed according to the revised procedure should be included within each site.

Prior to constructing the field sections, the control and comparative mixture designs should be prepared in the laboratory. Rutting and cracking performance testing of the control and comparative mixtures is recommended to initially screen for potential field performance differences. In addition to using these mixtures to validate the proposed revisions to mixture design procedures, design alternatives that include a PG 64-22 binder instead of the current specified PG 58-28 binder could be used evaluated using laboratory performance testing to study the implications of basing virgin binder grade selection on the effective versus total RBR. Based on the outcome of this evaluation, the appropriate virgin binder for the comparative mixture used in the field could be selected.

Field construction data collection and long-term pavement condition monitoring should follow the long-term pavement performance plan detailed in Appendix F of NCDOT RP 2019-20 (Underwood et al. 2022). The long-term performance of the adjacent control and comparative sections could be used to evaluate the implications of the proposed revisions on distress evolution and thus, service life.

6. Conclusions and Recommendations

6.1. Conclusions

- Recycled material agglomerations exist in asphalt mixtures that prohibit complete recycled binder availability in RAP and RAS materials.
- The fatigue fracture surfaces of the asphalt mixtures do not contain RAM agglomerations, suggesting that the fracture initiates and propagates around the agglomerations. These findings suggest that the agglomerations can be considered black rocks for the purposes of volumetric mixture design.
- Tracer-based microscopy measurements of local recycled binder contribution along the fracture surface of the asphalt mixtures evaluated indicate the presence of a partial blend of recycled and virgin binders that is rich in virgin binder. The recycled binder contribution in the mixtures evaluated ranged from roughly 40 to 90 percent. Local binder contribution values vary within a given mix, which suggests incomplete blending of the virgin binder and available recycled binder. The standard error of the local recycled binder contribution measurements, indicative of the degree of blending, was comparable for all mixtures evaluated suggesting that considering recycled binder availability.
- Recycled binder contribution and degree of blending inferences made from tracer-based microscopy measurements of the fatigue fracture surface and areas without the recycled material agglomerations in bulk mixture specimens are generally very similar, indicating that bulk specimens can be used to investigate recycled binder contribution without the need for cyclic fatigue testing and epoxy embedment.
- The RAP binder contribution in asphalt mixtures do not exhibit clear trends with respect to the high-temperature grade of the RAP binder or asphalt mixture RAP content based on tracer-based microscopy investigations. However, differences in recycled binder variability were observed among the four different RAP sources evaluated with values spanning approximately 50 to 90 percent.
- The comparison of the gradation of RAP and recovered RAP aggregate provides a measure of the extent of agglomeration that exists within asphalt mixtures. Tracer-based microscopy measurements were generally in good agreement with the estimations of recycled binder availability derived from the sieve analysis procedure developed in this study. The sieve analysis method requires only equipment found in a basic asphalt mixture testing laboratory as it requires neither extraction nor recovery of the asphalt binder, nor asphalt binder testing. This introduces the potential for routine testing of RAP stockpiles for asphalt mixture design as well as potentially for asphalt plant quality control and acceptance laboratories.
- The recycled binder availabilities of the two RAS sources evaluated in this study were estimated to be zero and 30 percent from tracer-based microscopy.
- RAM and virgin aggregate preheating procedures can impact the recycled binder contribution in laboratory-fabricated asphalt mixture samples and thus, it is recommended that the NCDOT specify the material preheating procedure to minimize mixture variability imparted by the laboratory fabrication procedure. The virgin binder may impact recycled binder contribution in an asphalt mixtures somewhat but additives and RAP age level had generally insignificant effects.
- Considering recycled binder availability has several important implications to the design and inference of asphalt mix composition for the purpose of volumetric mixture design. The

unavailable recycled binder bound within agglomerations should be considered as part of the bulk aggregate. This change has implications to the calculation of the VMA, VFA, DP of asphalt mixtures. Furthermore, the RAM gradation (i.e., black curve) may better reflect the gradation of RAM in a mix compared to the recovered aggregate (i.e., white curve) given that agglomerates may act as 'black rocks'.

- The cracking performance improved significantly for the three NCDOT approved 'control' mixtures redesigned on the basis of measured recycled binder availability to achieve an available VMA equal to the intended VMA specified in the corresponding control mixture. The redesigned mixtures contained higher virgin asphalt contents than the respective control mixtures. The permanent deformation was higher in the redesigned mixes compared to the control mixes. However, the rutting performance of the redesigned mixtures was still satisfactory, falling well below the maximum allowable APA rut depth specified by the NCDOT. Thus, the methods used to redesign mixes containing RAM proposed in this study may serve as a means to improve mixture cracking performance without substantially impairing rutting performance.

6.2. Recommendations

- Quantify the presence and size of RAM agglomerations using tracer-based microscopy of plant-produced asphalt mixtures to verify that the findings of the laboratory-produced mixtures obtained in this study are applicable to plant-produced asphalt mixtures.
- If the laboratory findings of this study are verified to be valid for plant-produced mixtures, measure and use the gradation of RAP and RAS materials (i.e., black curves) to design and evaluate aggregate blends containing RAM, including when calculating the DP of an asphalt mixture. Quantifying the gradation of RAM stockpiles as part of quality assurance and control practices may also be a means to improve the consistency of recycled asphalt mixtures. Consider implementing the sieve analysis procedure to estimate source-specific RAP binder availability for volumetric mixture design. An assumed RAP binder availability of 60 percent is recommended based on the collective results of this study if source-specific RAP binder availability is unknown.
- It is recommended that the recycled binder availability of additional RAS sources be measured using tracer-based microscopy of asphalt mixtures to gain an improved understanding of RAS binder availability and its variation among sources. In the interim, an assumed RAS binder availability of 30 percent (i.e., the maximum value of the two sources evaluated) is recommended.
- Standardize RAM and virgin aggregate preheating procedures for the laboratory fabrication of asphalt mixtures to minimize variability in recycled binder contribution imparted by different RAM temperatures.
- Future research is recommended to investigate the impacts of the partial recycled binder availability on the selection of an appropriate virgin binder grade and maximum permitted RBRs since considering availability lowers the effective RBR in the mixture.
- Consider implementing the long-term monitoring plan detailed in Section 5 to address the above recommendations and validate the proposed revisions to volumetric mixture design procedures.

7. Implementation and Technology Transfer Plan

The Materials and Tests Unit of the NCDOT are the primary users of the outcomes of this research. The proposed changes to volumetric mixture design procedures can be integrated into NCDOT specifications without the need for substantial training or new equipment. An example of volumetric mixture design calculations according to the proposed mixture design procedures is provided in Appendix C to help facilitate training. The research team recommends that the NCDOT consider allocating resources to implement the long-term monitoring plan outlined in Section 5.

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APPENDIX A: DETAILED LITERATURE REVIEW

Brief History of Asphalt Recycling

Recycling of asphalt materials has existed since the very early days of asphalt paving, with the Warren Brothers experimenting with recycling as early as 1915 (NCHRP 1978). Despite very early trials, recycling asphalt materials was not common throughout the first part of the twentieth century, and only intermittent projects were attempted until the 1973 oil crisis when the price of asphalt spiked rapidly (Epps et al. 1980). Research on high-recycled content mixtures commenced immediately, with at least half of the states trying some form of recycling by 1976 (NCHRP 1978). While most of those states were experimenting with in-place recycling or surface recycling, six were using central plant recycling. These central plant experiments have evolved into modern use of reclaimed asphalt pavement (RAP) in an asphalt plant.

The benefits of central plant recycling over in-place recycling methods noted in 1978 include: structural improvements, increased quality control, better treatment of pavement distresses, elimination of reflective cracking, potentially improved frost-susceptibility, improved skid resistance, easily altered geometrics, better control of the additional binder and aggregate, and improved ride quality. The only drawbacks noted were traffic disruption and air quality at the plant (NCHRP 1978). The early central plant recycling projects focused on maximizing RAP usage, with only a few projects using less than 50 percent RAP, and several attempting 100 percent RAP. These high levels of recycled material contents combined with limited options for plant modifications and pollution control caused air quality problems. It was assessed that most plant configurations could not achieve satisfactory air quality when producing 100 percent RAP mixtures. Most drum plants needed 30 to 50 percent new aggregate, additional moisture, reduced production rates, or more control of exit temperatures to achieve satisfactory air quality (NCHRP 1978). Because adding additional moisture and reducing production rates were not ideal, the addition of new aggregate became the prevalent form of recycling.

Two years later, Epps et al. (1980) published *Guidelines for Recycling Pavement Materials* in response to the rapid changes in the field of pavement recycling that rendered the NCHRP synthesis from 1978 outdated. The guidelines focused on conducting a preliminary analysis of recycling, selecting the appropriate methodology and technology to achieve the desired results, and developing a methodology to evaluate projects and compare recycling with traditional mixtures. They noted issues which have yet to be resolved, including the assumption that the field mixing process results in complete blending of recycled and virgin binders, and the lack of methods to determine the quality of a recycling modifier. Their laboratory work focused on a 70 percent RAP mixture, which was low compared to the early projects but is extremely high by modern standards. While the early projects developed substantial understanding of the problems faced with high-recycled mixtures, many of the conclusions made have since been proven invalid. Epps et al. (1980) predicted that the 30 to 40 percent new aggregate needed for successful operation in a drum plant would go down as technology and experience improved; however, that has not been the case, and in 2009, RAP usage was at an average of about 12 percent across the US, although that has been increasing (Copeland 2011).

When the Strategic Highway Research Program (SHRP) began in the late 1980s, RAP and other recycled materials were not directly considered. Consequently, there has been a lack of uniform guidance in how to account for RAP in Superpave volumetric mixture designs and virgin binder selection. Interim guidelines for the incorporation of RAP into mixture design were provided in the late 1990s, which were followed in the early 2000s with the more thorough guidelines developed under NCHRP 09-12 (McDaniel et al. 2000). The NCHRP 09-12 guidelines were eventually incorporated into AASHTO M 323 and R 35 (Copeland 2011). While some states have followed the guidelines from NCHRP 09-12, many others have developed their own procedures for handling and specifying recycled materials.

Asphalt prices spiked again in 2006 and yet again in 2008, and the availability of quality aggregates was also becoming an issue in many areas, resulting in a resurgence of interest in recycling (Copeland 2011). In 2007, the United States Environmental Protection Agency (EPA) also began a campaign to reduce construction and demolition debris in landfills, increasing the disposal costs of products like asphalt shingles (Farris 2016). While shingles had been used in asphalt pavements on a limited basis for decades, the EPA initiative made recycling asphalt shingles more cost-effective. Shingles also have much higher asphalt binder content than RAP, often in the range of 16 to 25 percent by weight, compared to the 3 to 6 percent in most RAP stockpiles. Consequently, relatively small amounts of RAS can replace a greater quantity of virgin binder. However, the asphalt binder contained within RAS is typically much stiffer than RAP binder, even more so for post-consumer RAS (PCRAS), which has experienced a service life of aging beyond that of manufacturer waste RAS (MWRAS). This increased stiffness limits the amount of RAS that can be incorporated into asphalt mixtures without compromising cracking performance.

Present Challenges to Increasing Recycled Material Use

Four main challenges to designing mixtures with RAP in the laboratory were identified by Zhou et al. (2011): evaluating the blending of binders, determining the bulk specific gravity of RAP aggregates, simulating plant handling in the laboratory, and selecting mixing and compaction temperatures. Although these questions were posed specifically for RAP, they also extend to RAS. These questions are not new to recycled materials, with the uncertainty in binder blending noted as far back as 1980 (Epps et al. 1980); however, definitive answers remain elusive. In light of the present challenges, the following provides a review of the current laboratory procedures employed with recycled materials, handling of recycled materials in plant operations, the effect of material and processing factors on blending between recycled and virgin materials, measurements of blending in reclaimed asphalt mixtures, and the simulation of blending using predictive models.

Laboratory Procedures with Recycled Materials

Mix Design Methods with Recycled Materials

Most RAP mix design methods assume complete binder blending, including the Superpave method (Epps et al. 1980, AASHTO R 35). This assumption simplifies the design procedure and does not appear to pose a problem in mixtures with low RAP contents (McDaniel and Anderson 2001). McDaniel and Anderson (2001) evaluated whether RAP behaves as a black rock by comparing mixtures made with different blending conditions. One mixture was made following typical practice, another was considered “total blending” as the RAP binder was extracted and mixed with the virgin binder, and the last was considered “black rock” as it was made with stripped RAP aggregates from the ignition oven and no additional binder. Superpave shear tests and indirect tensile strength tests were conducted on all three mixtures. At low RAP contents, there were negligible differences in results for all three mixtures, but at higher RAP contents the “black rock” scenario deviated from the other two. Based on the results of the testing, they concluded that RAP does not behave as a black rock; however, they were also not convinced that complete binder blending ever occurs. Therefore, the assumption of complete blending poses a problem in mixtures with RAS and/or and higher RAP contents as the recycled binder is contributing more to the mixture (Farris 2016, McDaniel and Anderson 2001). Blending charts developed in NCHRP 09-12 for virgin binder grade selection were based on complete blending and were found to work well for mixtures up to 40 percent RAP. Above 40 percent RAP, the linear blending chart approximation breaks down, possibly due to incomplete blending (McDaniel and Anderson 2001). In these mixtures, it is likely that not all the binder in the recycled material is contributing to the mixture, and thus the resulting mixture may have insufficient asphalt if a volumetric mix design procedure is used with an assumption of complete blending (AASHTO PP 53, Stroup-Gardiner 2016).

A survey of state agencies conducted in 2019 indicates that 9 out of 38 respondents assume partial availability in their mixture design procedures (Epps Martin et al. 2020[a], Abdelaziz et al. 2021). Given the lack of an accepted method to quantify recycled binder availability from RAM, these nine agencies currently use a single RAP recycled binder availability value and a single (often distinct) RAS recycled binder availability value, irrespective of the source. They adjust their volumetric mixture design procedures by either reducing credit given to recycled materials when calculating the total binder content of the mix or making an ad hoc adjustment to the virgin binder content after performing volumetric mixture design (Epps Martin et al. 2020[a]). Ad hoc adjustments to increase the asphalt content of the mixture after volumetric mixture design may improve cracking resistance but simultaneously compromise rutting susceptibility.

One example is the Georgia DOT Corrected Optimum Asphalt Content (COAC) method developed in 2013 based on experimental results that suggested incomplete blending in RAP mixtures (NCAT 2013). They heated RAP and observed the consistency and coating of binder on the RAP particles. Visual observation indicated that very little binder transfer occurs during dry mixing. After observing the RAP, the binder content was measured by ignition oven, and the clean RAP aggregates were collected. They then added virgin binder back to the clean RAP aggregates, in increments of 0.25 percent until they estimated that original RAP consistency was reached. The difference between the virgin binder content and the RAP binder content at the same consistency was evaluated as the effective asphalt content, and eventually an average effective asphalt content of 75 percent was selected (NCAT 2013). The COAC method has been formalized in Georgia DOT's Standard Operating Procedure 2, Appendix D, which is used to calculate the COAC for mixtures with RAP and PCRAS (Georgia 2019). To calculate the COAC, the mixture is first designed according to AASHTO R 35 with the assumption of complete blending, and the 'standard' RAP binder contribution is calculated based on the total mixture weight. Then, the Credited Asphalt Content is calculated as 60 percent of the standard binder contribution, and the Not Credited Asphalt Content (NCAC) is the remaining 40 percent. Additional virgin binder is added in the amount of the NCAC to compensate, with no adjustment to the other volumetric properties. The additional asphalt binder may impact the Superpave volumetric requirements of the final mixture, especially the voids filled with asphalt, as the original mixture was based on a lower asphalt content.

Balanced mix design (BMD) procedures integrate cracking and rutting tests into the mixture design process to ensure adequate performance is achieved. BMD offers a means to alleviate concerns associated with uncertainty in the effects of the assumed recycled binder availability since performance is directly quantified (Zhou et al. 2011). Four BMD approaches are outlined in AASHTO PP 105-20. Approach A constitutes the simplest and most conservative approach where performance testing is used to verify if the volumetric mixture design yields adequate performance. Approach D constitutes the most complex approach where the mix optimization is solely based on performance measures with no volumetric property requirements. The majority of implemented BMD procedures follow Approach A (NAPA 2021). Furthermore, estimates of volumetric properties typically guide establishment of the trial mixtures in the other BMD approaches. Consequently, an improved understanding of recycled binder availability may enable the design of mixtures with higher recycled contents that meet performance requirements.

Determination of the Bulk Specific Gravity of Recycled Aggregates

To perform volumetric mixture design with recycled materials, the bulk specific gravity of the recycled material aggregate must be quantified. The determination of RAP aggregate bulk specific gravity has been studied and most agencies follow a unified procedure. Intuitively, the bulk specific gravity of RAP aggregate should be easy to measure by recovering the RAP aggregate using the ignition oven or solvent extraction. However, solvent extraction can leave trace amounts of residual binder on the aggregates which confounds the results. In addition, aggregate recovered after conducting ignition oven testing has been found to produce unreliable results, which may be due to the high dust content of processed RAP that can coat the coarser fine aggregates and pose challenges with bulk specific gravity testing (Copeland 2011, AASHTO T 84). Construction records that document the RAP aggregate specific gravity can be obtained if the RAP source is traceable. However, sources are generally untraceable. In 2011, the FHWA recommended using the theoretical maximum specific gravity measured by AASHTO T 209 and an assumed absorption value based on typical local values to estimate the bulk specific gravity of RAP aggregate (Copeland 2011). The procedure proposed by the FHWA is now included as a nonmandatory note in AASHTO R 35. It is not without limitations but is implemented by most state agencies given the challenges in obtaining reliable measurements of bulk specific gravity on recovered aggregate samples (Stroup-Gardiner 2016).

The aggregate within RAS is not a ‘natural’ aggregate; rather, the aggregates are coated with ceramics for waterproofing, resulting in a spherical particle with negligible absorption (Farris 2016). Thus, the bulk specific gravity and effective specific gravity of RAS aggregate are equivalent. Therefore, both AASHTO PP 53 and PP 78 recommend using the effective specific gravity without correction as the bulk specific gravity of RAS aggregate until a more accurate method becomes available (AASHTO PP 53, AASHTO PP 78).

Binder Grade Selection with Recycled Materials

The standard procedure for selecting the virgin binder performance grade (PG) in mixtures containing RAP that is included in AASHTO M 323 was developed under NCHRP 9-12 (McDaniel et al. 2000). For the different materials studied, it was found that mixtures with less than 15 percent did not require a change in PG binder grade from virgin mixtures. They recommended that mixtures with 15 to 25 percent RAP should have one PG grade softer binders, and that mixtures with greater than 25 percent RAP require the use of a blending chart to determine an appropriate virgin binder PG grade (McDaniel and Anderson 2001). Blending charts are developed to determine the required virgin binder grade as a function of the asphalt binder replacement ratio based on the high and low temperature PG temperatures of the recycled binder using Equation (12).

$$T_{virgin} = \frac{T_{blend} - RBR \cdot T_{RAP/RAS}}{1 - RBR} \quad (12)$$

where: T_{virgin} = critical temperature of the virgin asphalt binder; T_{blend} = critical temperature of the blended asphalt binder (i.e., final desired); $T_{RAP/RAS}$ = critical temperature for the recovered recycled binder; and *RBR* = recycled asphalt binder replacement ratio;

The standard procedure indirectly incentivizes the use of low RAP contents because the contractor does not need to use a different binder source than those used in standard practice. At moderate RAP contents, there is need for a second, softer PG binder, but if sufficient recycled material was placed this may pay for itself. However, these guidelines made high RAP contents, above 25 percent, unpopular, as the blending chart approach requires that the RAP binder is extracted and recovered, and then tested to determine its grade. Extraction and recovery of binder is time consuming and requires the use of hazardous solvents, making it a difficult operation to do in a cost-effective manner (Copeland 2011).

When incorporating RAP stockpile with varying binder contents, as well as RAS which has a very high binder content, the RAP or RAS content may not give a good indication of how much recycled binder is in the mixture (i.e., a surface mixture with 30 percent fine RAP with 6 percent binder will have more recycled binder than a base course with 40 percent coarse RAP with 3 percent binder). In this case the RBR can be used to compare how much of the asphalt binder in the mixture is coming from recycled sources. If both the hypothetical mixtures above had 5 percent total asphalt content, the surface mixture would have an RBR of 36 percent, while the base course would have an RBR of 24 percent.

Many states do not follow the guidance from NCHRP 09-12 and AASHTO M 323 directly. For example, MDSHA requires no virgin binder grade change for RAP or RAP/RAS mixtures with RBRs of 30 percent or lower and RAS with RBRs of 20 percent or lower. They require that blending charts be used to select the appropriate virgin binder grade when the RBR exceeds these thresholds (MDSHA 2014). New York DOT limits RAP contents to 20 percent by weight of mixture and makes no mention of a virgin binder grade selection procedure (New York 2019). Texas DOT specifies allowable RBR values based on the specified and substitute virgin binder grades, the type of recycled materials, mixture type, and the pavement layer (Texas 2016). For HMA with a maximum RBR of 20 percent, a substitute binder with a high temperature PG that is one high temperature PG grade softer than specified and no adjustment to the low temperature grade is required (i.e., substituting PG 58-28 for PG 64-28). However, if the substitute binder is one PG grade softer in both high and low temperatures (i.e., substituting 58-28 for 64-22), the specification allows up to 30 percent RBR in the surface, 35 percent RBR in the intermediate layer, and 40 percent RBR in the base. NCDOT provides clean, concise tables in the Asphalt QMS Manual to specify virgin binder grades for RAP and RAP mixtures, without requiring blending charts. NCDOT allows up to 45 percent RBR in intermediate and base mixes, and up to 40 percent RBR in most surface mixes, with lower limits for RAS-only mixes and mixes with polymer-modified binders. A substitute binder grade is only used when the RBR is greater than 30 percent, or if RAS is used, allowing for higher recycled mixes to be used more easily than other agencies.

Simulating Plant Handling and Mixing of Recycled Materials in the Laboratory

A uniform laboratory procedure for handling recycled materials when preparing asphalt mixture samples is precluded by the wide variety of asphalt plant configurations in use today. Thus, local RAP handling procedures in the laboratory vary considerably. NCHRP 09-12 guidelines suggest heating RAP to 110°C for no more than 2 hours, as higher temperatures and longer times can change the properties of some RAP materials (McDaniel et al. 2001). However, this method does not consider what occurs in an asphalt plant, and purely focuses on limiting aging of the RAP binder. Most asphalt plants do not preheat RAP. Instead, ambient temperature RAP is added to superheated aggregates.

Kvasnak (2010) evaluated four different laboratory RAP preheating scenarios. The first three scenarios included conditioning of the RAP at the mixing temperature for 30 minutes, 3 hours, and 16 hours at the mixing temperature after which the RAP was mixed with virgin aggregate conditioned to the mixing temperature. The fourth scenario included mixing room-temperature RAP with superheated virgin aggregate to mimic typical plant operations. Dry mixing was conducted without the addition of virgin binder. After mixing, the RAP binder was recovered, with the exception of the 16-hour preheated RAP scenario where the binder could not be recovered, presumably due to the changes in binder properties from such an aggressive preheating time. Significant recovered binder property changes were observed in the superheated virgin aggregate scenario so the authors proposed that RAP should be preheated to the mixing temperature for 30 minutes to 3 hours. Zhou et al. (2011) used Kvasnak's guidance to propose a two-step procedure for pre-heating RAP materials prior to introducing to virgin materials. Their procedure includes drying the RAP at 60°C overnight (12 to 15 hours) and then preheating the RAP for two hours at mixing temperature. Lab-produced samples following this procedure were compared with quality control samples from contractors, and the results were satisfactorily consistent, although the specific measure of "consistent" was not reported. However, this procedure is still based on the premise that the laboratory mixing procedures should not alter the RAP binder properties significantly, which has not been validated by field observations.

Preheating temperatures for RAP from state agencies also vary. Some agencies lack any specific guidance for recycled material handling while the specifications that exist vary. MDSHA (2014) specifies conditioning of RAP at 60°C for a maximum of 4 hours, and then combining it with superheated virgin aggregate to achieve the desired mixing temperature when the two are mixed. TxDOT (2016) specifies conditioning RAP at the mixing temperature for a minimum amount of time. In contrast, NYDOT (2019) attempts to limit RAP heating by specifying that RAP is dried immediately before use, batched hot, and heated at the mixing temperature for no more than one hour. RAS preheating practices also vary. The former AASHTO PP 53-09 advised adding the RAS at ambient temperature to the virgin aggregates heated slightly above the mixing temperature. TxDOT (2016) specifies heating of RAS in the same manner as RAP. Practices for mixing and compaction temperature can also vary. AASHTO M 323-17 specifies selection of mixing and compaction temperatures based on the virgin binder viscosity whereas TxDOT (2016) specifies selection based on the intended blended binder grade.

Past research with recycled materials has incorporated an even wider range of handling procedures and preheating temperatures. Rinaldini et al. (2014) followed the standard procedure from the Swiss Federal Laboratories for Materials Science and Technology (EMPA) to prepare samples for their blending analyses. They preheated aggregate at 185°C for 24 hours, RAP in a 1 cm layer in the pan at 130°C for 3 hours, and virgin binder at 130°C for 1 hour. The aggregate and RAP were added to the mixing bucket and mixed for two minutes before adding the virgin binder and mixing for another two minutes. Cavalli et al. (2016) explored the impacts of the mixing and short-term aging temperature, modifying the Swiss standard 640431–8a-NA, to preheat the RAP at 135°C for 1 hour, the virgin aggregates at 180°C for 3 hours, and the binder at 130°C for 1 hour. Navaro et al. (2012) compared RAP mixtures with three different intended production temperatures: 110°C, 130°C, and 160°C. To achieve those production temperatures when mixed with RAP at a consistent temperature, the virgin aggregates were preheated at 105°C, 200°C and 296°C. Their results focused on demonstrating that the size of unblended clusters of RAP is a combined effect of production temperature and mixing time. Their work indicates that temperature has a more significant effect than mixing time. Specifically, for a 30°C reduction in production temperature the mixing time would have to be 2 to 3 times longer to produce the same level of blending.

Recycled Material Handling in Asphalt Plants

There are many ways that recycled materials are incorporated into plant operations (Kandhal and Mallick 1997). Recycled materials cannot be treated as aggregate because the heat from the burner flame will result in smoking of the residual binder, which can damage equipment and stop operations. To combat smoking, ambient temperature RAP is typically added to superheated virgin aggregate. To add the RAP into the plant, a wide variety of different plant modifications and configurations have been developed, each with their own strengths and weaknesses. Different plant configurations will result in different lengths of contact and mixing between RAP, virgin binder, and aggregates (Kandhal and Mallick 1997).

Asphalt plants can be divided into two broad categories: batch plants and drum plants. Batch plants add measured amounts of components to a pugmill and then mix and discharge before repeating the cycle in batches. Batch plants contain a separate aggregate dryer for the virgin aggregates. Drum plants operate a continuous feed of material into and out of the mixer, dispensing wet aggregate into the mixer and drying it before adding asphalt. Drum plants are prevalent in most parts of the US, and in North Carolina specifically, with drum plants comprising more than 80 percent of the 161 asphalt plants approved by NCDOT (Whittington 2018). Drum plants are generally preferred when using recycled materials, and can handle mixtures with higher recycled material contents than batch plants (NCDOT 2020, Kandhal and Mallick 1997). The Massachusetts Department of Transportation (2015) goes as far as to specify a limit of 20 percent recycled materials (including RAP, MWRAS, and processed glass aggregate) by total weight of mixture in a batch plant, but they allow up to 40 percent in a drum plant. A survey from 2009 indicates that at least four other states also have lower limits for RAP usage in batch plants than drum plants (Copeland 2011).

In a batch plant, RAP can be introduced in at least five different ways, as outlined by Kandhal and Mallick (1997). RAP can be mixed with virgin aggregates in the hot elevator, in a mixed hot bin, a separate hot bin, in the hopper, or in the pugmill. Each of these methods exposes the RAP to the virgin aggregate and heat for a different amount of time. The method in which the RAP is added directly to the pugmill from its own hopper would result in very little time of contact with the virgin aggregates, while mixing the RAP with virgin aggregate in a hot bin results in a much longer time of contact. It is also suggested that silo storage may be helpful to increase the time the recycled binders are conditioned at elevated temperature to promote blending.

Drum plants include many different configurations, including parallel flow and counter flow options, which describes how the material travels with respect to the burner flame. Some configurations have isolated mixing areas to help keep the RAP and virgin asphalt further from the burner flame. The aggregate dryer can be separate from the mixer, or it can be an all-in-one apparatus. Some drums even have extra barrels; double barrel drums are relatively common and triple barrel drums exist as well (Kandhal and Mallick 1997). Each of these plant configurations introduce differences in the heating times and temperatures of the RAP, as well as mixing times, and temperatures of the virgin aggregates.

Factors that Can Affect Blending between Virgin and Recycled Materials

Numerous factors have been reported to influence the blending between recycled and virgin materials during asphalt concrete production. Procedures for producing asphalt concrete with RAP can differ greatly depending on the application (e.g., in-place versus in-plant) and equipment utilized. As discussed, in-plant recycling procedures differ based on plant type (batch vs. drum) and plant configuration (e.g., double-drum vs. single drum). Blending initiates during mixing, therefore, mixing time and temperature have a great impact on the degree of blending (Howard et al. 2009). In addition, silo storage time can impact blending. Following storage, mixtures are dispatched into haul trucks. Longer hauls increase blending by prolonging exposure to elevated temperature (Howard et al. 2009).

The inherent properties of the RAP and fresh binders also influence blending. Low viscosity binders are expected to blend more rapidly than high viscosity asphalts (McDaniel et al. 2001). Aggregate absorption can influence blending as absorbed RAP binder is less likely to mix with fresh asphalt. Filler particles selectively adsorb asphalt components and disrupt the diffusion path of binders and thus will alter the resultant degree of blending (Yousefi Rad et al. 2014). Fine particles heat more rapidly than large aggregates. Thus, a higher fraction of fines in a mix is expected to improve blending during mixing (Howard et al. 2009). The film thickness of asphalt binder will also play a significant role in determining the degree of blending with thicker films requiring longer time at elevated temperature for complete blending (Kriz et al. 2014).
The incorporation of rejuvenators, also termed recycling agents, may also impact blending levels. Epps et al. (1980) borrowed a definition of a recycling agent from the Pacific Coast User-Producer Group, stating that a recycling agent is a “hydrocarbon product with physical characteristics selected to restore aged asphalt to requirements of current asphalt specifications”. Rejuvenators are intended to improve the cracking resistance and, in some cases, workability of RAP and RAS mixtures without adversely affecting rutting resistance (Epps Martin et al. 2015). Recycling agents should be easily dispersed, alter the viscosity to the desired level, be compatible with the asphalt binders, redisperse asphaltenes, improve the life expectancy of the RAP mix, have uniform properties between batches, and be resistant to smoking and flushing (Epps et al. 1980). While this list of characteristics is not recent, it is exhaustive and still applicable today to ensure that items purveying themselves as “rejuvenators” do in fact achieve their intended purpose and provides a basis for evaluating which products do better. Table 18 lists common classes of rejuvenators used in practice (NCAT 2014).

Table 18. Types of Rejuvenators (NCAT 2014)

Category	Description
Paraffinic Oils	Refined used lubricating oils
Aromatic Extracts	Refined crude oil products with polar aromatic components

Napthenic Oils	Engineered hydrocarbons
Triglycerides and Fatty Acids	Derived from vegetable oils
Tall Oils	Paper industry by-products, same chemical family as liquid antistrip agents and emulsifiers

Currently, specifications pertaining to recycling agents are sparse and variable (Daly 2017). Epps Martin et al. (2015) distributed a survey to state highway agencies to identify the laboratory tests used in practice to characterize asphalt binders modified by recycling agents. The results of their survey are presented in Figure 27, which demonstrates a wide variation in practices, with 40 percent of agencies that use recycling agents performing no characterization of the recycling agent itself or of the recycling agent blended with asphalt. As of 2015, 83 percent of state highway agencies did not allow the use of recycling agents in surface mixtures (Epps Martin et al. 2015). It is inferred that the limited use of recycling agents is largely a result the lack of robust specifications.

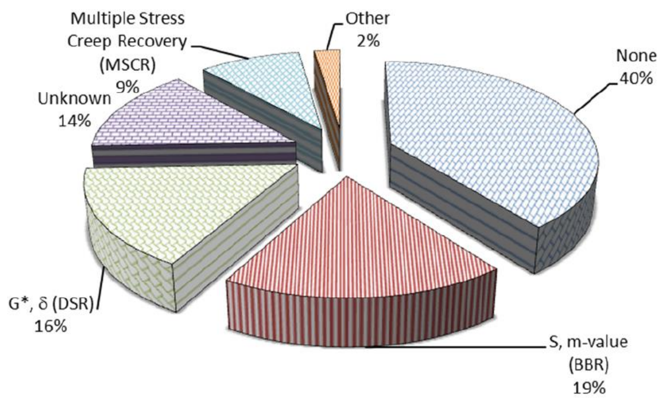

Figure 27. National survey results of laboratory tests used in practice to characterize the properties of asphalt binders modified by recycling agents (Epps Martin et al. 2015).

Experimental and Analytical Methods used to Infer Blending

Inferences from Asphalt Mixture Mechanical Properties

Several studies have used measurements of asphalt mixture mechanical properties to infer differences in the degree of blending among asphalt mixtures. Jacques et al. (2016) evaluated the effects of silo storage time on the dynamic modulus and fatigue cracking performance of RAP and virgin mixtures. Performance testing results indicated that both virgin and RAP mixtures aged with an increase in silo storage time. However, the RAP mixtures experienced greater changes in dynamic modulus and cyclic fatigue performance with silo storage time than the virgin mixtures, which could not be explained by oxidation levels. Therefore, the authors attributed the changes in performance in the RAP mixtures with silo storage time largely to continued blending of the virgin and RAP binders while in the silo. Wen and Zhang (2016) produced RAP mixtures with the same composition but different laboratory mixing, conditioning, and compaction procedures. They found that the asphalt mixture dynamic modulus and cracking resistance varied among the laboratory fabrication procedures, which they attributed to differences in blending levels.

RILEM TC 264 TG 5 proposed an alternative procedure to quantify recycled binder availability that utilizes 100 percent RAP mixtures (without the addition of virgin binder) (Menegusso Pires et al. 2021). RAP is conditioned for four hours at various temperatures spanning from 70°C to

190°C, compacted, and subjected to indirect tensile strength (ITS) testing. RAP specimens with higher ITS are assumed to have higher recycled binder availability. Correspondingly, the ratio between the measured ITS at the temperature of interest and a maximum ITS assumed to coincide with 100 percent availability is reported as the degree of activity (DoA). The RILEM procedure was recently evaluated using a wide range of RAP materials from the U.S. (Abdelaziz et al. 2021, Sobieski et al. 2021). Both studies suggested the method could be used to identify the production temperature to yield maximum availability in a given RAP source. However, the studies recognized there is considerable uncertainty in defining the maximum ITS for a given RAP source given that complete availability is unlikely at any production temperature. Also, differences in ITS of a given RAP as a function of conditioning temperature can arise from sources other than availability (e.g., oxidative age level differences), potentially compromising the use of ITS ratios as a measure of availability.

As an alternative to comparing the mechanical properties among asphalt mixtures to infer whether complete blending occurs, Bonaquist (2007) proposed that blending between recycled and virgin binders can be assessed by comparing the measured dynamic modulus of a mixture to that predicted from the Hirsch model using asphalt binder input properties corresponding to the extracted and recovered binder from the mixture. Bonaquist (2007) proposed that the latter reflects complete blending since solvent extraction and recovery yields all of the binder within a mixture, both recycled and virgin. The Hirsch model (Christensen et al. 2003) allows for estimating the dynamic modulus of an asphalt mixture based on the dynamic shear modulus of the asphalt binder contained within the mixture and the asphalt mixture volumetric properties. If the measured and Hirsch model predicted asphalt mixture dynamic moduli agree, Bonaquist (2007) suggested that it could be concluded that complete blending exists within the mixture whereas differences suggest incomplete blending. Booshehrian et al. (2013) applied the method proposed by Bonaquist (2007) to plant-produced asphalt mixtures and found that the extent of blending is dependent on the plant discharge temperature. The uncertainty in Hirsch model predictions can be high even in virgin mixtures (Sakhaeifar et al. 2015). Thus, discrepancies between measured asphalt mixture dynamic moduli and those predicted using the Hirsch model can arise from other sources than incomplete blending, bringing into question the reliability of the approach for assessing blending within asphalt mixtures. Furthermore, the methodology does not allow for quantifying the expected percentage of the recycled binder that is activated and blends with the virgin binder in cases where the results indicate incomplete blending.

Binder Diffusion Measurements

Past studies have proposed that quantifying the rate of diffusion between recycled and virgin binders can yield important insight about blending in asphalt mixtures (e.g., Oliver 1974, Sreeram et al. 2019, Ding et al. 2016, Karlsson and Isacsson 2003, Karlsson et al. 2007, Kriz et al. 2014, Yousefi Rad et al. 2014, He et al. 2016). Diffusion is defined as the net movement of molecules driven by a difference or gradient in concentration. Several recent studies have investigated the role of mutual solubility (Sreeram et al. 2019) and interactions among molecules (e.g., Ding et al. 2016)) in an effort to understand possible inter-diffusion mechanisms in asphalt binder systems. However, the exact mechanisms of diffusion are currently unknown. Consequently, the majority of past studies seeking to quantify diffusion have relied on bulk rheological measurements of binders placed in contact (Karlsson et al. 2007, Kriz et al. 2014, Yousefi Rad et al. 2014, He et al. 2016). In these experiments, RAP and the virgin binder wafers are placed in contact and conditioned. The wafer systems are subjected to oscillatory loading to monitor the diffusion process. Trends in the dynamic shear modulus or complex viscosity with conditioning time are translated to a concentration gradient. The evolution of the concentration gradient with time is then used to calculate the diffusion coefficient using Fick's second law (Karlsson and Isacsson 2003, Karlsson et al. 2007, Kriz et al. 2014, Yousefi Rad et al. 2014, He et al. 2016).

While relatively simple in principle, past efforts have noted significant challenges when implementing the procedure. Oxidation of the binders during the conditioning can confound test results (Kriz et al. 2014, Yousefi Rad et al. 2014, He et al. 2016). In addition, flow due to gravity and/or the application of strain to monitor the sample response as a function of conditioning time can induce mechanical mixing and therefore, impede the accurate quantification of diffusion when conditioning samples within the DSR at elevated temperatures that reflect asphalt mixture production (Kriz et al. 2014). To circumvent mechanical mixing induced by the application of strain, several researchers have conditioned samples in an external chamber at high temperature and then tested the composite sample in the DSR at a lower temperature where further blending would not be expected and lower strain amplitudes could be used in testing without approaching minimum torque limits of the DSR (Yousefi Rad et al. 2014, He et al. 2016).

Mastic and Mortar Experiments

Several studies have attempted to use experiments that combine fine aggregate and asphalt binder to aid in the understanding of blending within asphalt mixtures. Gundla and Underwood (2017) compared the rheology of mastics (i.e., combination of mineral filler and asphalt binder) prepared with and without RAP to predictions using the Herve and Zaoui (1993) micromechanical model to infer blending levels. The Herve and Zaoui (1993) micromechanical model allowed for simulating filler, virgin binder, RAP binder, and blended binder phases within the mastic. Based on adjustments to the proportions of blended and unblended binder to best match DSR test results, the authors concluded that the amount of blending that occurs within mastics, as a proportion of the total recycled binder, decreases as the RAP content increases. The approach employed to infer blending levels assumes that the Herve and Zaoui (1993) model accurately reflects asphalt mastic microstructure; the authors, however, note that this assumption may be invalid due to the effects of aggregate shape and contact within the mastic which are not accounted for within the model but impact the measured rheology.

Other researchers have conducted DSR and Bending Beam Rheometer (BBR) tests on mortar (i.e., combination of asphalt binder and aggregate passing the No. 50 sieve and retained on the No. 100 sieve) samples to determine the effective grade of the binder contained within the mortar (e.g., Swiertz et al. (2011), Hajj et al. (2012)). The procedure is based on the hypothesis that any differences in the rheological properties of two mortar samples created with the same gradation and total asphalt content can be attributed to the differences in the constituent binder properties. This hypothesis implies that the ratio between the rheological properties of two mortars is equivalent to the ratio between the effective binder properties contained within the two mortars. Thus, the ratio between the results of mortars prepared with and without recycled binder can be multiplied by the virgin binder properties to determine the effective recycled binder properties within the mortar. Swiertz et al. (2011) verified this hypothesis using laboratory prepared mortars where complete blending was expected. Hajj et al. (2012) applied the methodology by creating mortars using virgin binder combined with recovered RAP aggregate and virgin aggregate. Comparative mortars were preparing using RAP, virgin aggregate, and virgin binder, maintaining the same total binder content as the virgin mortar. The results of the mortar experiments were compared to corresponding results of extracted and recovered binders. The authors found that the mortar procedure resulted in lower performance grades for the materials containing recycled binder, which they indicated suggests that the recycled binder did not fully mobilize and blend with the virgin binder. A drawback of the mortar approach to infer effective binder properties within asphalt mixtures is the unknown impact of the unblended binder on the effective aggregate gradation. In addition, the mesoscale studies focus on a very narrow aggregate size range may fail to reflect blending within asphalt mixtures containing a broad range of aggregate sizes.

Dry Mixing

To visualize the activation of recycled binders, researchers have employed dry blending of the recycled materials with virgin aggregate to evaluate recycled material activation. In development of an adjusted binder contribution factor for recycled materials, Georgia DOT attempted to use dry mixing in a pugmill to evaluate their RAP stockpiles. However, all that was observed was minor scuffing of recycled binder on to the virgin aggregates, with no measurable binder transfer (NCAT 2013). They then moved on to other methods to assess recycled binder activation. Farris (2016) attempted to use dry blending to observe RAS activation. He observed that the virgin fine aggregate adhered to the RAS particles with no RAS binder coating the coarse aggregates directly following dry blending. Aging for two hours at 275°F led to some binder transfer, but this was significantly lower than the amount of blending observed with other methods.

Huang et al. (2005) attempted to assess RAP blending using a very aggressive dry mixing condition where materials were heated to 190°C and mixed for three minutes. The observed binder transfer was roughly 10 percent, indicating the RAP behaved primarily as a black rock. Shirodkar et al. (2011) built on the work of Huang et al. (2005) to propose a procedure to measure partial blending of recycled binder. As part of that procedure development, they conducted a similar dry mixing experiment, however they preheated materials to 176°C and mixed for ten minutes. This additional mixing resulted in transfer rates of 24 and 15 percent for mixtures containing 25 and 35 percent RAP, respectively. However, the authors felt that this was still lower than expected, and rounded both numbers up, to 30 and 20 percent, respectively. Wang et al. (2017) proposed a methodology to assess rejuvenators in RAP mixtures using a mortar transfer ratio and image analysis. However, the baseline mortar transfer ratio, with no rejuvenators present, was measured at less than 3 percent. The limited transfer observed in all experiments indicates one of two possibilities: either recycled asphalt is behaving as a black rock, or the virgin binder plays a critical role in activating recycled binder.

Size Exclusion

Once virgin binder is added to the mixture, it can be difficult to impossible to separate virgin and recycled aggregates. To address this, many researchers have employed size-exclusion methodologies. Most commonly this is achieved by mixing coarse virgin aggregates above a threshold sieve size with fine RAP particles below the threshold sieve size. Each research effort uses a slightly different procedure with a slightly different goal

Huang et al. (2005) used the No. 4 (4.75 mm) sieve to separate RAP particles from virgin aggregates after mixing, to allow for staged extraction and recovery on the RAP particles alone. Based on the work by Huang et al. (2005), Shirodkar et al. (2011) used the No. 4 sieve as the threshold for virgin aggregate, while using RAP particles below a No. 8 (2.36 mm) sieve. The gap was intended to minimize blurred lines between particle sizes during separation.

D'Angelo et al. (2011) attempted to simplify the concepts proposed by Huang et al. (2005) to investigate RAP binder blending. They used size-exclusion methods to separate RAP and virgin aggregate particles then evaluate each group separately. Complete extraction and recovery allowed for both binder content determination and binder property testing. The binder content results indicated that the RAP aggregates generally had an increased binder content, while the virgin aggregates had only the minimum needed binder, indicating that the RAP binder may not be contributing significantly to the mix. This was backed up by the binder property testing which showed very similar properties between the original virgin binder and the recovered binder from virgin aggregates.

NCHRP 09-58 (Kaseer et al. 2019, Epps-Martin et al. 2020[b]) built on the work of D'Angelo et al. (2011) to develop a more formal procedure to estimate RAP binder availability, using the binder content measurement technique. Their methodology used four size fractions of virgin aggregate, with all material passing through a 1/2" sieve and then collecting the materials retained on the 3/8", No. 4, No. 8 and No. 30 sieves. A virgin mixture was fabricated with purely virgin aggregates, and a RAP mixture was fabricated where the "intermediate" material on the No. 4 sieve was replaced with RAP of the same size. Each mixture was then separated to the original size fractions, and binder content was measured on each size fraction in the ignition oven. To evaluate RAP binder availability, the binder content on the intermediate size fraction in each mixture was compared. If the binder contents were the same, then the conclusion is that the RAP binder is completely incorporated. A theoretical maximum value, in which the RAP does not contribute any binder and takes its proportion of the virgin binder was calculated. The researchers then suggested a linear relationship between these two points to allow evaluation of the RAP binder availability factor (BAF) based on the binder content of the RAP particles.

The method presented by Kaseer et al. (2019) is a reasonably simple method to attempt to estimate the RAP BAF. However, it relies on only a single size of "intermediate" RAP aggregates, thus it does not address the question of clusters, which may not be adequately represented in the single intermediate size evaluated. It also does not consider the impacts of the dust and dust-to-binder ratio on the overall mixture behavior and cannot provide assessment of the fines due to the limitations of the ignition oven. Thus, it is not an ideal research tool for fundamental assessment of recycled materials, but it may present a practical method for agencies and contractors to assess materials and guide use of more comprehensive adjustments to mixture design methodologies.

Clear and Pigmented Binders

Evaluating the blending of binders is a challenge in recycled mixtures as the two binders are indistinguishable under most conditions. Typical paving asphalt binders are black and opaque, so the virgin and recycled are not visually distinguishable. Some researchers have distinguished binders visually by using a clear virgin binder to allow optical visualization of blending. Nguyen (2009) used Mexphalte C/SHELL, a clear asphalt-like binder which is typically used in tunnels to improve visibility and can be colored with pigments for decorative pavements and safety markings (Shell 2010). The goal was to investigate laboratory mixing practices and propose an improved procedure. To further improve visual delineation of the virgin and recycled binders, 10 percent iron oxide was added to the clear binder, resulting in a red virgin binder. Samples were sliced and photographed, showing clear areas of black RAP binder unincorporated into the red matrix, which were termed "lumps" by the researcher. These lumps were clearly indicative of incomplete blending, however the visual inspection methodology used could not quantify the actual blending, thus performance testing was used to make quantitative assessments.

Navaro et al. (2012) also used Mexphalte C/SHELL, which has polymers that fluoresce under ultraviolet (UV) light. These fluorescing polymers were used to differentiate the virgin binder from the RAP binder within a mix. They proposed an observational technique using image analysis under white and UV light, to quantify blending levels. Their objective in evaluating blending was to determine the impact of production temperatures on blending kinetics and RAP clustering.

Farris (2016) used a clear binder-like product produced by Sealoflex to evaluate RAS activation in the laboratory. He found that the clear asphalt indicated good blending based on the uniform mixture color observed after mixing. He also noted that while the clear asphalt showed good blending, additional research was needed to evaluate whether the clear asphalt behaves like conventional paving asphalt.

Wu et al. (2018) eschewed clear binder, instead selecting to use 30 percent iron oxide in a typical paving asphalt. This amount of pigment decreased the ductility sixfold, but other empirical binder tests did not show as significant of changes. The results from color image analyses were linked to indirect tensile stiffness modulus tests, to assess the impacts of mixing times and temperatures. The researchers concluded that the concept had merit however the tinting technique used had major flaws as the color was difficult to distinguish and had a significant impact on the measured binder properties.

Staged Extraction and Recovery

While standard extraction and recovery results in a thorough mixing of virgin and recycled binders, staged extraction procedures have been used since 1979 to attempt to measure asphalt blending. Zearly (1979) used only two stages in his work and found that mixing appeared to be extensive between the RAP and virgin binders, based on the measured penetration of the recovered binders. Carpenter and Wolosick (1980) used the same two-stage extractions to evaluate rejuvenator interactions directly with RAP, and Noureldin and Wood (1987) built on that work to develop a four-stage extraction procedure. Noureldin and Wood (1987) used their procedure to evaluate RAP alone, RAP with modifiers, and a more realistic mixture with RAP, modifiers, and virgin aggregate. It was found that the RAP itself had different layers of aging within the binder film, specifically having a harder outer shell. While the rejuvenator dispersed well through this shell when mixed with RAP alone, the trends were much less consistent when virgin aggregate was added. The researchers attributed the inconsistency to the tendency of the virgin aggregate to absorb the rejuvenated binder leaving the original RAP binder on the individual aggregates.

Huang et al. (2005) used staged extraction and recovery on the RAP particles to assess how well the virgin binder “cut” into the RAP binder layers, using a four-layer procedure. The RAP particles were soaked in subsequent containers of trichloroethylene for three-minute increments to remove each “layer”. The recovered binder from each “layer” was tested for complex modulus and rotational viscosity. The results of binder testing indicated that the recycled asphalt was staying with the recycled aggregates and not contributing completely.

Bowers et al. (2013) used a staged extraction to allow visualization of the layers surrounding the RAP particles. The staged extraction took place in 30-second and 1-minute increments, with the loose mixture sample submerged in beakers of trichloroethylene. The recovered binders were subjected to gel permeation chromatography (GPC) and Fourier-transform infrared spectroscopy (FTIR) to assess the levels of aging, and thus RAP blending.

There is some concern surrounding the possibility that the staged extraction does not remove even layers but takes various fractions of asphalt in order of their solubility in the extraction solvent. To address this concern, Bowers et al. (2013) also performed staged extraction on a virgin mixture, which showed little change in GPC measurements between each stage when trichloroethylene was used as the solvent. To avoid that concern, Zhao et al. (2016) compared four layers of binder films on virgin aggregates to those on RAP aggregates to assess the overall mixture homogeneity. The mixture was designed to allow for size exclusion separation of virgin and recycled aggregate particles. They found that in RAP mixtures the binder films on virgin aggregates were uniformly blended, while those on the RAP particles were not. The outer layers of RAP were not statistically different from all the layers on the virgin particles, with just the inner layers of binder being markedly different. In RAS mixtures, the binder films were much more heterogeneous, with the outer layers of each matching closely, but the inner layers differing significantly.

An additional problem with staged extraction methods is that they do not allow visualization of the arrangement of aggregate particles, including the possible clustering of RAP particles, which is has been seen in some RAP experiments (Nguyen 2012, Bressi et al. 2015). These clusters of RAP particles may cause non-uniform blending and could become sites for crack initiation and propagation.

Fluorescence Microscopy

Fluorescence microscopy was first applied to recycled asphalt by Navaro et al. (2012), who used a clear virgin binder that would fluoresce under ultraviolet light to delineate RAP and virgin binder. Micrographs of the asphalt concrete sample were taken under both white and UV light. They used image analysis to identify and mask the aggregates using both images. The aggregate mask was used to hide aggregates in a grayscale UV image, with lighter areas indicating virgin binder and darker areas indicating RAP. In addition to the drawbacks of using a clear binder, the image processing relies on differentiating aggregate and asphalt in grayscale, and developing appropriate thresholds is subjective.

Building on the work of Navaro et al. (2012), Ding et al. (2017) further investigated the fluorescence characteristics of asphalt binders. Rather than using asphalt concrete, glass cullet was added to the laboratory mixer and retrieved after mixing. In addition, clear asphalt was not used, as the chemical changes to aged asphalt result in a marked decrease in fluorescence. The proposed procedure requires the development of a blending chart using blends of recycled and virgin binder, then comparing to the results in situ on the glass cullet. To avoid the need for glass cullet, Ding et al. (2018) compared results on glass cullet to typical aggregates and found no significant difference. The modified procedure was then applied to a hot mix both with and without rejuvenator, as well as a foamed warm mix to evaluate the effects of these material factors on blending. These initial results indicated that the foamed warm mix indicated the greatest blending, which seems counterintuitive. The impact of foaming on fluorescence was not quantified, and the addition of water for foaming may impact the fluorescence of the virgin binder.

EDS-SEM

Lee et al. (1983) were the first to attempt to measure blending within recycled mixtures using a scanning electron microscope (SEM) with an energy dispersive X-ray spectroscopy (EDS) detector. While an SEM alone can only generate images of relative composition, EDS allows for the detection of specific elements. EDS can be used to generate a quantitative estimate of the elemental mass composition on an area of a sample surface or approximate maps of an element over the sample surface. Lee et al. (1983) added trace amounts of titanium to the virgin binder to distinguish it from the recycled binder within compacted asphalt mixtures using EDS-SEM. Their intent was only to validate a different technique though, and with the limitations of SEMs at that time, it appears the method was not investigated further.

Al-Qadi et al. (2009) thoroughly explored conventional SEM imaging of backscattered and secondary electrons, finding that the black and white images of surface topography and compositional contrast could not provide useful information about recycled material interactions. The potential of using a titanium dioxide tracer as used in Lee et al. (1983) is mentioned, however it is not acted upon. A specific reason is not cited, but EDS detector limitations are likely, as older silicon-lithium detectors required longer processing times than modern silicon drift detectors, often several orders of magnitude. Having given up assessing recycled material interaction, their focus shifted to attempting to measure binder film thicknesses.

Rinaldini et al. (2014) developed an EDS image of recycled asphalt concrete as part of a multiscale investigation of blending between RAP and virgin materials. Their mixture was prepared with 0.94 percent titanium dioxide by weight total mixture, with an average particle size of 2 microns. The particle size informs the maximum resolution, as larger particles will appear as discrete particles instead of a diffuse distribution at higher image magnification. This is especially apparent in EDS maps, although the particle distribution will likely also impact local elemental spectrum measurements. The microscopy specimens were cut from gyratory samples before being impregnated with epoxy and cured under pressure, then the surface was polished to create a smooth surface for imaging.

A Phillips ESEM was used in low vacuum mode to provide sufficient resolution without the need for sample coating. The backscattered electron images showed some microcracks in the binder between RAP and virgin particles, indicating that the binders might not be well-mixed. The one EDS image obtained was developed overnight, as older detectors require several orders of magnitude more time to map a sample. The EDS image indicated good blending as the titanium dioxide was well-distributed between the virgin and RAP aggregates, contradicting the backscattered electron images. Bressi et al. (2015) applied a similar EDS-SEM method to investigate clustering of RAP particles in a mixture but did not use it to evaluate blending directly.

Castorena et al. (2016) built on previous work, investigating RAP blending in a FEI Verios SEM, with a stronger EDS detector that could develop EDS maps in minutes. Gyratory samples were prepared with 20 percent titanium dioxide by weight of virgin binder. The titanium dioxide particle size was 0.15 microns. The microscopy specimens were cut from the gyratory samples but did not require epoxy impregnation before polishing. Due to the lack of variable pressure control in the microscope used, the microscopy specimens did require a gold-palladium coating to avoid surface charging. Preliminary investigations pointed to some impacts on blending from laboratory preheating, mixing, and conditioning temperatures, however pre-processing appeared to be a very important factor as well.

Past EDS-SEM analyses confirm that partial recycled binder availability and blending exist in asphalt mixtures. Bressi et al. (2015) observed agglomerations of adhered recycled material exist in asphalt mixtures using EDS-SEM. Recycled binder bound within the agglomerations does not come into contact with, and is therefore, unavailable to blend with the virgin binder. Castorena et al. (2016) also observed regions of unavailable RAP and found that RAP pre-processing (i.e., fractionation) can affect the uniformity of recycled binder dispersion. Bressi et al. (2015) hypothesized that the agglomerations of RAP serve as fracture initiation sites. However, this hypothesis was not tested. Cavalli et al. (2017) also demonstrated that films of RAP binder can exist on the periphery of RAP aggregate particles that is not blended with virgin binder. Rinaldini et al. (2014) observed microcracks in the vicinity of the boundary between unavailable RAP binder and virgin binder in asphalt mixtures and thus, speculated that such boundaries may constitute ‘weak spots’ in the mix. Several studies have observed heterogeneous concentrations of recycled binder within virgin binder matrix of asphalt mixtures further suggesting incomplete blending exists between the available recycled binder and virgin binder; the extent of heterogeneity was found to increase with the increase in recycled material content (Jiang et al. 2018, Abdulfattah et al. 2021) and decrease with prolonged aging at elevated temperature or the incorporation of a recycling agent into the mix (Jiang et al. 2018).

Quantitative Analysis of Elemental Spectra

Castorena et al. (2016) attempted to quantify the visual observations made from the EDS maps, using local elemental spectra of specific areas of interest on the map. These local spectra were within the mastic phase of the asphalt but contained varying degrees of small aggregates. To minimize the effect of the aggregate content, the measured titanium concentration was normalized against carbon to develop a Ti:C ratio. Due to a lack of calibration specimens, it was not possible to reliably estimate a degree of blending from these local ratios, except in locations where the values were approximately zero, indicating no blending.

Wavelength dispersive X-ray fluorescence spectroscopy (WDXRF) has been evaluated in Texas to determine the presence and concentration of re-refined engine oil bottoms in supposedly neat asphalts (Barborak et al. 2016, Karki and Zhou 2017). WDXRF bears a striking similarity to EDS; as discussed previously, when an atom is bombarded by an electron, it emits both a secondary electron and an X-ray photon. The same is true when the atom is hit by an X-ray photon. In EDS, the energy of the X-ray photon is used to determine the element of the atom, however in wavelength-dispersive spectroscopy, the detected wavelength of the photon can be used instead (Egerton 2016). Thus, the analysis of the elemental spectra is similar. Barborak et al. (2016) developed a preliminary procedure to detect REOB, which contains some elements not typically present in neat asphalts and estimate whether the REOB is below the 5 percent limit specified by Texas DOT. The detection of REOB on the basis of elements not typically present in neat asphalt was fairly straightforward, however quantifying the REOB was not as straightforward. Even using the same REOB source, different virgin binders would yield high errors when compared to a different calibration binder. Thus, several calibration binders were tested, and binders were grouped based on sulfur and vanadium content to select the most appropriate calibration standard. This worked reasonably well, with relative errors below 20 percent, when using a new batch of REOB from the same initial source, as compared to errors of up to almost 50 percent when a single random calibration standard. When additional REOB sources were tested, the matched calibration standards yielded errors below 50 percent, while the single random standard yielded errors exceeding 90 percent on some samples. Karki and Zhou (2017) built on the previous work to develop a more sophisticated methodology for selecting an appropriate calibration standard, using more REOB sources and asphalt binders. While REOB is not directly important to recycled asphalt materials, this work demonstrates the significance of using calibration standards, as different asphalts can have widely different elemental compositions.

Jiang et al. (2018) used a similar approach to Castorena et al. (2016) normalizing their titanium to sulfur instead of carbon, and developing a simple model to estimate the degree of blending. The model uses the initial Ti:S ratio of the virgin binder and the final Ti:S ratio of the blended mixture as well as the total asphalt content, RAP content, RAP binder content, and sulfur content of both the RAP and virgin binders.

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APPENDIX B: ASPHALT BINDER DIFFUSION MEASUREMENTS

Introduction

Blending of virgin and RAP materials in asphalt mixtures is a complex process. Blending occurs by two primary time-dependent processes: mechanical mixing and diffusion (Zhang et al. 2019). Initially, mechanical mixing occurs when RAP aggregates, virgin aggregates, and virgin binder are combined and agitated. Once mechanical mixing initiates contact between virgin binder and RAP binder, diffusion also ensues. Diffusion is defined as the net movement of molecules driven by a difference or gradient in concentration (Crank 1979). Consequently, numerous studies have postulated that understanding the temperature and time-dependence of the rate of diffusion of virgin binder into the RAP binder is critical to the understanding of blending in RAP mixtures (e.g., Oliver 1974, Karlsson and Isacsson 2003, Karlsson et al. 2007, Kriz et al. 2014, Yousefi Rad et al. 2014, He et al. 2016)

It is generally understood that inter-diffusion occurs as a result of interactions among polar species and associated energy imbalance. While several recent fundamental studies of the chemical compatibility of asphalt binder blends and interactions among molecules have shed light on possible inter-diffusion mechanisms in asphalt binder systems (e.g., Ding et al. 2016, Sreeram et al. 2019), the exact mechanisms of diffusion remain unclear. The complexity of the inter-diffusion process has led many researchers to employ methods that rely on bulk rheological rather than chemical measurements of asphalt blends to infer diffusion.

Several protocols for measuring the diffusion coefficient between RAP and virgin binders using the Dynamic Shear Rheometer (DSR) have been proposed (Karlsson et al. 2007, Kriz et al. 2014, Yousefi Rad et al. 2014, He et al. 2016). In these experiments, RAP and the virgin binder wafers are placed in contact and conditioned, which is shown schematically in Figure 28. The wafer systems are subjected to oscillatory loading to monitor the diffusion process. Initially, most of the deformation takes places in the softer, virgin binder layer, leading to a relatively low dynamic shear modulus ($|G^*|$) response of the layered composite system. As the test progresses and the layers blend, the $|G^*|$ increases. Once the layers reach complete blending, the $|G^*|$ attains a constant value. Trends in the $|G^*|$ or complex viscosity (η^*) (equal to the $|G^*|$ divided by angular frequency) with conditioning time are translated to a concentration gradient. The concentration gradient evolution with time can be used to calculate the diffusion coefficient using Fick's second law (Karlsson et al. 2007, Kriz et al. 2014, Yousefi Rad et al. 2014, He et al. 2016).

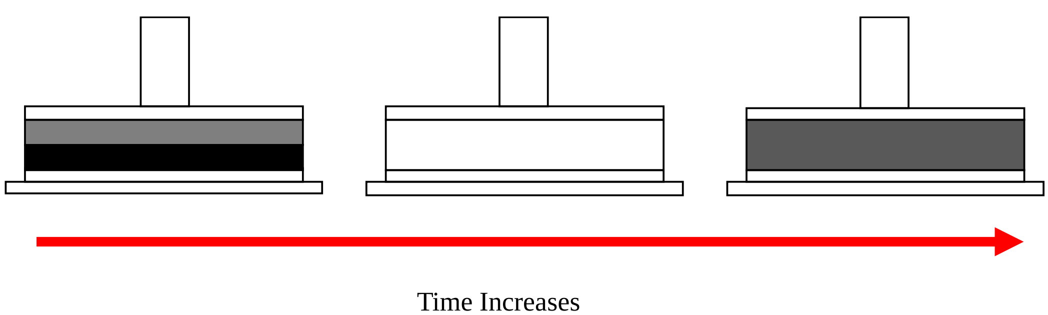

Figure 28. Schematic of the DSR-based diffusion experiment (black indicates RAP, grey indicates virgin binder).

While relatively simple in principle, past studies have noted experimental challenges when implementing DSR procedures to quantify diffusion between asphalt binders. Diffusion can occur during thermal equilibration of the composite system (Kriz et al. 2014). To minimize thermal equilibration time, several studies have used very thin samples on the order of 50 μm to 250 μm (Karlsson et al. 2007, Kriz et al. 2014). Flow of the top wafer into the second wafer due to the influence of gravity can also induce undesired blending (Kriz et al. 2014). Past studies have tried to mitigate the influence of gravity by using thin wafers and placing the denser material, assumed to be the recycled binder, below the virgin binder (Karlsson et al. 2007, Kriz et al. 2014). While the use of thin films can help mitigate test errors, past researchers have found that accurately controlling the thickness of very thin samples is challenging (Karlsson et al. 2007, Kriz et al. 2014); this led Kriz et al. 2014 to allow the film thickness to vary as part of the least squares optimization of test data used to calculate the diffusion coefficient.

Oxidation of the binders during conditioning may further confound test results. Oxidation leads to an increase in the modulus of binder samples, similar to blending (Kriz et al. 2014, Yousefi Rad et al. 2014, He et al. 2016). Past efforts have measured the rate of change in the properties of fully blended samples subjected to the same conditioning procedure as the composite wafer samples in an effort to isolate the effect of oxidation from diffusion (Kriz et al. 2014, Yousefi Rad et al. 2014, He et al. 2016).

Past studies demonstrate that diffusion coefficients quantified using DSR experiments depend on the strain amplitude employed during conditioning of the wafers, suggesting that mechanical mixing can also confound test results (Karlsson et al. 2007, Kriz et al. 2014). Consequently, past studies have used low strain amplitudes in an effort to minimize the influence of mechanical mixing (Kriz et al. 2014). However, torque limitations of rheometers may limit the ability to apply sufficiently low strain amplitudes to avoid disturbing the sample given the relatively low viscosity of asphalt binder at hot-mix asphalt mixture production temperatures where diffusion is of most interest.

Edge failure has been visually observed when wafers are conditioning in the DSR at high temperatures that reflect hot-mix asphalt mixture production (Karlsson et al. 2007). Edge failure manifests as an indentation of the sample at its periphery as a result of flow instability when a critical shear rate is exceeded (Hemingway and Fielding 2019). Edge failure reduces the effective sample diameter, leading to uncertainty in the interpretation of the test results. To circumvent the effects of edge failure, Karlsson et al. 2007 normalized the time-dependent response by the initial response, which was assumed to have been recorded before the onset of edge failure; however, this approach assumes that the extent of edge failure does not change with time which may not be accurate. Inertia artifacts, surface tension generating torque, and wall slip can also compromise parallel plate DSR measurements of very soft materials (Ewoldt et al. 2015).

To mitigate the challenges associated with conducting DSR tests at hot-mix asphalt production temperatures, several researchers have conditioned samples in an external chamber at high temperature and then tested the composite sample in the DSR at a lower temperature (Yousefi Rad et al. 2014, He et al. 2016); this approach requires the preparation of separate samples for each conditioning time of interest and is therefore more time consuming than conditioning samples directly in the DSR. Also, mechanical mixing may be induced when removing samples from the external chamber to cool.

Based on the aforementioned challenges associated with measuring the diffusion coefficient between RAP and virgin binders, the viability of measuring the diffusion coefficient in the DSR merits further investigation. The objective of this study is to critically evaluate the ability to quantify the diffusion coefficient of virgin binder into RAP binder using the DSR.

Materials and Methods

Theoretical Background of the DSR-based Diffusion Experiments

Fick's second law, given in Equation (13), was the basis for the interpretation of the diffusion experiment results (Crank 1979).

$$\frac{\partial \phi_{RAP}}{\partial t} = D \frac{\partial^2 \phi_{RAP}}{\partial y^2} \tag{13}$$

where: D = diffusion coefficient; ϕ_{RAP} = volumetric fraction of RAP binder; y = vertical distance from the bottom plate; t = conditioning time.

The boundary conditions for the diffusion experiment in the DSR are given by Equations (14), (15) and (16) (Kriz et al. 2014).

$$\partial \phi_{RAP} / \partial t = 0 \; at \; y = 0 \; and \; y = L \tag{14}$$

$$\phi_{RAP}(y,0) = \phi_0^{RAP} = 1, \; for \; 0 < y < \alpha L \tag{15}$$

$$\phi_{RAP}(y,0) = \phi_0^{virgin} = 1, \; for \; \alpha L < y < L \tag{16}$$

where: ϕ_{Virgin} = volumetric fraction of virgin binder; ϕ_0^{RAP} = volumetric concentration of RAP binder at t = 0; ϕ_0^{virgin} = volumetric concentration of virgin binder at t = 0; α = fractional thickness of the RAP binder layer; L = total thickness of the combined layers of RAP and virgin binder.

Based on the above boundary conditions, the solution to the Equation (13) can be expressed using Equation (17); this equation applies when the asphalt binder exhibits viscous behavior (Kriz et al. 2014).

$$\phi_{RAP}(y,t) = (1-\phi_0^{virgin})(1-\alpha) - \frac{2}{\pi}(1-\phi_0^{virgin}) \sum_{n=1}^{\infty} \frac{sin(n\pi\alpha)}{n} cos(\frac{n\pi y}{L}) e^{-(\frac{n\pi}{L})^2 Dt} \tag{17}$$

The $|G^*|$ as a function of the volumetric concentration of RAP binder can be estimated if the virgin and RAP binder $|G^*|$ values are known using Equation (18). The applicability of Equation (18) to predict the complex viscosity (i.e., |G*| divided by angular frequency) of mixtures of binders has been verified in past studies (Karlsson et al. 2007, Kriz et al. 2014).

$$|G^*| \; (y,t) = \exp(\phi_{RAP} \ln|G^*|_{RAP} + (1-\phi_{RAP}) \ln|G^*|_{virgin}) \tag{18}$$

where: $|G^*|(y,t)$ = |G*| of the mix of virgin and RAP binders with a RAP binder concentration of ϕ_{RAP} coinciding with position y at time t; $|G^*|_{virgin}$ = $|G^*|$ of the virgin binder, $|G^*|_{RAP}$ = $|G^*|$ of the RAP binder.

If the binder exhibits purely viscous behavior (i.e., phase angle equals 90°), Equation (19) can be used to relate the $|G^*|(t)$ of the layered binder composite measured by the DSR to the gradient in modulus with respect to distance from the bottom plate (y). The $|G^*|$ gradient can be converted to the concentration gradient using Equation (18) to enable the application of Equation (17). A least squares optimization of the $|G^*|(t)$ data measured as a function of conditioning time can thus, be used to determine the diffusion coefficient.

$$|G^*|(t) = \frac{L}{\int_0^L |G^*|(y,t)^{-1} dy} \tag{19}$$

Experimental Procedures

Five experimental procedures to measure the diffusion coefficient between virgin and RAP binders were tried, which are detailed in Table 19. All experiments were conducted using an Anton Paar MCR 502 DSR. The RAP binder was placed below the virgin binder in all experiments conducted to minimize the effect of gravity. The procedures tried varied in terms of several influential factors that are described below.

Table 19. Summary of the Experimental Procedures

Procedure	Sample Diameter mm	Wafer Thickness mm	Preparation Method	Conditioning Method	Conditioning Temperature ºC	Testing Temperature ºC
1	25	0.5	A	Inside DSR	120	120
2	25	0.3	B	Inside DSR	80 – 120	80 – 120
3	50	0.3	B	Inside DSR	100 and 140	100 and 140
4	25	1	C	Outside DSR	140	64
5	25	0.3	B	Inside DSR	120	64

Film thickness

Minimizing the film thickness of the RAP and virgin binder wafers is desirable to minimize both the thermal equilibration time and vertical flow due to gravity. However, thin samples are very delicate. Thus, as the sample film thicknesses reduces, the sample heterogeneity may increase. In this study, it was found that the minimum film thickness that could be achieved was governed by the sample preparation method.

Sample Preparation Methods

Three sample preparation methods were tried in this study, which yielded three different film thicknesses as shown in Table 19.

Method A: In Method A, asphalt binder wafers were prepared within the DSR using a procedure adapted from the one proposed by Kriz et al. (2014). The asphalt binders were first annealed in an oven for the minimum time necessary to achieve workability. The DSR was set to the target temperature for the diffusion experiment and allowed to equilibrate. The DSR gap was then zeroed and the spindle was raised. A pre-calculated mass of the RAP binder to achieve the target film thickness given the sample diameter and density was then placed on the bottom DSR plate. A transparency plastic film was then placed on top of the binder. The spindle was lowered by setting the gap in the DSR equal to the desired thickness sample plus the thickness of the transparency film. The chamber temperature was then reduced to 45°C, which was found to be just high enough to allow insertion and compression of the virgin binder without compression of the RAP binder. The DSR spindle was raised and a pre-calculated mass of virgin binder was applied to upper plate. A second transparency film was placed on the bottom of the virgin binder film and the DSR gap was set to the desired thickness of the two wafers combined plus the thickness of the two transparency films. The normal force was allowed to dissipate and then the DSR temperature was set to 5°C. The samples were conditioned at 5°C for 10 min.

The spindle was raised, and the transparency films were removed. Kriz et al. (2014), trimmed the wafers individually after removing the transparency films but before bringing the wafers in contact. This procedure was tried. However, the authors were unable to accurately trim the samples while adhered to a single plate due to the lack of confinement. Therefore, samples were trimmed after placing the wafers in contact. To do so, the DSR spindle was lowered to bring the two wafers into contact and the temperature of DSR chamber was increased to 60ºC. After reaching 60ºC, excess binder was trimmed using a heated spatula. The DSR temperature was then set to the test temperature, which was 120ºC for the initial trials conducted using Method A. An initial wafer thickness of 500 μm was tried. However, two critical problems were encountered:

It was suspected that trimming at 60ºC using a heated spatula induced mechanical mixing of the wafers. It took the DSR approximately two minutes to ramp up from 60ºC to the conditioning temperature of 120ºC during which significant diffusion may have taken place. These drawbacks led the authors to abandon Method A for sample preparation and hence, no results of Procedure 1 in Table 19 are presented.

Method B: Method B constitutes an alternative method to prepare binder wafer samples. Like Method A, the procedure uses the DSR gap control to compress asphalt binder films to the desired wafer film thickness. However, the RAP and virgin binder wafers are prepared individually and trimmed to the desired diameter outside of the DSR using a specially designed sample cutter akin to a cookie cutter. The procedure is depicted in Figure 29. To prepare the wafers, a binder sample (i.e., virgin or RAP binder) was placed between two thin transparency films as shown in Figure 29 (a). The composite was then placed between the DSR plates and compressed to the desired thickness by setting the DSR gap to the total thickness of the films plus the desired binder film thickness as shown in Figure 29 (b). In this step, temperatures of 70ºC and 90ºC were found to work well for the virgin binder and RAP binder samples evaluated in this study, respectively. Once the sample reached the desired thickness, the normal force was monitored. After the normal force dissipated, the DSR temperature was reduced to -20ºC for 20 minutes after which the sample was removed from the DSR as shown in Figure 29 (c). The transparency films were peeled off and the sample was placed on a flat silicone surface and allowed to equilibrate to room temperature. The sample cutter shown in Figure 29 (d), which has an internal diameter equal to the desired sample diameter, was heated using a heat gun and used to trim the binder sample to the desired diameter as shown in Figure 29 (e). Excess binder was removed as shown in Figure 29 (f) and the sample was placed in a refrigerator to prevent distortion.

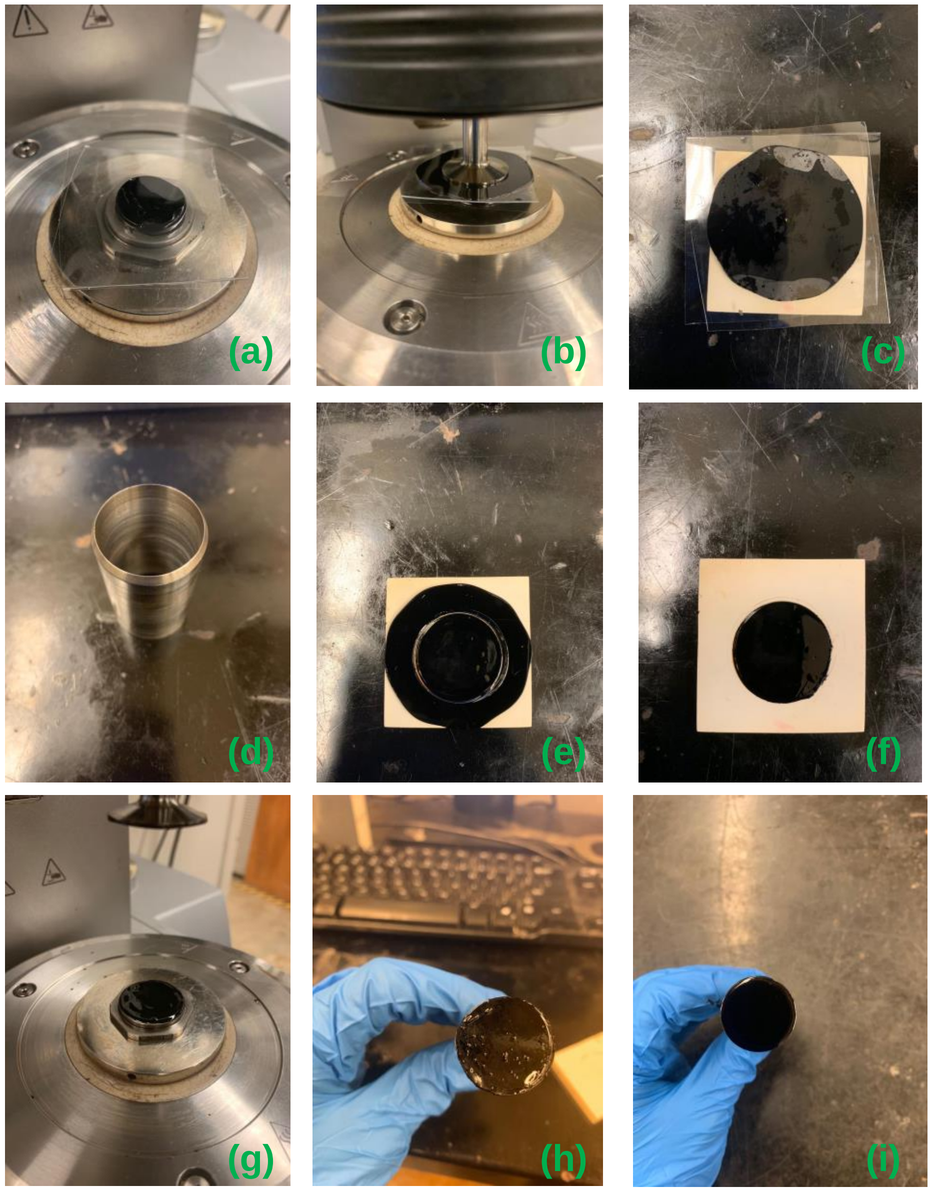

Figure 29. Depiction of Sample Preparation Method B: (a) sample placed on the lower DSR plate between films, (b) sample after compression to the desired thickness, (c) compressed sample after removal from the DSR, (d) sample cutter, (e) sample after application of sample cutter, (f) wafer sample after removal of excess binder, (g) wafers in the DSR, (h) wafer with air bubbles after first coming into contact with DSR plate, and (i) wafer with smooth surface after conditioning at elevated temperature.

Once the virgin and RAP binder wafers had both been prepared, the DSR temperature was set to 70°C. The virgin binder wafer was placed on the spindle and the RAP binder wafer was placed on the lower DSR plate as shown in Figure 29 (g). The temperature was increased to the conditioning temperature. Upon initial contact with the DSR plates, the samples exhibited bubbles as shown in Figure 29 (h). However, after several minutes of conditioning, smooth surfaces were observed as shown in Figure 29 (i). After smooth surfaces were observed, the samples were brought into contact and oscillatory loading commenced. Several film thicknesses were tried when using sample preparation Method B. It was found that a minimum film thickness of 300 μm could be fabricated. The transparency films could not be removed from the sample without tearing if the film thickness was less than 300 μm.

Method C: In Method C, samples were prepared completely external to the DSR following a procedure very similar to those used by Yousefi Rad et al. 2014 and He et al. 2016. The asphalt binder was annealed and poured into a silicone mold with the desired sample diameter on a high precision scale. The mass of binder was monitored during pouring. The mass of binder poured into the sample was pre-calculated to achieve the desired thickness given the binder's specific gravity and sample diameter. The RAP and virgin binder wafers were prepared separately and then brought into contact in a single mold. Due to surface tension between the silicone mold and asphalt binder, it was found that the preparation of samples thinner than 1 mm using this approach is not feasible. When implementing Method C, uniform film thickness within the mold and uniform adherence between the plates and samples when placed between the DSR plates were visually verified prior to testing.

Sample diameter

A larger sample diameter may improve the ability to obtain quality data in DSR diffusion experiments where testing is conducted at the conditioning temperature. Diffusion experiments should seek to minimize the strain amplitude during the DSR test to minimize the effects of mechanical mixing and edge failure. The minimum strain amplitude that can be used while obtaining quality data will theoretically by governed by the minimum oscillatory torque limit of the DSR. Very low shear stresses are required to induce a given strain in asphalt binders at the high temperatures where diffusion is of most interest compared to pavement temperatures at which DSR tests are routinely conducted. The relationship between the torque (T), sample radius (r), and resulting shear stress (τ) in parallel plate DSR tests is given in Equation (20).

$$\tau = \frac{2 \times T}{\pi \times r^3} \tag{20}$$

Equation (20) shows that a larger sample radius requires a larger applied torque to achieve a given shear stress than a smaller sample radius and is thus, more may be more desirable in diffusion experiments. In this study, two sample diameters were considered: 25 mm and 50 mm as indicated in Table 19. The 25-mm diameter samples were tried because 25-mm parallel plates are used in routine asphalt binder characterization and larger diameters are not required in DSR tests conducted at pavement temperatures (i.e., 64°C in this study). The 50-mm parallel plates were tried to reduce the strain amplitudes required in tests while still generating sufficient torque for reliable measurements.

Conditioning procedure

Wafers can be conditioning in contact either inside of or outside of the DSR. The advantage of conditioning the samples within the DSR is that it allows data collection to happen as the diffusion occurs, thereby reducing the number of samples required per experiment to determine the diffusion coefficient. Conditioning the samples outside of the DSR has advantages despite requiring many samples (i.e., one for each conditioning time excluding replicates). The samples can be tested at a lower temperature than that used for conditioning, allowing for the use of low strain amplitudes while still generating sufficient torque to minimize the effects of mechanical mixing. Also, multiple samples can be conditioned simultaneously. However, blending may take place during cooling and transfer of the samples to the DSR.

Procedures that relied on conditioning within and outside of the DSR were both tried, as indicated in Table 19. Several procedures where samples were conditioned within the DSR and tested at the conditioning temperature were tried (i.e., Procedures 1, 2, and 3) as well as a procedure that used a lowered temperature for testing (i.e., Procedure 5). When implementing procedures that used different conditioning and testing temperatures (i.e., Procedures 4 and 5), separate samples were used for each conditioning time evaluated. The conditioning temperatures evaluated ranged from 80°C to 140°C as indicated in Table 19.

<u>DSR testing conditions</u>

To minimize mechanical mixing, the oscillatory strain amplitude that is used to monitor diffusion should be minimized, as previously discussed. In addition, it is important to use a strain amplitude that is sufficiently low to stay within the linear viscoelastic region. Minimizing the applied strain given fixed torque limits of a DSR can be achieved by increasing the sample diameter (as discussed above). In addition, the strain can be minimized by decreasing the test temperature, increasing the loading frequency, or both. Asphalt binders experience an increase in modulus with increasing loading frequency and decreasing temperature due to their inherent viscoelasticity. However, DSR data quality can become compromised at high frequencies and edge failure is more likely to occur as the applied strain rate increases (Hemingway and Fielding 2019). Therefore, procedures that used a testing temperature of 64°C where obtaining quality data at low strain amplitudes is very feasible were tried (i.e., Procedures 4 and 5 in Table 19).

To establish the DSR loading conditions in the procedures where the testing and conditioning procedures were equal, oscillatory stress sweeps were conducted to identify the minimum strain amplitude and maximum loading frequency that could be employed without compromised data quality that could be used. Trends in the dynamic shear modulus as a function of strain amplitude were assessed to ensure that the selected strain amplitude for diffusion experiments was within the linear viscoelastic regime.

Materials

A single virgin binder and artificially produced RAP binder were evaluated in this study to negate the need for costly extraction and recovery. The virgin binder used in this study is a PG 58-28. To produce the artificial RAP binder, this virgin binder was aged in the oven at 120ºC for 3 days to achieve a target high temperature grade of 70ºC. The high temperature grade of the oven aged binder was determined according to AASHTO M 320-17, Standard Specification for Performance-Graded Asphalt Binder, and confirmed to be 70ºC based on the criteria for original binders. Note that the use of a relatively soft virgin binder and corresponding artificial RAP binder was intentional to coincide with the materials that would pose the most challenges associated with testing samples at very high temperature in the DSR; a soft binder will be most prone to edge failure, flow under gravity, and data quality challenges associated with the minimum torque limits of the rheometer. The master curves of the virgin binder and the oven aged binder are shown in Figure 30.

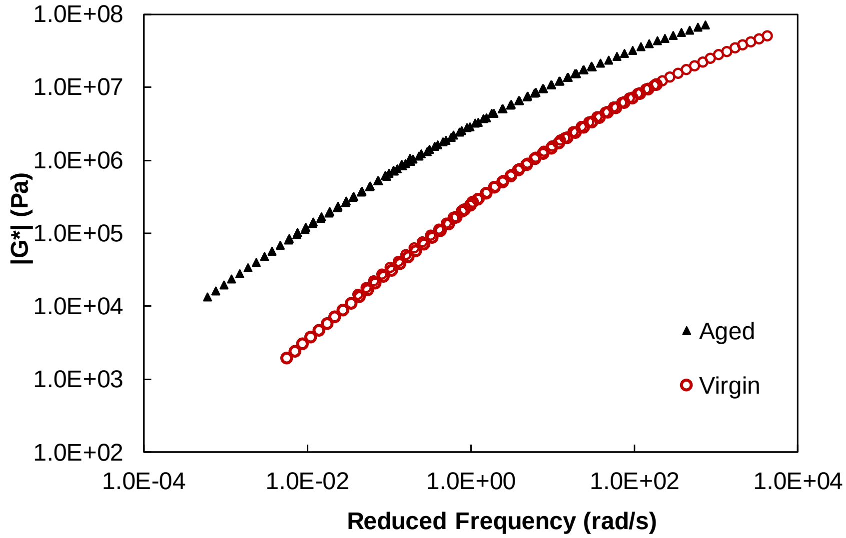

Figure 30. Virgin and aged (i.e., artificial RAP) binder master curves.

Analysis

Several different metrics were used to assess the results of the different procedures evaluated. These are described below.

Waveform Quality

Waveform quality was assessed by analyzing the full stress and strain time history of the experiments. It was visually apparent in all experiments that the strain waveform was sinusoidal and matched the input strain amplitude in the case of strain-controlled tests. However, it was visually apparent that the stress waveforms in some of the experiments were distorted. Therefore, the standard error of the stress waveform was calculated and used to critically evaluate data quality. The standard error of the stress waveform generated was calculated according to Equation (21), which was taken from AASHTO T 342-11, Standard Method of Test for Determining the Dynamic Modulus of Hot Mix Asphalt; note that a comparable data quality statistic is not included in AASHTO T 315-19, Standard Method of Test for Measuring the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer.

$$se = \sqrt{\sum_{i=1}^{n} \frac{(x_i - x)^2}{n-4}} \left(\frac{100\%}{x_0}\right) \tag{21}$$

where: *se* = standard error of the applied stress; x_i = measured stress at point *i*; *x* = expected stress at point *i*; *n* = total number of data points; and x_0 = amplitude of the best fit sinusoidal stress.

To calculate the se, expected stress as a function of time was calculated using the stress amplitude reported by the DSR software combined with the input loading frequency. Data was deemed of acceptable quality when the *se* was less than 5 percent based on the acceptance threshold for dynamic modulus testing of asphalt mixtures given in AASHTO T 342-11.

Trends in the dynamic shear modulus and phase angle with conditioning time

Based on the theoretical background presented, the |G*| of the composite layered sample measured by the DSR should increase with conditioning time due to diffusion. Also, at the very high temperatures used in the procedures where the conditioning and testing temperatures were equal, the phase angle is expected to equal 90º, irrespective of conditioning time, indicating viscous behavior (Kriz et al. 2014). Therefore, trends in the |G*| and phase angle with time were used as an additional metric of the test viability.

Visual Assessment of Samples after Testing

All samples were visually assessed directly following testing and before raising of the DSR spindle to check for edge failure. Edge failure was not visually apparent in any of the samples.

Results and Discussion

The following sections describe the results of the Procedures 2 through 5, detailed in Table 19. Note that results of Procedure 1 are not presented because the procedure was abandoned prior to in-depth testing due to concerns over the sample preparation, as discussed within the Methodology.

Procedure 2

Procedure 2 used 25-mm diameter specimens prepared according to Method B. These specimens were conditioned and tested at the same temperature within the DSR. Pilot test results were conducted on 600-μm thick virgin binder wafer samples (i.e., total thickness of the RAP and virgin binder wafers planned for the diffusion experiments) at 120°C in an effort to select the loading frequency and strain amplitude for diffusion experiments that would yield good data quality while best minimizing mechanical mixing. The virgin binder alone was used for these experiments since it is softer than the RAP and thus, most prone to challenges when tested at high-temperatures in the DSR. Stress-controlled tests were used to ensure the stress amplitude would exceed the rheometer's specified minimum oscillatory torque of 100 nNm by several orders of magnitude.

Initial pilot tests consisted of stress-controlled oscillatory tests conducted at 25 rad/s with an amplitude of 5 Pa. It was expected that the asphalt binder would exhibit purely viscous behavior under these conditions based on the results reported by Kriz et al. (2014). However, the phase angle values reported by the DSR software were found to vary considerably among replicate tests. Initially, it was hypothesized that these unexpected trends were the result of air bubbles within the samples. However, samples were carefully examined before and after testing and bubbles were not visually apparent. Therefore, the waveform of the applied stress was evaluated to better understand the source of the unexpected phase angle values.

The applied stress waveform differed from the expected waveform as shown in Figure 31, indicating that the test conditions did not yield reliable data despite exceeding the specified minimum oscillatory torque limit of the DSR. However, torque generated by surface tension can compromise the ability to measure the response of very soft materials. Surface tension does not produce a torque in ideal (symmetric) samples. In contrast, if a binder sample is rotationally asymmetric due to slight under filling on one side of the sample, as shown schematically in Figure 32, surface tension caused by traction forces at the periphery of material contact with the parallel plates can lead to the generation of torque that may be significant if the torque carried by the material is very small (Johnston and Ewoldt 2013). Surface tension torque effectively raises the minimum torque limit of the DSR because it cannot be deterministically isolated from the torque felt by the material (Johnston and Ewoldt 2013). Asymmetric samples herein could have resulted from imprecise trimming of the samples using the sample cutter during preparation or the failure to accurately center the sample when placed in the DSR. Wall slip between the plate and sample can also lead to asymmetric or chaotic distortion of the stress waveform in oscillatory experiments (Walter et al. 2017).

To further evaluate the source of poor data quality and identify test conditions that would yield quality data, additional stress-controlled tests with varying stress levels and temperatures were conducted on 600-μm thick virgin binder sample. All tests were conducted at 25 rad/s. The corresponding results are presented in Table 20 and Figure 32. At temperatures exceeding 80°C, the stress waveform distortion is considered significant based on stress standard error threshold of 5 percent specified in AASHTO T 342.

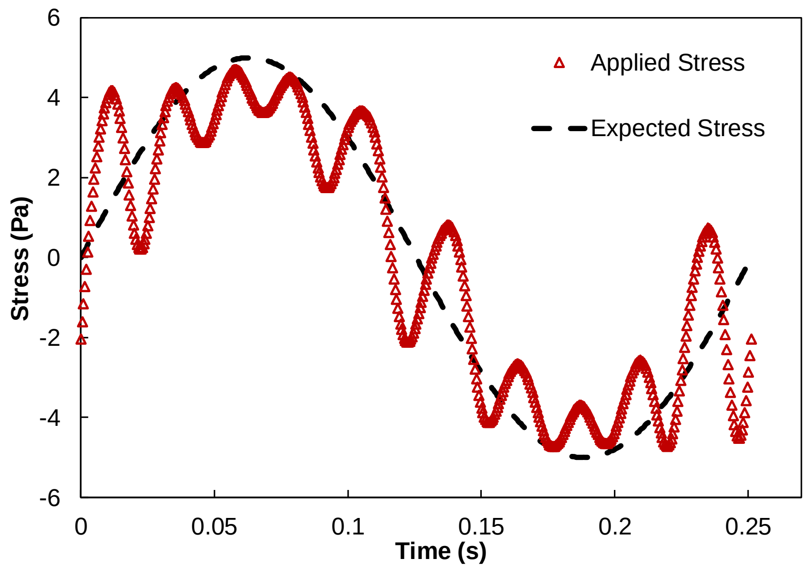

Figure 31. Variation between expected stress and applied stress for 5 Pa, 25 rad/s at 120 ºC.

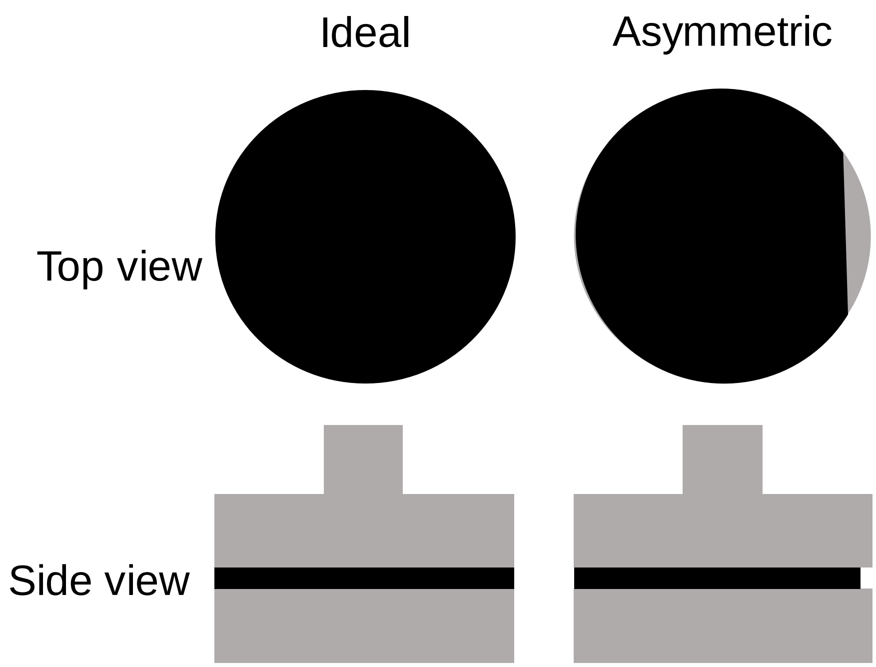

Figure 32. Ideal versus asymmetric geometries. Note that the asymmetry is exaggerated for illustrative purposes.

While not universally followed, the results demonstrate that the data quality generally improves as the temperature decreases for a given stress amplitude. Furthermore, the results demonstrate that at temperatures of 100°C or higher, the data quality generally improves as the stress amplitude increases. The relative contribution of surface tension torque to the total torque measured by the rheometer increases as the torque carried by the material decreases. The torque carried by the material decreases when the temperature is increased (due to a reduction in the asphalt binder modulus), the stress amplitude is decreased, or both. Thus, the results shown in Figure 33 generally suggest that surface tension torque could be contributing to the poor waveform quality, prohibiting reliable measurements at temperatures exceeding 80°C using the virgin binder and conditions evaluated under Procedure 2 in this study. Wall slip is another potential contributing factor. However, the extent of wall slip is expected to increase as the stress (and strain) amplitude increase (Walter et al. 2017). Therefore, wall slip alone cannot explain the observed trends.

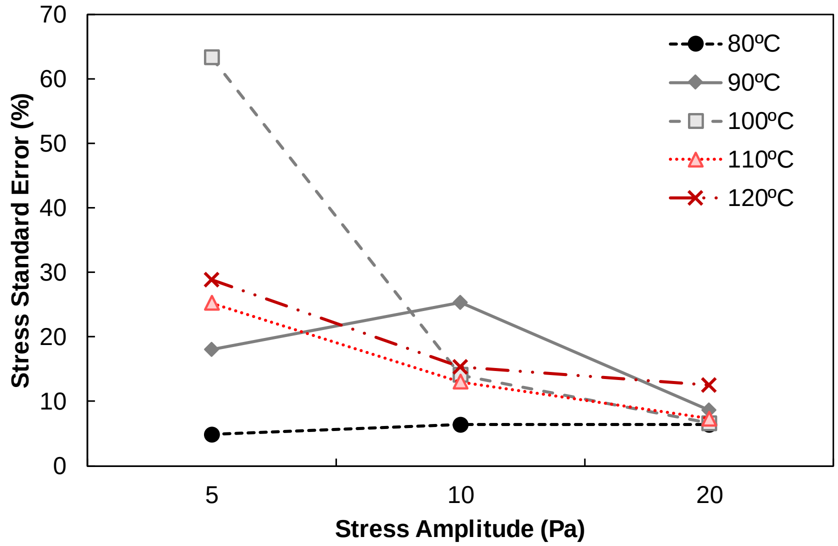

Figure 33. Effect of stress amplitude and temperature on the stress standard error using Procedure 2 at 25 rad/s on virgin binder samples.

Table 20. Procedure 2 Test Results Conducted on Virgin Binder Samples at 25 rad/s

Temperature, °C	Input Stress Amplitude Pa	Resultant Strain Amplitude %	Stress Standard Error %
80	5	1	4.90
	10	2	6.37
	20	4	6.37
90	5	3	18.08
	10	5	25.31
	20	11	8.63
100	5	6	63.34
	10	12	14.06
	20	24	6.61
110	5	12	25.25
	10	25	13.02
	20	49	7.33
120	5	23	28.82
	10	0.45	15.31
	20	0.91	12.48

Procedure 3

Procedure 3 closely followed Procedure 2 with the exception that the sample diameter was increased from 25 mm to 50 mm in an effort to increase the torque carried by the material for a given stress level. Pilot tests were conducted using 600-µm thick virgin binder samples to identify suitable test conditions for the diffusion experiment. All pilot tests were conducted at 140°C to constitute a more representative hot-mix asphalt production temperature than those used in Procedure 2. Initial oscillatory pilot tests were conducted in stress-control mode using varying frequencies and stress levels. The resulting strain amplitude, |G*| and phase angle reported by the DSR software, and the stress standard error were used to evaluated the pilot test results; these results are shown in Table 21. In addition, the effects of loading frequency and stress amplitude on the stress standard error are present in Figure 34. The results obtained at 20 rad/s demonstrate that the increased sample diameter from 25 mm to 50 mm greatly improves the ability to obtain reliable results. When the stress amplitude is at or exceeds 2 Pa, the 50-mm diameter sample experiments conducted at 140°C and 20 rad/s (shown in table 21) yield comparable standard error values to those at 80°C and 25 rad/s using 25-mm diameter samples (shown in Table 20).

Figure 34 shows that the stress standard error decreases as the stress level increases, which matches expectations if surface tension torque is a contributing factor to the waveform quality. Surface tension torque is expected to remain constant with the applied stress level. The effect of loading frequency on the significance of surface tension torque is less intuitive. Figure 34 also shows that the stress waveform quality improves as the loading frequency decreases for a given stress level, suggesting that the effective minimum torque limit of the rheometer may be frequency dependent.

Table 21 demonstrates that the DSR test output consistently indicates a phase angle of 90° for all test conditions, as expected, indicating the binder exhibits purely viscous behavior for the test conditions evaluated at 140°C. These results demonstrate that at a frequency of 20 rad/s, the |G*| remains constant for stress levels where the stress standard error is low (i.e., 2 Pa, 5 Pa, 10 Pa, and 20 Pa); this indicates that these test conditions are within the linear viscoelastic regime.

Table 21. Procedure 3 Pilot Test Results Conducted in Stress-control Mode at 140ºC

Input Stress Amplitude Pa	Frequency rad/s	Resultant Strain Amplitude %	Reported |G*| Pa	Reported Phase Angle º	Stress Standard Error %
1	20	27	3.7	90	28.40
1	50	11	9.0	90	284.46
1	100	6	16.6	90	376.51
2	20	55	3.6	90	8.73
2	50	22	9.1	90	90.14
2	100	11	18.1	90	598.55
5	20	140	3.6	90	2.98
5	50	55	9.1	90	32.22
5	100	28	17.8	90	64.15
10	20	277	3.7	90	1.95
10	50	112	9.1	90	28.48
10	100	55	18.1	90	22.8
20	20	550	3.6	90	1.78

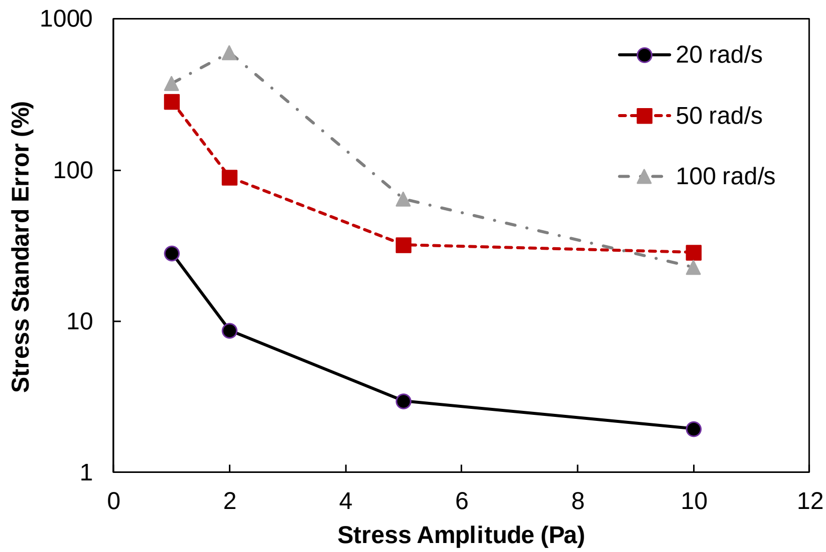

Figure 34. Effect of stress amplitude and frequency on the stress standard error of virgin binder samples tested at 140°C using Procedure 3.

Next, oscillatory strain sweep pilot tests were conducted using 600-µm thick virgin binder samples at 140°C and a frequency of 10 rad/s to select the final test conditions for the diffusion experiments. The loading frequency of 10 rad/s was used because the stress-controlled pilot test results suggested that decreasing the testing frequency improves the data quality for a given stress level. The results of the oscillatory strain sweeps are shown in Figure 35 and Table 22. Figure 35 demonstrates the stress standard error is very low when the strain amplitude is greater than or equal to 100 percent. Furthermore, Table 22 demonstrates that the |*G**| and phase angle values were independent of strain amplitude, indicating they fall within the linear viscoelastic regime. Therefore, 100 percent was selected as the minimum limit for the applied strain amplitude for the diffusion experiments conducted using Procedure 3 at a temperature of 140°C and 10 rad/s.

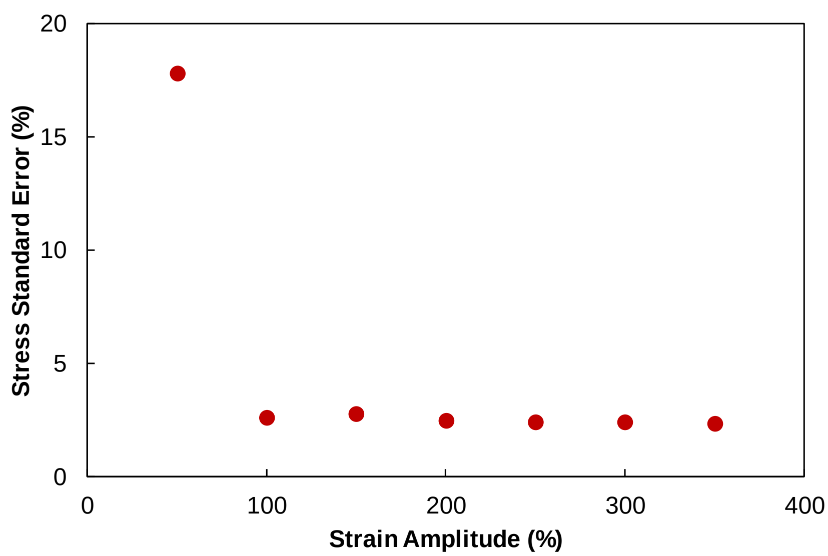

Figure 35. Effect of strain amplitude on standard error at 140°C.

Table 22. Effect of Strain on the Quality of Waveform at 140°C

Input Strain Amplitude %	Frequency rad/s	Resultant Stress Amplitude Pa	Reported |G*| Pa	Reported Phase Angle °	Stress Standard Error %
50	10	0.9	1.8	90	17.82
100	10	1.8	1.8	90	2.61
150	10	2.7	1.8	90	2.77
200	10	3.7	1.8	90	2.48
250	10	4.6	1.8	90	2.42
300	10	5.5	1.8	90	2.43
350	10	6.4	1.8	90	2.36

Diffusion experiments were conducted using 300-µm thick virgin and RAP binder wafers conditioned at 140°C with applied oscillatory loading at 10 rad/s using 100 percent strain amplitude. In these experiments, the phase angle was consistently 90°. The results indicated that the composite sample acquired a constant $|G^*|$ value within seconds, which was approximately equal to that of the completely blended sample according to Equation (18); therefore, it was hypothesized that significant mechanical mixing was induced from the strain amplitude used.

To further evaluate this hypothesis, additional diffusion experiments were conducted at 100°C where the diffusion process was expected to occur more slowly. Two oscillatory loading amplitudes at frequency of 10 rad/s were tried: 100 percent and 200 percent strain amplitude. The results are shown in Figure 36. The initial data point for each test displayed a markedly higher modulus than the second data point. The waveform quality in the initial cycle was poor, which is speculated to have caused the unexpectedly high modulus values. Poor data quality in the first cycle of a DSR test is common irrespective of the test conditions. While not visually evident, it is also possible that edge failure may have occurred reducing the effective sample diameter. The results show the expected trend of an increase in $|G^*|$ with conditioning time initially followed by a plateau, indicating complete blending. Initially, the initial rate of increase in $|G^*|$ is higher for the 100 percent strain condition. However, it can be seen that the 200 percent strain amplitude yields a shorter time to reach complete blending than the 100 percent strain amplitude, indicating that the applied strains induce significant mechanical blending, which confounds the measurement of diffusion.

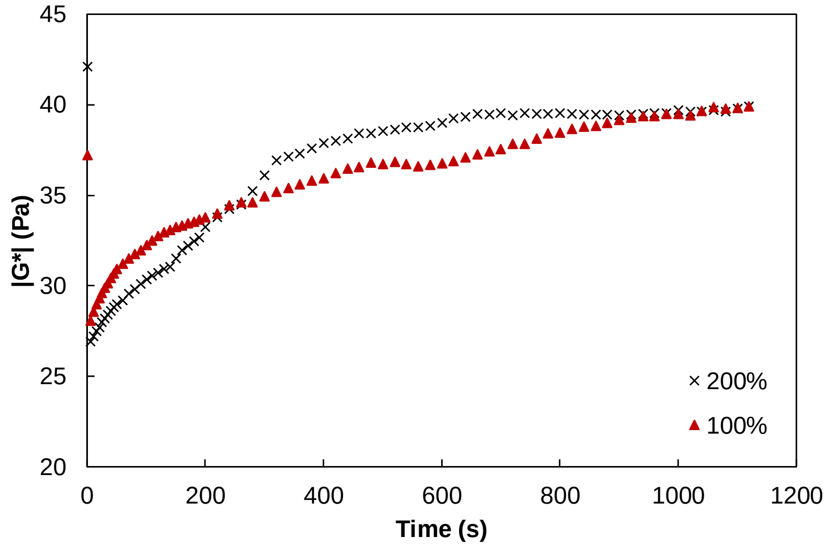

Figure 36. Effect of strain amplitude on mechanical blending.

It should be noted that past studies have reported the use of much smaller strain amplitudes and higher frequencies than employed herein despite similar rheometer specifications and test temperatures (Karlsson et al. 2007, Kriz et al. 2014). However, the stress waveform quality was not reported and is therefore, unknown.

Procedure 4

Procedure 4 was tried to avoid the needed to conduct oscillatory measurements at very high temperatures and thus, enable the use of smaller strains to minimize mechanical mixing. In Procedure 4, samples were prepared and conditioned outside of the DSR and then tested in the DSR at 64°C using 2 percent strain amplitude at a loading frequency of 10 rad/s. Procedure 4 herein was similar to the procedures employed by Yousefi Rad et al. 2014 and He et al. 2016. The stress standard error was well below 5 percent under these test conditions. Two replicate samples were prepared for each conditioning time. To mitigate the influence of oxidative aging on the test results, all samples underwent the same thermal history. All samples were first heated to allow pouring into a silicone mold. Then, pre-calculated masses of virgin and RAP binder to yield a thickness of 1 mm were poured into separate 25-mm and conditioned at 135°C for 15 min. Next, RAP and virgin binder wafers were 'aged' at 140°C while still isolated such that the combined aging time and conditioning time where the binders were in contact was 30 minutes for all conditioning durations evaluated. After aging for the prescribed time, the samples were placed in contact and conditioned at 140°C for the prescribed conditioning time. Following conditioning, samples were carefully transferred to a refrigerator for 15 min prior to DSR testing.

The Procedure 4 results are shown in Table 23, which demonstrates that the values of $|G^*|$ of the composite samples do not vary with conditioning time. Furthermore, the $|G^*|$ values are approximately equal to the theoretical modulus of the completed blended sample according to Equation (18). Thus, the result indicates that complete blending is attained even at low conditioning times. It is speculated that complete blending was caused by mechanical mixing induced during movement of the samples from the oven to the refrigerator.

Table 23. Procedure 4 Results when Samples were Transferred to a Refrigerator for Cooling

Annealing time min	Aging time min	Conditioning time min	|G*| Rep 1 Pa	|G*| Rep 2 Pa
15	25	5	1,569	1,686
15	20	10	1,638	1,542
15	10	20	1,530	1,575

In an effort to reduce mechanical mixing, additional trials of Procedure 4 were conducted in which the binders were cooled in liquid nitrogen immediately following conditioning in close proximity to the oven. The composite |*G**| results are shown in Table 24. Similar to Table 23, the results demonstrate |*G**| values approximately equal to that of the fully blended sample irrespective of the conditioning time. Therefore, movement of the samples into the liquid nitrogen may have still induced mechanical mixing.

Table 24. Procedure 4 Results when Samples were Cooled using Liquid Nitrogen

Annealing time min	Aging time min	Conditioning time min	|G*| Rep 1 Pa	|G*| Rep 2 Pa
15	25	5	1,663	1,705
15	20	10	1,758	1,701
15	10	20	1,663	1,575

The means to account for oxidative aging in the interpretation of test results used herein differed from past studies (Yousefi Rad et al. 2014, He et al. 2016), which may have contributed to the contradictory findings regarding the viability of the method. The past studies relied on measuring the evolution of the |*G**| of a fully blended sample conditioned at the same temperature as the wafer samples rather than ensuring the total conditioning times of all prepared samples were the same. The slope of |*G**| versus time of the fully blending sample was used to adjust the |G*| results of the fully blended samples (Yousefi Rad et al. 2014, He et al. 2016).

Past studies have suggested that complete blending of RAP and virgin binders does not occur in asphalt mixtures (McDaniel and Anderson 2001). Thus, the laboratory results herein that suggest complete blending induced by mechanical mixing of DSR binder specimens through limited handling does not necessarily translate into complete blending of binders during asphalt mixture preparation (either in the laboratory or within an asphalt plant) due to factors that are not replicated in the DSR wafer experiment (e.g., aggregate absorption, physical interactions between coated aggregate particles, etc.). Future research is, therefore, needed to evaluate mechanical mixing in asphalt mixtures.

Procedure 5

A final procedure was tried to further mitigate mechanical mixing. In Procedure 5, 300-μm thick wafer samples with 25-mm diameter were prepared according to Method B and conditioned using the same procedure implemented in Procedure 2 but without the application of oscillatory loading. Following conditioning, samples were cooled within the DSR to 64°C and then subjected to oscillatory loading at 2 percent strain amplitude using a loading frequency of 10 rad/s, which resulted in stress standard error values that were all below 5 percent. A conditioning temperature of 120°C was used in all experiments. Separate samples were prepared for each conditioning time.

The |*G**| results of the composite wafer specimens are shown in Figure 37. The results show no clear trend in |*G**| with conditioning time. While the |*G**| values vary, all are well below that of the fully blended sample, which is 1,478 Pa according to Equation (18). Despite the authors' best efforts to ensure the consistency of the samples produced, it is speculated that variability in the film thickness of the individual binder wafers may have contributed to the variability in the reported |*G**| values. However, given the lack of a clear trend with time, it is also deduced that minimal blending of the RAP and virgin binders occurred in the absence of mechanical mixing. Therefore, it is speculated that quantifying the diffusion coefficient would yield limited information about blending in RAP mixtures due to the important role of mechanical mixing.

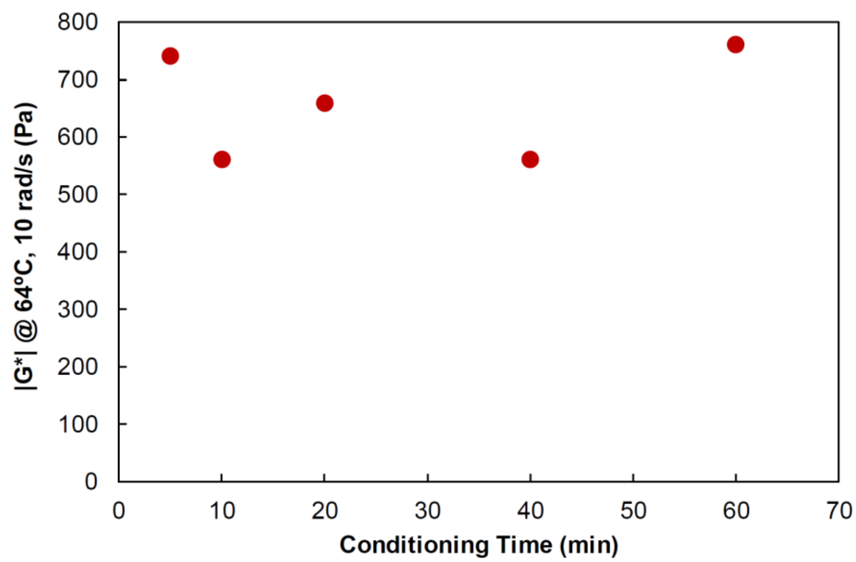

Figure 37. Procedure 5 diffusion experiment results where samples were conditioned at 120°C and tested at 64°C.

Conclusions and Recommendations

The diffusion coefficient between RAP and virgin binders could not be quantified using DSR-based experiments in this study due to the following findings:

When samples were conditioned within the DSR at hot-mix asphalt production temperatures, poor stress waveform quality, speculated to be caused by surface tension torque, precluded the application of oscillatory loading using sufficiently low strain amplitudes to prevent mechanical mixing. The use of relatively large, 50-mm diameter, samples did not alleviate the data quality limitations.

When binder samples were conditioned externally to the DSR, mechanical mixing was induced when the samples were removed from the conditioning chamber. This mechanical mixing was found to lead to complete blending of the binder samples based on DSR measurements. It also suggests that the blending of binder wafer samples does not fully replicate blending within asphalt mixtures where incomplete blending has been inferred despite mechanical mixing during mixture fabrication.

In the absence of mechanical mixing, time-dependent blending between binders specimens conditioned in contact in the DSR at 120°C was not observed, suggesting diffusion was minimal.

Based on the lack of observed diffusion in the absence of mechanical mixing, it is hypothesized that understanding mechanical mixing is critical to understanding blending in RAP mixtures. This supports findings from microscopy and sieve analysis that suggests agglomerations of adhered RAP and RAS particles are the primary inhibitors of blending and that when recycled and virgin binders are placed in contact under mixing conditions that blending generally ensues.

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APPENDIX C: EXAMPLE MIXTURE VOLUMETRIC CALCULATIONS WHEN CONSIDERING RECYCLED BINDER AVAILABILITY

An example of the calculation of the compacted asphalt mixture volumetric properties on the basis of availability as described by Equations (7) through (11) is presented in this appendix. The calculation of the available VMA, available VFA, available DP and effective RBR values shown in Table 25 for the redesigned Mix C40 are demonstrated. The basis for the example calculations that follow is an assumed total mass of the asphalt mixture of 100 g. The input properties for the following calculations of Mix C40 are given in Table 25.

Table 25. Summary of the Redesigned Mix C40 Properties

Property	Value
Mix bulk specific gravity (G_{mb})	2.309
Mix air void content (V_a)	4.0%
Total asphalt content of the mix ($P_{b\ total}$)	7.0%
Virgin asphalt content of the mix ($P_{b\ virgin}$)	4.8%
Mix aggregate percent passing the 0.075 mm sieve when using the 'black curve' to represent the RAP gradation ($P_{0.075}$)	5.0%
Virgin aggregate blend bulk specific gravity ($G_{sb\ virgin\ aggregate}$)	2.640
RAP content of mix, expressed as the aggregate stockpile percentage comprised of RAP aggregate ($RAP_{content}$)	40%
RAP aggregate effective specific gravity ($G_{se\ RAP}$)	2.761
RAP asphalt content ($P_{b\ RAP}$)	5.7%
RAP recycled binder availability ($Availability_{RAP}$)	62%
Specific gravity of binder (G_b)	1.02

The available VMA calculation considers the unavailable recycled binder as part of the bulk aggregate volume. Correspondingly, calculation of the available VMA using Equation (9) requires the total volume of mix (V_{mb}) and the bulk volume of virgin aggregate ($V_{sb\ virgin}$), effective recycled aggregate volume ($V_{se\ RAP}$), and unavailable recycled binder volume ($V_{unavailable\ RAM\ binder}$). The V_{mb} can be obtained from the measured bulk specific gravity of the mix (G_{mb}) using Equation (22).

$$V_{mb} = \frac{100}{G_{mb}} = \frac{100}{2.309} = 43.3 \tag{22}$$

The revised calculation of the mixture V_{sb} when considering availability (i.e., Equation (9)) requires the bulk volume of virgin aggregates ($V_{sb\ virgin}$), the effective RAP aggregate volume ($V_{se\ RAP}$) and the volume of unavailable recycled binder ($V_{unavailable\ RAM\ binder}$). To obtain the bulk volume of virgin aggregates, the mass of virgin aggregates in the mix ($M_{virgin\ aggregate}$) is needed, and can be obtained by subtracting the mass of RAP aggregates ($M_{RAP\ aggregate}$) from the total mass of aggregates in the mix (P_s). Correspondingly, Equation (23) is first applied to obtain P_s given the total binder content of the mix given in Table 25.

$$P_s = 100 - P_{b\ total} = 100 - 7.0 = 93 \tag{23}$$

The $M_{RAP\ aggregate}$ is then obtained by multiplying the P_s by the RAP content in the mix, as shown in Equation (24).

$$M_{\mathrm{RAP}\ aggregate} = RAP_{content} \times P_s = 40\% \times 93 = 37.2 \qquad (24)$$

The $M_{virgin\ aggregate}$ can then be calculated by subtracting the $M_{RAP\ aggregate}$ from the P_s as shown in Equation (25).

$$M_{virgin\ aggregate} = P_s - M_{\mathrm{RAP}\ aggregate} = 93 - 37.22 = 55.8 \qquad (25)$$

Using the $M_{virgin\ aggregate}$ and the measured bulk specific gravity of the virgin aggregates ($G_{sb\ virgin\ aggregate}$), the $V_{sb\ virgin}$ is calculated according to Equation (26).

$$V_{sb\ virgin} = \frac{M_{virgin\ aggregate}}{G_{sb\ virgin\ aggregate}} = \frac{55.8}{2.640} = 21.1 \qquad (26)$$

Similarly, the effective RAP aggregate volume (V_{se}) is calculated using the RAP aggregate mass and RAP aggregate effective specific gravity ($G_{se\ RAP}$) using Equation (27).

$$V_{se\ RAP} = \frac{M_{RAP\ aggregate}}{G_{se\ RAP}} = \frac{37.22}{2.761} = 13.48 \qquad (27)$$

The $V_{unavailable\ RAM\ binder}$ (Equation (8)) is calculated with the measured RAP recycled binder availability and the volume of RAP binder in the mix ($V_{RAP\ binder}$). The $V_{RAP\ binder}$ is obtained by subtracting the virgin binder content from the total binder content, as per Equation (28), and then the $V_{unavailable\ RAM\ binder}$ is calculated as shown in Equation (29) below.

$$V_{RAP\ binder} = P_{b\ total} - P_{b\ \mathrm{virgin}} = 7.0 - 4.8 = 2.2 \qquad (28)$$

$$V_{unavailable\ RAM\ binder} = V_{RAP\ binder} \times (1 - Availability_{RAP}) = 2.2 \times (1 - 0.62) = 0.82 \qquad (29)$$

Subsequently, the V_{sb} is calculated according to Equation (30), which coincides with Equation (9).

$$V_{sb} = V_{sb\ virgin} + V_{se\ RAP} + V_{unavailable\ RAM\ binder} = 21.15 + 13.48 + 0.82 = 35.5 \qquad (30)$$

With the calculated V_{mb} and V_{sb}, the available VMA (Equation (11)) is obtained through Equation (31).

$$VMA = \frac{V_{ma}}{V_{mb}} = \frac{V_{mb} - V_{sb}}{V_{mb}} \times 100\% = \frac{43.3 - 35.5}{43.3} \times 100\% = 18.1\% \qquad (31)$$

The percent voids filled with asphalt (VFA) is then calculated according to the available VMA using Equation (32).

$$VFA = \frac{VMA - V_a}{VMA} \times 100\% = \frac{18.1 - 4.0}{18.1} \times 100\% = 78.0\% \qquad (32)$$

The dust-to-binder ratio (*DP*) also changes in the redesigned mixes due to the changes in the effective binder content (P_{be}) and gradation. The P_{be} of the mix is obtained by multiplying the effective volume of binder in the mix (V_{be}) by the specific gravity of the binder (G_b) (Equation (33)). The amount of material passing the No. 200 sieve (P_{200}) for the aggregate blend when using the black curves to represent the RAM gradation is lower than when using the white curves. The available DP is calculated in Equation (34).

$$P_{be} = V_{be} \times G_b = \left(\left(V_{mb} \times (VMA - V_a)\right)\right) \times G_b = \left(43.3 \times \left(18.1\% - 4.0\%\right)\right) \times 1.02 = 6.2\% \tag{33}$$

$$DP = \frac{P_{200}}{P_{be}} = \frac{5.0\%}{6.2\%} = 0.8 \tag{34}$$

Lastly, the effective RBR is calculated in Equation (35), according to Equation (7) presented previously, with the measured availability and the known volumes of RAP binder ($V_{RAP\ binder}$) and virgin binders ($V_{virgin\ binder}$).

$$Effective\ RBR = \frac{V_{RAP\ binder} \times Availability_{RAP}}{V_{RAP\ binder} \times Availability_{RAP} + V_{virgin\ binder}} = \frac{2.2 \times 0.62}{2.2 \times 0.62 + 4.8} = 22.3\% \tag{35}$$