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Research on High-RAP Asphalt Mixtures with Rejuvenators - Phase II

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16. Abstract: A previous study by the authors have demonstrated effectiveness of three rejuvenators: R1 (triglyceride/fatty acid: agriculture-tech based), R2 (aromatic extract: petroleum-tech based), and R3 (tall oil: green-tech based) on rejuvenating properties of the aged binder. In that study, it was observed that the rejuvenators made high-RAP mixtures softer and more compliant, which may increase the rutting potential, while they simultaneously improve cracking resistance of the high-RAP materials. Research outcomes and findings from the previous study resulted in consequential research needs for more specific investigation of high-RAP mixtures with rejuvenators in order to achieve realistic implementation into future high-RAP paving projects in Nebraska. This study thus aimed to investigate the effects of type, dosage, and treating methods of rejuvenators when they are added in aged asphalt materials. To meet the goal, we used the three rejuvenators (R1, R2, and R3) by conducting various binder-level and mixture-level tests in this study. For the binder-level testing, the performance grading (PG) method was used to primarily determine proper dosages targeting desired binder grades, and two chemical tests (i.e., Fourier Transform Infrared and Saturates-Aromatics-Resins-Asphaltenes analysis) were also conducted to examine chemical characteristics altered by rejuvenation and further aging process. The selected dosage levels from the binder testing were then applied to asphalt concrete (AC) mixture-level performance evaluation by conducting two tests: flow number for rutting and semicircular bending fracture with and without moisture conditioning for cracking. AC mixtures treated with rejuvenators at the dosage levels selected from the binder PG testing showed improved fracture resistance compared to unrejuvenated mixtures. Test-analysis results also indicated that PG binder testing, although it can successfully determine the proper dosage range of rejuvenators, is limited by only assessing the effects of rejuvenators in mechanical properties, which can be better aided by integrating chemical characterization that provides a more in-depth material-specific rejuvenation process. In addition, it appears that rejuvenation methods (e.g., blending and/or curing) can alter performance of aged mixtures. Therefore, the selection of rejuvenators and their implementation into practice should be carried out by considering multiple aspects not only by its PG recovery.

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Abstract

A previous study by the authors have demonstrated effectiveness of three rejuvenators: R1 (triglyceride/fatty acid: agriculture-tech based), R2 (aromatic extract: petroleum-tech based), and R3 (tall oil: green-tech based) on rejuvenating properties of the aged binder. In that study, it was observed that the rejuvenators made high-RAP mixtures softer and more compliant, which may increase the rutting potential, while they simultaneously improve cracking resistance of the high-RAP materials. Research outcomes and findings from the previous study resulted in consequential research needs for more specific investigation of high-RAP mixtures with rejuvenators in order to achieve realistic implementation into future high-RAP paving projects in Nebraska. This study thus aimed to investigate the effects of type, dosage, and treating methods of rejuvenators when they are added in aged asphalt materials. To meet the goal, we used the three rejuvenators (R1, R2, and R3) by conducting various binder-level and mixture-level tests in this study. For the binder-level testing, the performance grading (PG) method was used to primarily determine proper dosages targeting desired binder grades, and two chemical tests (i.e., Fourier Transform Infrared and Saturates-Aromatics-Resins-Asphaltenes analysis) were also conducted to examine chemical characteristics altered by rejuvenation and further aging process. The selected dosage levels from the binder testing were then applied to asphalt concrete (AC) mixture-level performance evaluation by conducting two tests: flow number for rutting and semicircular bending fracture with and without moisture conditioning for cracking. AC mixtures treated with rejuvenators at the dosage levels selected from the binder PG testing showed improved fracture resistance compared to unrejuvenated mixtures. Test-analysis results also indicated that PG binder testing, although it can successfully determine the proper dosage range of rejuvenators, is limited by only assessing the effects of rejuvenators in mechanical properties, which can be better aided by integrating chemical characterization that provides a more in-depth material-specific rejuvenation process. In addition, it appears that rejuvenation methods (e.g., blending and/or curing) can alter performance of aged mixtures. Therefore, the selection of rejuvenators and their implementation into practice should be carried out by considering multiple aspects not only by its PG recovery.

Chapter 1 Introduction

The use of reclaimed asphalt pavement (RAP) materials in producing asphalt mixtures offers great benefits by reducing costs for producers and highway agencies as well as reducing the environmental impact associated with the extraction, transportation, and processing of virgin materials. Currently, most states allow up to 40–50% of RAP for primary types of asphalt mixtures. Maximum use of RAP materials into asphalt mixtures has been desired; however, it is not a simple task because of undesired inherent characteristics of RAP such as aged (stiff) asphalt binder and inconsistent aggregate properties. To overcome the inherent concerns of RAP materials, many researchers have made significant efforts in various ways.

Incorporation of the rejuvenators in the high-RAP asphalt mixtures implies clear economical-technical-environmental benefits; however, the effects of complex blending on mixture properties and long-term performance leave many questions remaining. In an attempt to investigate the effects of rejuvenators on high-RAP mixture properties and performance characteristics, for the last three years, the authors have conducted a research project on high-RAP mixtures treated with different rejuvenators [1]. 65% RAP was applied to a typical Nebraska mixture (i.e., SPR) by applying three different rejuvenation additives: the R1 (triglyceride/fatty acid: agriculture-tech based), R2 (aromatic extract: petroleum-tech based), and R3 (tall oil: green-tech based). The research project evaluated various mechanical and chemical properties of asphalt concrete (AC) mixtures, fine aggregate matrix (FAM) mixtures, and binders modified by the rejuvenators and a warm mix asphalt (WMA) additive (i.e., Evotherm). Test results in multiple scales (i.e., AC, FAM, and binder) demonstrated that the rejuvenators made high-RAP mixtures softer and more compliant (ductile), which may increase the rutting potential, while they helped improving cracking resistance of the high-RAP materials.

Research outcomes and findings from the previous effort resulted in consequential research needs for more specific investigation of high-RAP mixtures with rejuvenators in order to achieve realistic implementation into future high-RAP paving projects. In the previous research, as Table 1-1 shows, the dosage of rejuvenators and their blending procedures were followed based on producers' recommendation. Although the recommended practices satisfied research goals set for the previous study, it is necessary to further investigate how mixtures/materials perform due to different treatments of rejuvenators so that an optimum rejuvenation practice can be achieved.

Table 1-1. Dosages and blending methods of rejuvenators used in phase I research.

Additives	Type	ID	Description
Triglyceride/Fatty Acid	Agriculture Technology	R1	dosage: 5% of virgin binder (or 1.6% of total binder) Added to virgin binder for HMA and WMA
Aromatic Extract	Petroleum Technology	R2	dosage: 9% of RAP binder (or 6.2% of total binder) Added to virgin binder for HMA and WMA
Tall Oil	Green Technology	R3	dosage: 0.65% of RAP material (or 8.2% of total binder) Added to HMA batch
Warm Mix Additive	M1 Formulation	AS	dosage: 0.9% of virgin binder (or 0.29% of total binder) Added to virgin binder for WMA

1.1 Research Objectives and Scope

The overall goal of this research effort is to investigate how high-RAP mixtures and materials perform due to different applications (dosages and blending methods) of rejuvenators so that an optimum rejuvenation practice can be explored. More specifically, different rejuvenators were tested by changing their dosages applied to the typical mixture/binder to determine optimum mixture designs with rejuvenators. The effects of rejuvenation procedures (e.g., treatment to RAP, treatment to entire mixture, or treatment to virgin binder, etc.) that are case-specific were also investigated. For binders, the Fourier transform infrared (FT-IR) spectroscopy, saturates-aromatics-resins-asphaltenes (SARA) analysis, and dynamic shear rheometer (DSR) were applied to characterize the chemical and mechanical aspects of the asphalt binders. For the testing of AC mixtures, the flow number and semicircular bending (SCB) fracture tests (dry and wet condition) were conducted.

1.2 Research Methodology

To meet the objectives, an experimental method was proposed and conducted. Figure 1-1 and Figure 1-2 presents the testing plan designed for this study. As shown in Figure 1-1 , first the optimum range of each rejuvenator is found using Superpave performance grade (PG) tests and short and/or long-term performance of rejuvenated binder is evaluated by various chemical and rheological tests (Figure 1-1).

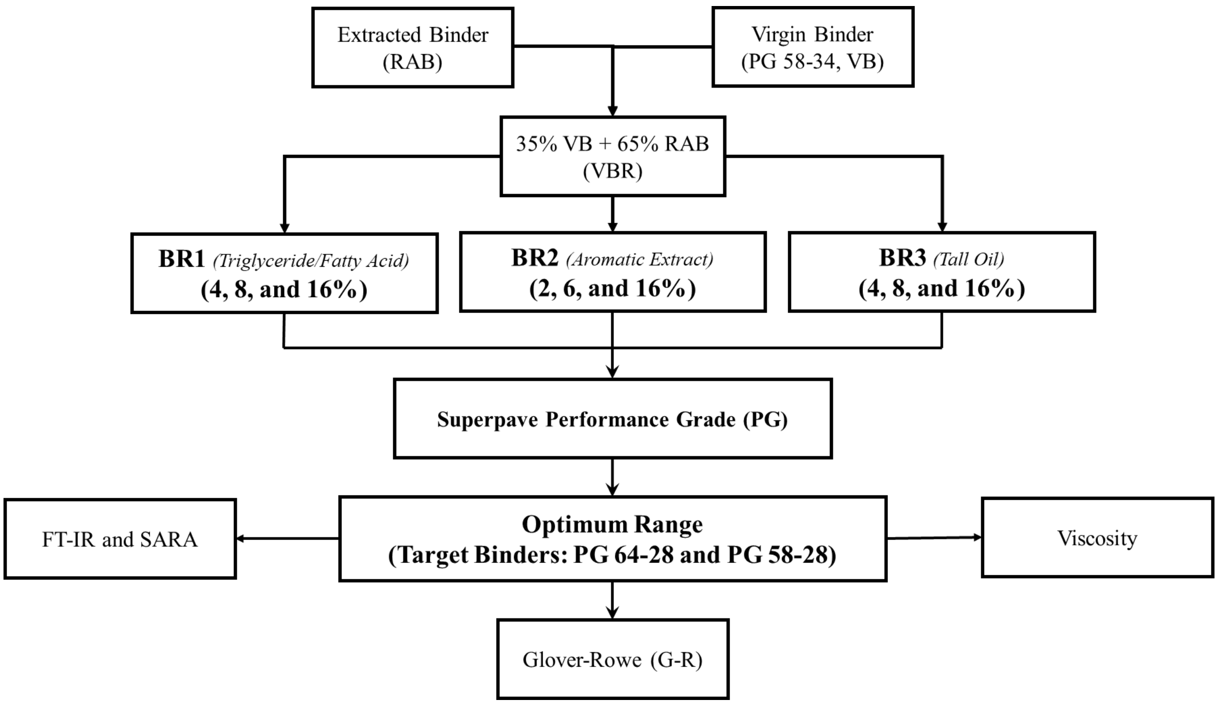


Figure 1-1. Testing plan designed asphalt binder.

Then each rejuvenator with its optimum dosage was applied to mixture level testing for further investigation as elucidated in Figure 1-2. It should be noted that the R3 is further incorporated with an anti-stripping agent as R3 used in the Phase I of this study showed the highest detrimental effect on moisture resistance of the high-RAP mixture than the other two rejuvenators (R1 and R2). Details of each phase of this study are described in the following chapters.

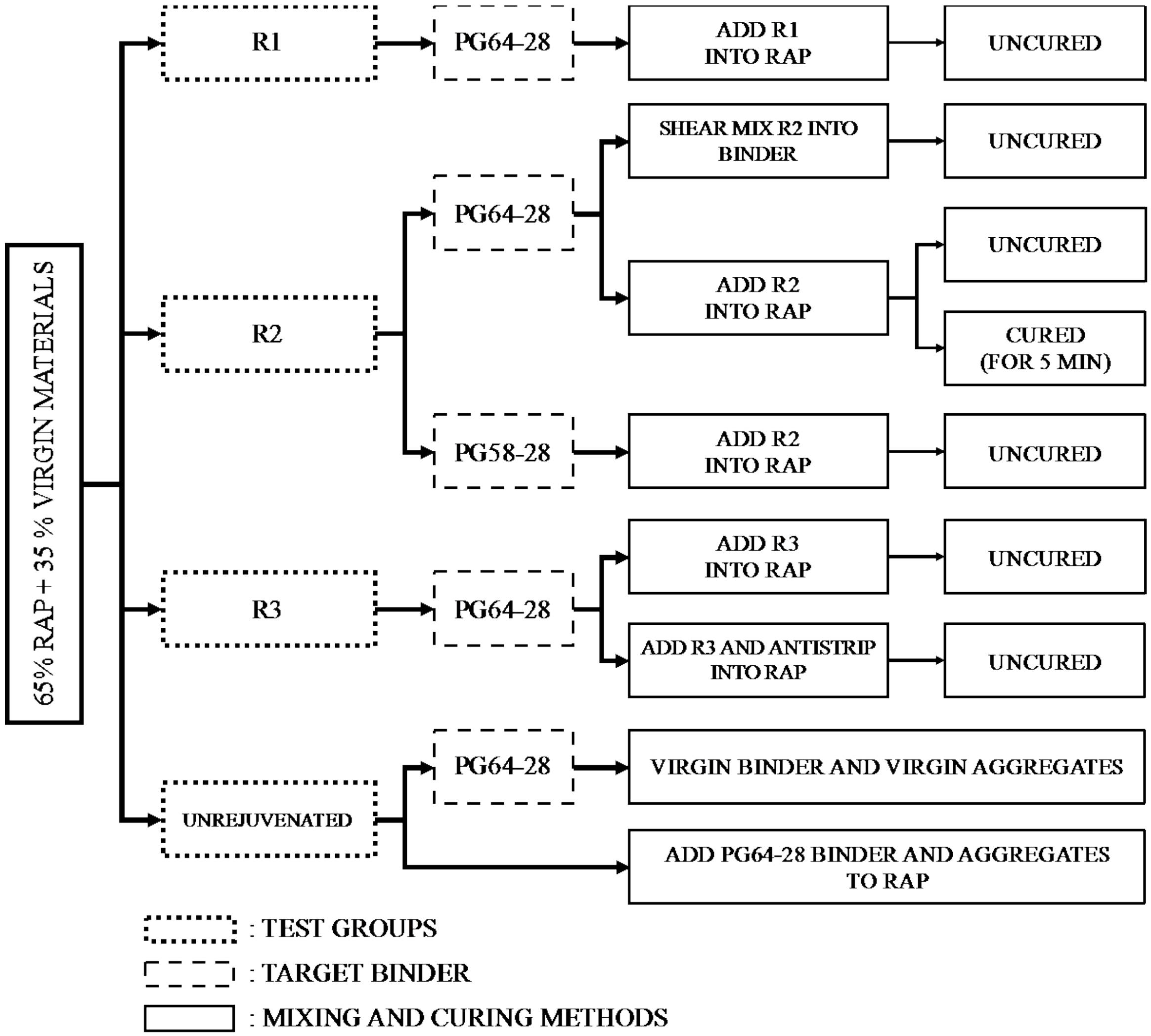


Figure 1-2. Testing plan designed asphalt mixture.

1.3 Organization of the Report

This report consists of five chapters. Following this introduction, Chapter 2 summarizes the literature review on the rejuvenators and performance of mixtures and pavements due to the addition of rejuvenators. In Chapter 3, the material selection and sample fabrication procedures to conduct various laboratory tests are described. Chapter 4 introduces the laboratory tests performed to examine mechanical and chemical characteristics of AC and binders that are mixed with and without aged materials and rejuvenators. Test results and analyses of test data are also presented and discussed in Chapter 4. Finally, Chapter 5 summarizes the main findings and conclusions of this study.

Chapter 2 Background

The use of recycled materials including RAP, reclaimed asphalt shingles (RAS), glass, and ground tire rubber is definitely a great achievement in the asphalt paving industry. Although recycling of asphalt pavements started in 1915 [2], increased interest on the use of recycled material occurred during the 1970s due to the rise in asphalt binder prices (Arab oil embargo) as well as the improvement in oil exploration tools and devices. The main consequence of these attempts is that RAP materials is now regarded as routine materials for pavement construction and rehabilitation [3].

The main obstacle in using recycled materials (e.g., RAP or RAS) is the drop of fatigue and low-temperature cracking resistance of the mixtures compared to the mixtures with virgin binder [4-7]. The cracking resistance has a direct relationship with the durability of the asphalt mixtures. This is because of the presence of aged asphalt binder in RAP/RAS which leads to an increase in the stiffness of the mixtures [8, 9].

In order to cope with the main drawback in using RAP materials in asphalt mixtures, the drop in the cracking resistance, durability of the asphalt mixtures has usually been resolved by five strategies [10]:

- Minimizing the RAP used in mixtures (or binder replacement amount),
- Improving the design density (lowering design air voids) or reducing N_{design},
- Addition of soft virgin binders, especially for the low-temperature grade,
- Employing rejuvenators,
- Mixing RAP materials with warm-mix additives.

Among aforementioned strategies, using the rejuvenator has become more conventional, since it can improve some of the mechanical properties of the asphalt mixtures with high RAP content. The rejuvenator term comes from this concept that the stiffness of aged materials can be returned (rejuvenated) to the original stage by adding these modifiers. Although the softening mechanism between additives is different, each additive is aimed to reduce the stiffness of the aged materials by modifying chemical/physical characteristics.

In order to reduce the stiffening of RAP binder induced by the aging phenomenon as well as the non-uniform chemical composition of RAP (compared to virgin asphalt binder), the RAP binder must be reconstituted using rejuvenators [11]. For the effective performance of rejuvenator, its uniform distribution within the mixture and its diffusion into the surface of the aggregates are essential. Table 2-1 lists the five conventional types of rejuvenators [12].

Table 2-1. Types of rejuvenators [12].

Category	Examples	Description
Paraffinic Oils	Waste Engine Oil (WEO) Waste Engine Oil Bottoms (WEOB) Valero VP 165® Storbit®	Refined used lubricating oils
Aromatic Extracts	Hydrolene® Reclamite® Cyclogen L® ValAro 130A®	Refined crude oil products with polar aromatic oil components
Naphthenic Oils	SonneWarmix RJ™ Ergon HyPrene®	Engineered hydrocarbons for asphalt modification
Triglycerides & Fatty Acids	Waste Vegetable Oil Waste Vegetable Grease Brown Grease Oleic Acid	Derived from vegetable oils, Has other key chemical elements in addition to triglycerides and fatty acids
Tall Oils	Sylvaroad™ RP1000 Hydrogreen®	Paper industry byproducts, Same chemical family as liquid anti-stripping agents and emulsifiers

2.1 Performance of Asphalt Mixtures Containing Rejuvenators

In the following sections, some significant findings from many relevant studies that examined the effect of rejuvenators on different properties of asphaltic materials are summarized.

2.1.1 Stiffness

As mentioned earlier, one major concern about RAP blended asphalt mixtures and aged asphalt binders is higher stiffness of these mixtures compared to the virgin ones which may result in lower cracking resistance. It was reported that the rejuvenator forms a very low viscosity layer surrounding the asphalt-coated aggregate, and then the rejuvenator starts penetrating into the aged asphalt binder layer and thus softens and rejuvenates the aged asphalt binder. Many research groups studied the effect of different types of rejuvenators such as vegetable oil waste and bio-rejuvenators on the stiffness of RAP-blended mixtures or aged asphalt binders and reported that the addition of rejuvenators result in a significant drop in the stiffness of field-aged asphalt binders and RAP-blended mixtures [13-16].

Im and Zhou [17] examined the effect of three rejuvenators (R1, R2, and R3) on stiffness of asphalt mixtures using dynamic modulus test. The R1 and R2 introduced to virgin binder at a rate of 0.6 and 1.5% of total asphalt binder while R3 directly introduced to the recycled materials at a rate of 2% by weight of the recycled materials. The results showed that for the lower temperature range (4 °C and 20 °C), the addition of the rejuvenator did not impact the stiffness compared to the control mixture. This effect was not rejuvenator type dependent. The stiffness of the recycled mixtures in high-temperature ranges (40 °C zone), or lower loading frequency levels dropped when the rejuvenators introduced to the control mixtures. The effect of rejuvenators on stiffness of aged binders has been evaluated by means of softening point, penetration and strain sweep. Ongel and Hugener [18] reported that the softening effect of rejuvenator with the lowest viscosity was more significant than that of the other rejuvenators. As a result, the lower amount of this rejuvenator (with the lowest viscosity) could reduce the stiffness of the aged asphalt binder more efficiently.

Recently, Kaseeret al. [16] studied the effect of two types of rejuvenators (tall oil and aromatic extract) at different dosages on stiffness of mixtures prepared using three different aggregates (limestone, sandstone and slag), five asphalt binders (Texas PG 70-22, Texas PG 64-22, and Texas PG 64-28, Indiana PG 64-22 and Indiana PG 58-28), and three types of recycled materials (reclaimed asphalt pavement, manufactured waste asphalt shingles, and tear-off asphalt shingles). They performed dynamic modulus test to characterize the viscoelastic stiffness of the mixtures. They concluded that rejuvenators significantly decreased the stiffness of asphalt mixtures blended with recycled materials and the recycled mixtures with an optimum rejuvenator dosage exhibited similar stiffness as that of the virgin mixture. More recently, Oldhamet al. [15] employed bending beam rheometer (BBR) to identify the effect of bio-rejuvenator on stiffness of aged and rejuvenated asphalt binders. In this study, a dosage of bio-rejuvenator ranging from 5 to 30% was added to the laboratory and field aged asphalt binders. The rheological test results indicated that the addition of bio-rejuvenator (5 to 30%) could decrease the stiffness of aged materials.

2.1.2 Moisture Susceptibility

The damage caused by moisture is one the most important causes of asphalt pavement failure [19-23]. Although the rejuvenators can improve some of the mechanical properties of asphalt mixtures containing recycled materials [24-32], the effect of rejuvenators on the moisture susceptibility of these mixtures is still unclear. For example, it was reported that the moisture susceptibility of an asphalt mixture improved after rejuvenators were introduced to the asphalt mixture containing RAP and RAS [33]. Hossain, Karakas [34] simulated the aging that happens during construction and in-service. A PG 64-22 binder was selected and aged in the lab. In addition, two types of aggregate (i.e., limestone and granite) were used and two different rejuvenators were introduced to the asphalt binders. The contact angle test was used to investigate the moisture resistance of the mixtures. The results showed that the rejuvenators could enhance the cohesive energy of asphalt binder. In other words, the rejuvenators can reduce the moisture susceptibility of asphalt mixtures.

In contrast, some researchers claimed that mixtures containing RAP with a soft binder or a rejuvenator had the same level of moisture damage resistance as the virgin mixture [35] or better than virgin mixtures after freeze-thaw cycles [36]. In addition, Nazzal, Mogawer [37] evaluated the moisture resistance of four different mixtures with 50% RAP, three different rejuvenators, virgin aggregate and asphalt binder using Hamburg wheel tracking and showed that addition of rejuvenators led to a decrease in moisture resistance and durability of RAP mixtures. Such controversial findings to the effects of rejuvenators on moisture susceptibility were also reported in studies by Tranet al. [26] and Haghshenas, Kim [38]. Based on the literature review it can be inferred that the moisture resistance of rejuvenated RAP mixtures is dependent on the type and origin of rejuvenators added to the mixtures [17, 39, 40].

2.1.3 Rutting

There are many studies regarding the effect of different rejuvenators on rutting resistance of RAP-blended asphalt mixtures [13, 14, 27, 33, 41, 42]. Due to the fact that the rejuvenators decrease the stiffness of recycled materials (e.g., RAP), a significant drop in the rutting resistance of the mixture has been reported by introducing the rejuvenator into the mixture [41]. Jia, Huang [43] prepared three different mixtures including 0, 25, and 40% RAP. The RAP blended mixtures treated using waste oil at different dosages (0, 2, and 5%). The rutting potential of mixtures were analyzed using asphalt pavement analyzer (APA). The results showed that the addition of waste oil decreases the rutting resistance of mixtures containing RAP materials. Im, Karki [44] designed three mixtures including: (1) mixture with PG 70-22 (without RAP), (2) mixture with PG 64-22 binder and 30% RAP. The Evotherm® P15 in 0.5% was used in both mixtures. Three different rejuvenators were introduced in various dosages (0% as a control, 2%, 5% and 10%). Using Hamburg wheel tracking they showed that the rut depth of all modified asphalt mixtures increased. Recently, Arámbula-Mercado, Kaseer [45] examined the effect of tall oil rejuvenator on rutting potential of mixture prepared using two types of aggregate including dolomitic limestone and sandstone with a binder PG 64-22. The loose mixture was subjected to a short-term oven aging protocol for 2 hours at 135˚C before compaction. They concluded that the rejuvenated mixtures with 12.5% tall oil could not pass the rutting criterion.

On the other hand, some researchers presented different findings that showed improved rutting resistance with the addition of rejuvenators into the mixture [25-27]. Im and Zhou [17] and Zaumaniset al. [39] have presented that the mixtures associated with recycled materials have higher rutting resistance than that of the virgin mixtures if they are modified with some rejuvenators. Zaumaniset al. [39] extracted binder from RAP (PG 94-12) and used six different rejuvenators (two petroleum products and four organic products with dosages from 6 to 18% from binder mass) to target asphalt binder of PG 64-22. They reported that the rejuvenators could not reduce the high-performance grade of asphalt binder to the level of virgin one. This indicated that the rejuvenators did not affect the rutting resistance of aged materials.

2.1.4 Cracking

There is a general agreement among researchers regarding the positive effect of rejuvenators on resistance to cracking (i.e., fatigue cracking, reflective cracking, and thermal cracking) of asphalt mixtures associated with aged materials [13, 33, 46-50]. For example, Mogaweret al. [33] and Jiet al. [13] investigated the effect of different rejuvenators on the mechanical/rheological properties of asphalt binders/mixtures incorporated with RAP and/or RAS. They reported that the addition of rejuvenators improved the cracking resistance of all mixtures [13, 33]; however, the degree of improvement was dependent on the type of rejuvenators [33]. According to the results obtained by Zaumaniset al. [39], the fatigue life and lower critical cracking temperature of the 100% recycled samples can be higher than those of the virgin mixtures if the appropriate rejuvenators are used. In addition, Tranet al. [14] designed three different mixtures including virgin, 50% RAP, and 50% RAP treated by rejuvenator. Rejuvenator was added in rate of 6.8% by weight of the RAP binder and different aging conditions (short and long-term) was applied. Overlay tester for intermediate-temperature and TSRST critical low-temperature were used to evaluate cracking resistance of the mixtures. They found out that the 50% RAP mix with rejuvenator show much better intermediate-temperature cracking resistance than the 50% RAP mix without rejuvenator, though not to the level of the virgin mix by the average values. The low critical temperature of 50% RAP mix with rejuvenator was similar to the virgin mix.

2.2 Optimum Rejuvenation Practices

Some researchers tried to investigate how mixtures/materials perform due to different treatments and dosages of rejuvenators so that an optimum rejuvenation practice can be achieved. For instance, Tranet al. [26] employed the Superpave performance grade (PG) to find the amount of rejuvenator required to recover the performance properties of the recycled binders. Zaumaniset al. [39] evaluated the effect of different dosages of six rejuvenators on rheological and physical properties of extracted binder from RAP. They reported that the high and low PG temperatures have linear correlation with dosage of rejuvenators while the penetration changes exponentially. They concluded that the Superpave PG requirements can be pursued for optimization purpose of rejuvenators. More recently, Arámbula-Mercadoet al. [45] evaluated three different methods including (1) rejuvenating low temperature PG and verifying high temperature PG, (2) achieving $\Delta T_c = -5C$ after 20–hour pressure aging vessel (PAV) aging, and (3) rejuvenating high temperature PG to explore the optimum dosage of rejuvenators. They claimed that the method to recover high temperature PG is a reliable approach to determine the optimum dosage of rejuvenators. Summary of methods which have been used by researchers to find the optimum rejuvenator dosage are listed in Table 2-2.

Table 2-2. Studies to examine optimum rejuvenation practice.

Study	Experimental Plan	Method(s)
Tranet al. [26]	- Extracted binder from RAP and RAS were blended in different ratio. - Different dosages of rejuvenator were used to target asphalt binder (PG 67-22).	Superpave performance grade
Zaumaniset al. [39]	- Extracted binder from RAP (PG 94-12) and six different rejuvenators (two petroleum products and four organic products with dosages from 6 to 18% from binder mass) were used to target asphalt binder (PG 64-22).	Superpave performance grade and Penetration
Imet al. [44]	- Three different rejuvenators were introduced in various dosages (0% as a control, 2%, 5% and 10%).	Superpave performance grade and Glover-Rowe
Arámbula-Mercadoet al. [45]	- Extracted binder from RAP and RAS from different sources were blended in different ratio. - Different rejuvenators including: Tall Oil, Aromatic Extract, Vegetable Oil, and Bio-Based Oil were added in different dosages. - At least three recycled blends were prepared: (1) blend with no recycling agent, (2) blend treated with a low recycling agent dosage, and (3) blend treated with a high recycling agent dosage. - Dosages varied based on type of rejuvenators used.	Superpave performance grade, ΔT_c = -5C, and Glover-Rowe
Ameri, Mansourkhaki [51]	- Two virgin binders (60/70 and 85/100) were selected. - The aged binder was extracted from RAP materials. - Six different combinations including combinations of RAB and softer binder (85/100), combinations of rejuvenated RAB and control binder (60/70) were studied. - Different dosages of petroleum-derived were used to achieve the same penetration grade as penetration grade 60/70.	Penetration

Chapter 3 Materials and Sample Fabrication

In this chapter, the materials used, and the sample fabrication procedure are illustrated.

3.1 Materials

3.1.1 Rejuvenators and Anti-Stripping Additive

Three different rejuvenators, obtained from petroleum, green, and agriculture technologies, and an anti-stripping additive were employed in this study. In Table 3-1, the information of the rejuvenators and anti-stripping additive are listed.

Table 3-1. Rejuvenators and anti-stripping additive.

Additives	Type	ID
Triglyceride/Fatty Acid	Agriculture Technology	R1
Aromatic Extract	Petroleum Technology	R2
Tall Oil	Green Technology	R3
Anti-Stripping	Liquid	AS

3.1.2 Aggregates

Aggregates used in here were collected from conveyor belt of asphalt plant before mixing. The aggregates in Table 5 were transported to laboratory at University of Nebraska-Lincoln (UNL) in sampling sacks as shown in Figure 3-1(a). In addition, RAP mixtures sacks shown in Figure 3-1(b) were also collected from the mixing plant. By collecting the materials directly from the plant, the cumbersome sieving process could be avoided. However, before using the collected materials, consistency between sacks was checked by randomly picking two sacks from Figure 3-1(a) and conducting sieve analysis. The results of this process are shown in Figure 3-2. In the figure, aggregated from the two random sacks (i.e., Sack 1 and Sack 2) were divided per AASHTO T 248. The divided samples sieved (Figure 3-2(a) and (b) for Sack 1 and Sack 2, respectively) were then combined into cumulative gradations from both sacks. The two cumulative gradations are compared in Figure 3-2(c).

(a) (b)

Figure 3-1. Aggregates used: (a) virgin aggregates, (b) RAP.

Table 3-2. Original aggregate gradation of SPR mixture.

AGGREGATES	%	PIT LOCATION 1/4 SEC T R	19 3/4	12.5 1/2	9.5 3/8	4.75 4	2.36 8	1.18 16	0.6 30	0.3 50	0.075 200
RC-1 LIMESTONE	10	KERFORD	100	53.6	22.1	5.5	3.5	3	2.7	2.5	1.5
MAN SAND	15	MARTIN MARIETTA	100	100	100	92.6	57.5	26	12	5.2	2.2
2A GRAVEL	10	NE 23 16N 1E	100	97.1	91.9	64.1	14	2	0.9	0.3	0.1
SCREENING	5	KERFORD	100	100	100	94.4	75.6	51.6	40.7	32.2	17.8
1/4" LIMESTONE	15	KERFORD	100	100	99.7	73.1	38.5	9.3	7.1	6.4	5.4
RAP	45	CONTRACTOR SUPPLIED	98.2	93.1	88.7	74.2	60	38.3	27.3	19.5	6.8
		COMBINED GRADATION	99.2	92.0	86.3	69.9	46.9	25.6	17.5	12.4	5.3
		SPECIFICATION	98		81		46			12	4
		RANGE	100		89		56			21	9

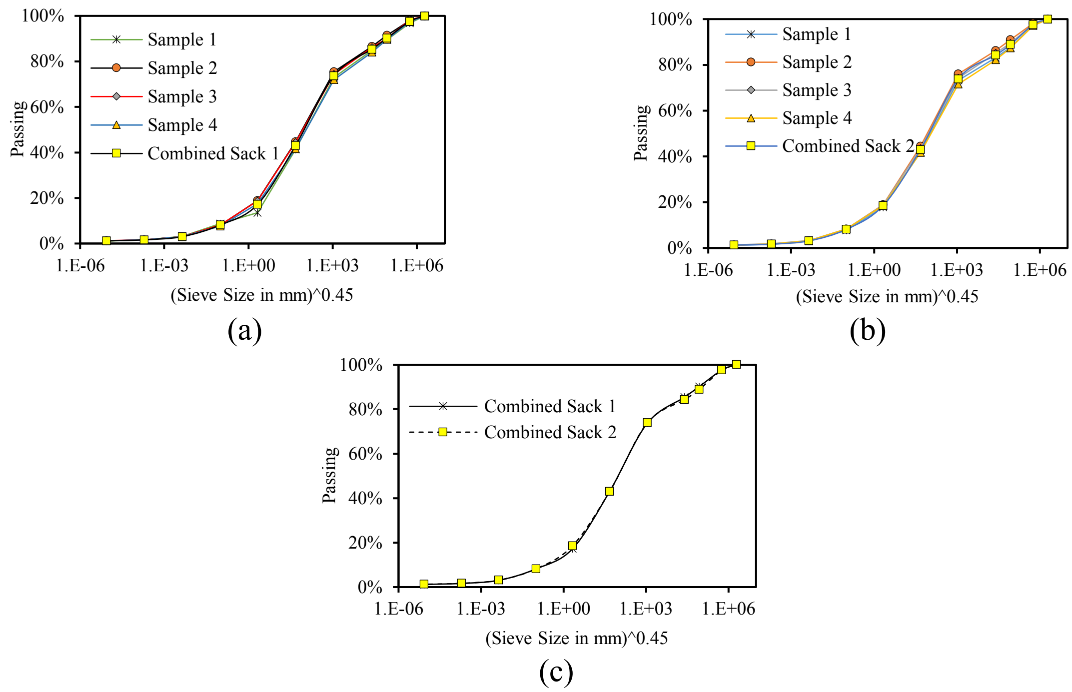


Figure 3-2. Checking consistency of virgin aggregates collected from plant: (a) sack 1, (b) sack 2 and (c) combined gradations of the sacks.

The target gradation of all mixtures prepared in here was derived from the SPR mixture which is a typical mixture used in Nebraska on medium trafficked road sections. As it can be seen in Table 5, in addition to virgin aggregates from different sources, gradation of SPR originally contained 45% of RAP. However, since in this study 65% RAP was used, the gradation was adjusted by simply reducing the proportion of virgin aggregates added to the total blend. It should be noted that the RAP materials (Figure 3-1(b)) was also obtained from plant then divided using AASHTO T 248 [52] into multiple sacks.

3.1.3 Binder

For the characterization of the old asphalt in the RAP, the old (aged) asphalt is separated from the RAP aggregates using a solvent, and then it was retrieved from the solvent. In the extraction step as shown in Figure 3-3, the RAP mixture (about 650 to 2,500 grams) was kept in contact with toluene (as the solvent), and this two-phase system was stirred for a sufficient time. After the extraction step, the mixed sample (RAP mixture and toluene) was separated using a centrifuge. The centrifuge washing was repeated at least three times until the color of extracted material became the color of straw. To remove the fine particles from the extracted binder, micro-centrifugation was conducted as shown in Figure 3-4.

Figure 3-3. Extraction apparatus.

Figure 3-4. Micro-centrifugation of the solution after extraction.

In order to retrieve the asphalt binder from the solution, a rotary evaporator, equipped with distillation flask, was employed (Figure 3-5). First, the oil bath was heated up to 150 $\pm$ 5 °C, and cooling water was circulated through the condenser. While the flask was above the oil bath, 200 to 300 mL of asphalt-toluene solution was drawn into the distillation flask by applying a vacuum of 72.0 $\pm$ 0.7 kPa. Then the flask was rotated at 40 rpm and lowered into the oil (approximately 40 mm into the oil bath). The vacuum was gradually decreased to 45.3 $\pm$ 0.7 kPa without allowing the solution to backflow into the condenser while applying vacuum. The 200 to 300 mL of asphalt-toluene solution was maintained in the flask until all the solution has entered the flask. The solution was gradually fed into the distillation flask. After distillation of the solvent, the vacuum was slowly increased to 6.7 $\pm$ 0.7 kPa. Carbon dioxide gas was then purged, and maximum vacuum was maintained for 45 $\pm$ 2 min. The flask was inverted and then placed into the oven at 165 $\pm$ 1°C to allow the asphalt binder to drain into a sample cup.

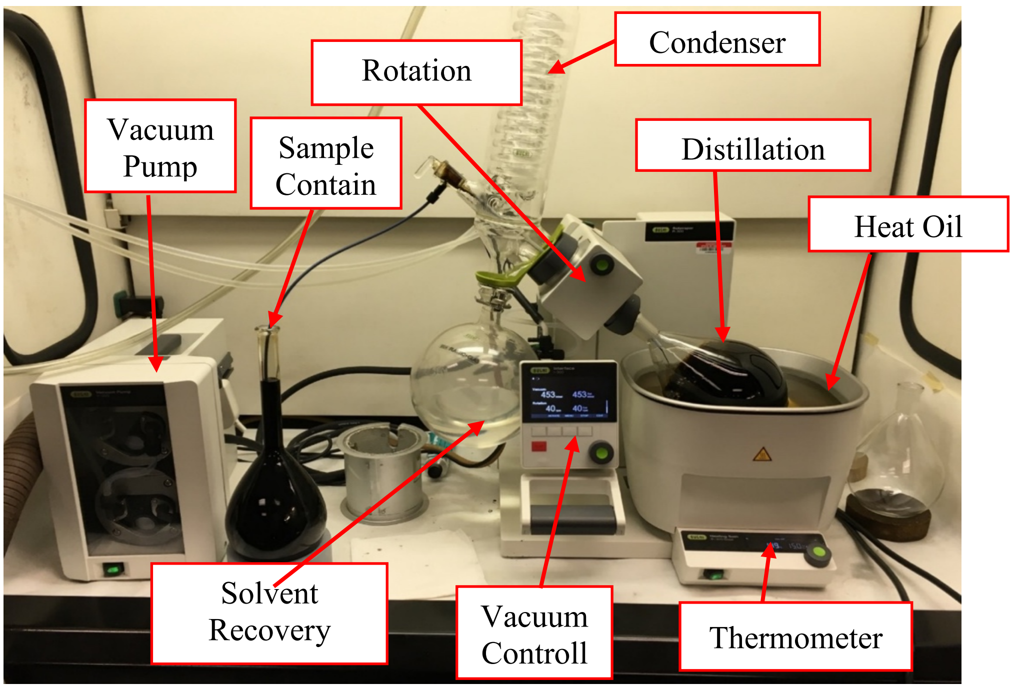


Figure 3-5. Rotary evaporator.

In addition to the recycle asphalt binder (RAB), a Superpave performance graded binder (PG 58-34) was used as a control binder (VB). As mentioned in Section 3.1.1, three different rejuvenators (R1, R2, and R3), based on different production technologies (i.e., petroleum, green, and agriculture) were also involved in three different dosages. For binder level study, as previously mentioned and shown in Figure 1-2(a), the dosages were selected based on findings of Phase I of this research [1] and added to the RAB (before blending with hot virgin binder). It should be noted that the dosage of rejuvenators was based on total weight of binder. Table 3-3 summarizes the information of binders.

Table 3-3. Binder information used in this study.

Binder Description	Binder ID
Control Binder: Virgin Binder (PG 58-34)	VB
Extracted Binder from RAP	RAB
Virgin Binder (35%) + RAB (65%)	VBR
VBR + Rejuvenators No.1 (4, 8, 16%)*	BR1 (4, 8, 16)
VBR + Rejuvenators No. 2 (4, 8, 16%)*	BR2 (2, 6, 16)
VBR + Rejuvenators No. 3 (4, 8, 16%)*	BR3 (4, 8, 16)

*Based on total weight of binder

3.2 Asphalt Concrete (AC) Mixture

3.2.1 Experimental Design for AC Mixtures

AC testing was conducted to evaluate mixture-level performance of AC treated with rejuvenators. Several factors that can affect the performance of the treated mixtures were also investigated. These factors include curing time, mixing method and rejuvenator dosage. Curing time refers to time allowed after mixing rejuvenators to RAP before mixing with virgin materials. It should be noted that rejuvenators were added directly to RAP in all cases. In the mixture level study, two dosages, three mixing methods and two curing methods were investigated. It should be noted that, similar to binder case, all dosages are expressed as percentage of total binder in mixture.

Figure 3-6 shows the experimental design employed for AC mixture. Besides to observe the effect of rejuvenators, the test plan was also conceived to provide key insights on the effect of antistripping agent for R3 mixtures and the effect of dosage for R2 mixtures. It is noted that a common antistripping agent provided by a local firm was used on R3 mixtures. The first control (CONTROL-RAP) represents a mixture in which no rejuvenator was applied while the second control (CONTROL -VIRGIN) is a mixture with only virgin materials (i.e., virgin aggregates and binder).

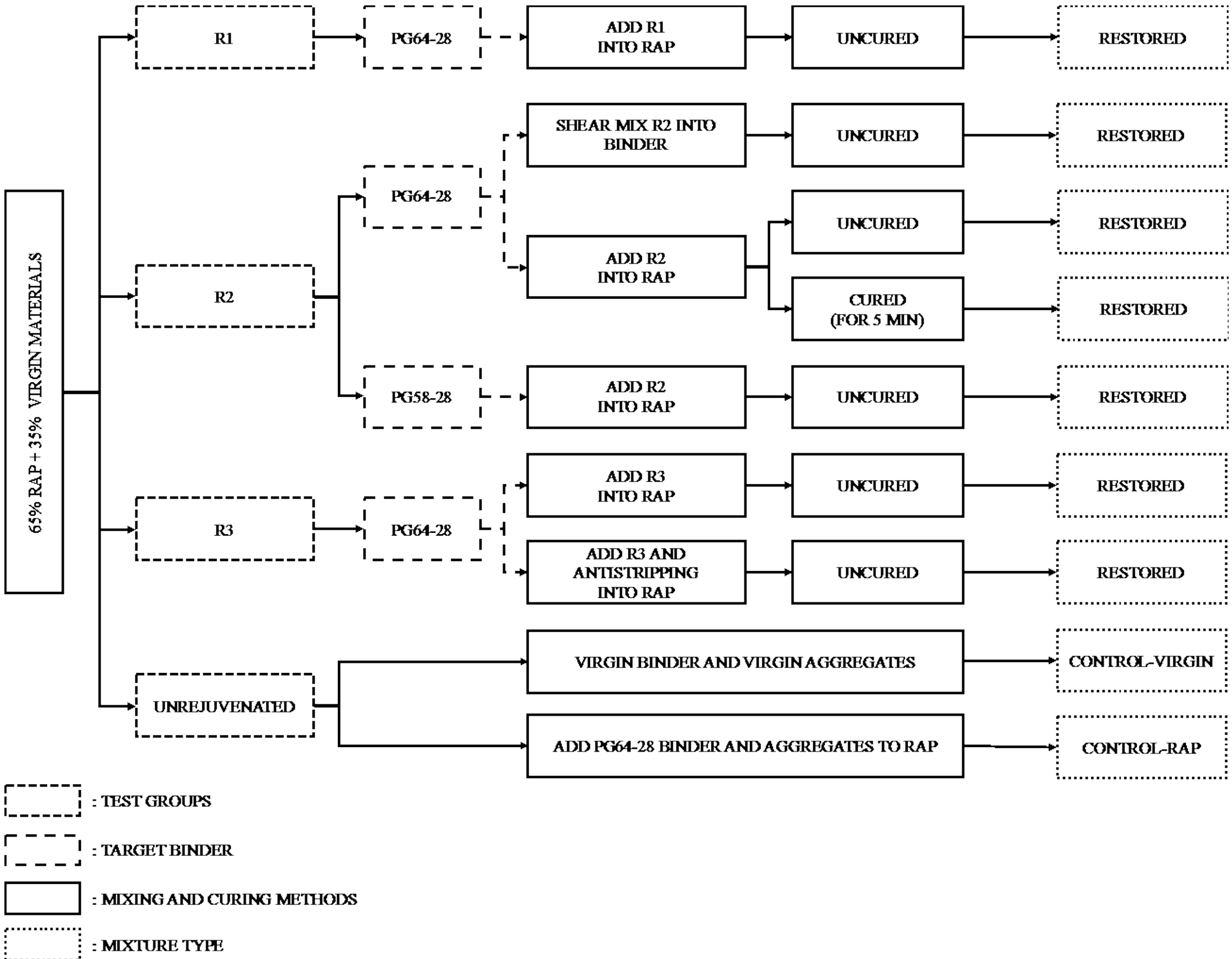


Figure 3-6. Experimental design used for AC mixtures.

3.2.2 Aggregate Gradation

Aggregate gradation for all mixtures were designed targeting SPR mixture in Nebraska. As 65% RAP was targeted instead of 45% RAP contained in typical SPR mixtures, aggregate blending proportions were adjusted by decreasing virgin aggregate content to 35%. Figure 3-7 shows the resulting mixture gradations compared to design gradation of SPR. It can be seen that the virgin (Figure 3-7(a)) and RAP (Figure 3-7(b)) gradations in laboratory were similar to those in SPR mixtures which resulted in a combined gradation of 65% RAP and 35 % virgin materials, that met requirements of SPR mixture (Figure 3-7(c)).

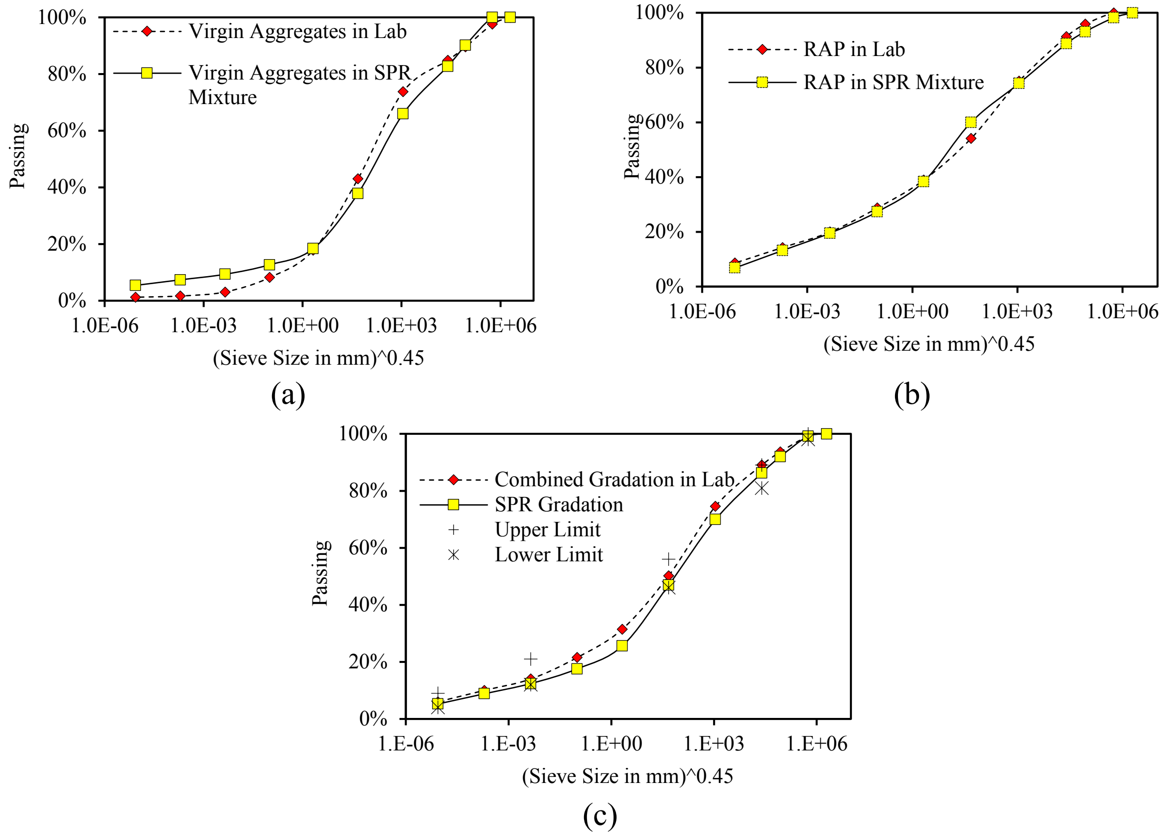


Figure 3-7. Aggregate gradations of AC mixture: (a) virgin, (b) RAP, and (c) combined.

3.2.3 Mixing/Compaction of Asphalt Concrete

With the aggregate gradations determined, AC mixtures were prepared by adding virgin binder, rejuvenators and antistripping agent where applicable according to the experimental design in Figure 3-6. To determine the required amount of virgin binder (VB) content, Equation 3-1 which accounts for the effect of pre-existing binder in RAB, rejuvenator dosage and the total binder was used in this study. It should be noted that 0.7% of total binder (RAB + VB) was used for the antistripping agent in the R3 mixture.

$$VB(\%) = \left(\frac{100}{100+Rejuvinator\ Dosage(\%)}\right) \times Total\ Binder(\%) - RAB(\%) \qquad \text{Equation 3-1}$$

For AC mixing and compaction, materials were heated in an oven as shown in Table 3-4. Virgin aggregated were heated overnight at SPR compaction temperature (160 °C) for at least 12 hours to remove moisture. However, VB and RAP were heated for only two hours in the oven at the compaction temperature. Rejuvenators were not heated prior to mixing due to the very volatile nature. They were added to RAP right before mixing. After mixing, a short-time aging period was allowed at the compaction temperature of 138 °C.

Table 3-4. Mixing/Compaction temperatures and time.

Material	Time in the oven (hours)	Oven temperature (˚C)
Virgin Aggregate	12	160 ± 3
Virgin Binder/RAP	2	160 ± 3
Short-time Aging	2	138 ± 3
Compaction	-	138 ± 3

3.3 Specimen Fabrication

3.3.1 Asphalt Binder Specimens (Aging Process)

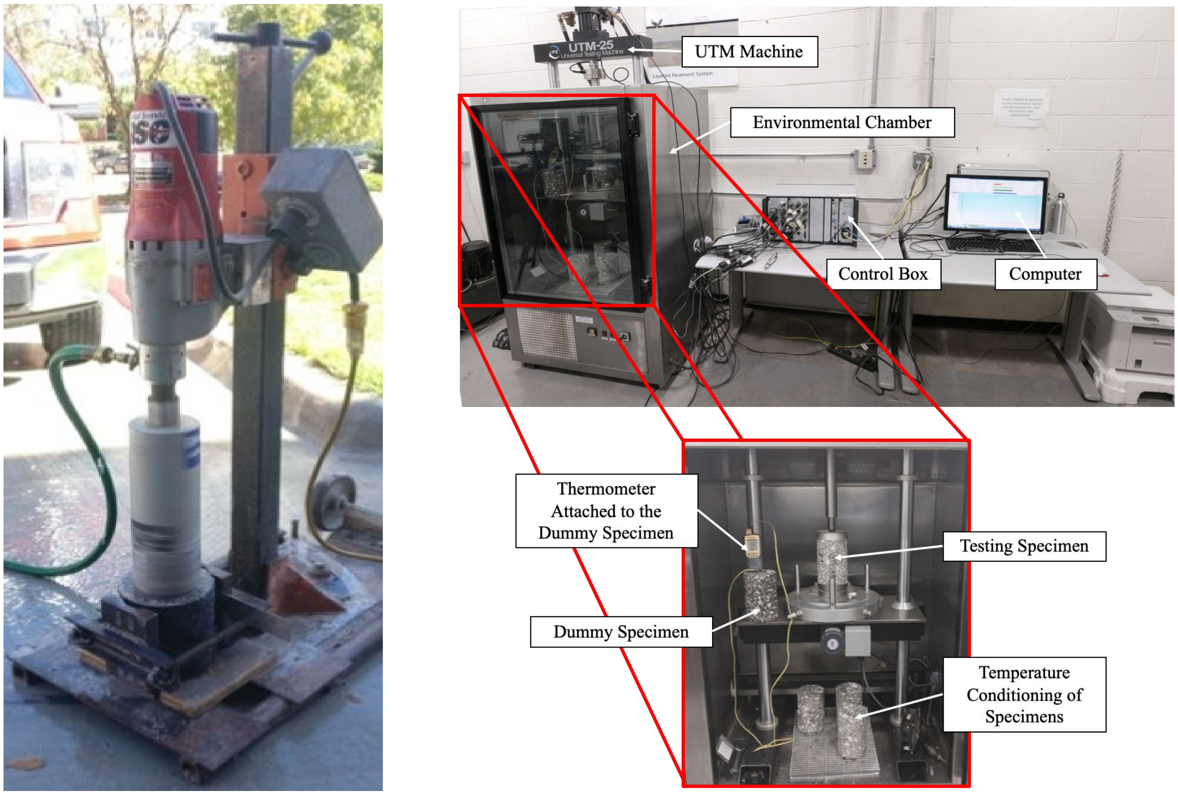
To prepare the aged asphalt binders, the Rolling Thin Film Oven (RTFO) and Pressure Aging Vessel (PAV) were employed. The RTFO [53] procedure was used to mimic the short-term aging process that occurs during blending, transporting, and paving an asphalt binder. In addition, the RTFO provides useful information about the amount volatiles lost during the aging process. To perform the RTFO test, the virgin asphalt binder was poured in cylindrical glass bottles and placed in a rotating carriage within an oven. The carriage rotates within the oven while the temperature is maintained at 163 °C for 85 minutes. The PAV test [54] was performed to simulate the aging that happens during the service life of asphalt pavement. In the PAV procedure, 50 grams of the RTFO aged binder was poured into a preheated 140 mm diameter pan. This amount of material in the PAV pan can provide a thin asphalt binder layer around 3.2 mm. The temperature of the aging vessel was maintained at either 90 ºC, 100 ºC, or 110 ºC and at a pressure of 2.1 MPa. The aging during a standard PAV was 20 hours; however, 4 PAVs (total 80 hours) were also attempted to conduct the Glover-Rowe test. More details about short-term and long-term aging processes are described in ASTM D2872 [53] and ASTM D6521 [54], respectively.

3.3.2 Asphalt Concrete (AC) Specimens

3.3.2.1 Flow number specimens

To prepare AC flow number specimens, the Superpave gyratory compactor (SGC) was used to fabricate 170 mm tall and 150 mm wide cylindrical samples. The tall samples were trimmed 10 mm on each end then cored using a drill bit (Figure 3-8(a)) to obtain 150 mm tall and 100 mm diameter cylindrical specimens. All specimens were compacted at target air voids of 4% ± 0.5.

The test set up shown in Figure 3-8(b) was used for testing. It comprises a load frame (UTM 25kN) and an environmental chamber for temperature conditioning of specimens. Flow number testing was conducted at 54.0 °C which required specimens to be temperature conditioned for at least 24 hours inside the environmental chamber. Temperature of specimens was crosschecked using a dummy specimen possessing a thermocouple in its core (Figure 3-8(b)). Loading was applied axially in a cyclic fashion at a deviatoric stress of 600 kPa and contact stress of 32 kPa. The testing stresses and temperature were determined following recommendation by Witczak, Pellinen [55]. It is noted that flow number test involves purse loading of the deviatoric stress for 0.1 second and a rest period (at the contact stress) for 0.9 seconds. The accumulated strain due to specimen deformation was recorded after each loading cycle.

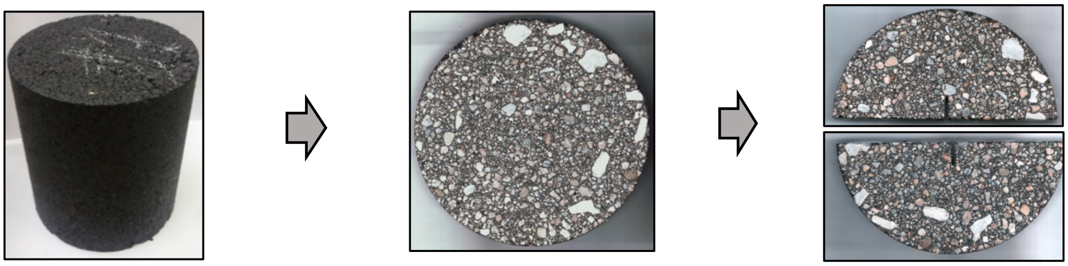


(a) (b)

Figure 3-8. Flow number testing: (a) sample fabrication (coring) and (b) test set-up.

3.3.2.2 Semi-circular bend fracture test specimens

Specimens for SCB test were compacted to target air voids of 7% using the SGC which produced 170 mm tall and 150 mm diameter cylindrical samples as shown in Figure 3-9(a). The tall samples were allowed to cool down to room temperature before slicing them into 50 mm thick disks (Figure 3-9(b)). The disks were then halved and notched in the middle (Figure 3-9(c)). The introduced notch was 15 mm tall and 2 mm wide and served to initiate crack since the main objective of SCB is crack propagation. From one SGC sample, a total of 6 SCB specimens were obtained.



(a) (b) (c)

Figure 3-9. SCB specimen fabrication process: (a) compacting, (b) slicing and (c) notching.

SCB specimens were tested at a room temperature using the set up shown in Figure 3-10. A monotonic load was applied from the top middle point (i.e., load-point) in a displacement-controlled mode at a rate of 3 mm/min. It should be noted that the SCB testing conditions (e.g., temperature, thickness, notch length) were adopted from previous studies performed by Nsengiyumva et al. [56, 57]. In addition to dry specimens, SCB testing was also conducted on moisture conditioned specimens, according to AASHTO T283 [58], to assess moisture susceptibility of AC mixtures.

Figure 3-10. SCB testing set-up.

Chapter 4 Laboratory Tests and Data Analysis

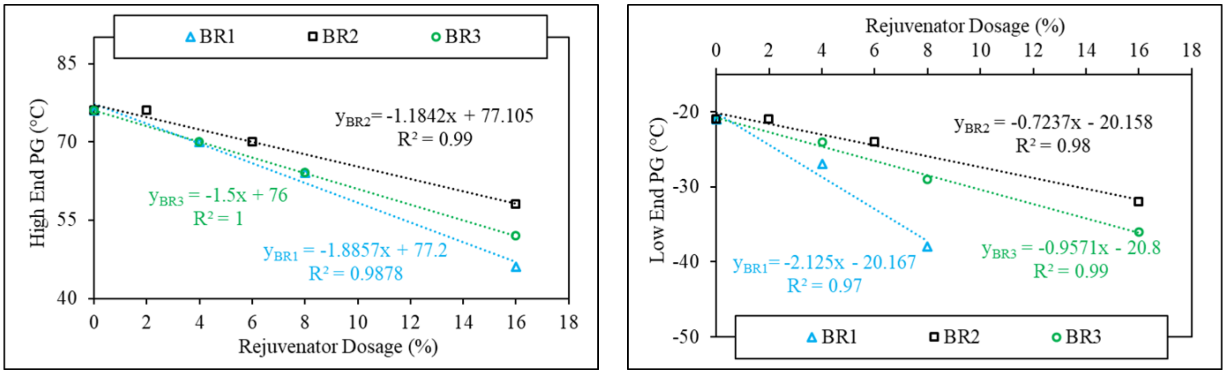
This chapter describes laboratory tests conducted in this study and test results. In the case of asphalt binders, five tests were performed using the viscometer, dynamic shear rheometer (DSR), bending beam rheometer (BBR), Fourier transform infrared (FT-IR) spectroscopy, and the saturates-aromatics-resins-asphaltenes (SARA) analysis. For AC mixtures, flow number test and SCB fracture test (dry and wet condition) were conducted.

4.1 Binder Tests and Results

4.1.1 Performance Grade (PG)

Dynamic shear rheometer tests were conducted on 25 mm diameter binder samples using the AR2000ex rheometer to obtain the temperatures at which the permanent deformation (rutting) parameter (i.e., $G^*/\sin\delta$) of original and short-term (RTFO) aged binders meet the performance grade (PG) criteria [59]. Bending beam rheometer (BBR) tests were also performed on binder samples aged through RTFO plus PAV procedures to determine the temperatures at which relaxation constant (*m*-value) and flexural creep stiffness (*S*) at 60 s of loading were equal to 0.3 and 300 kPa, respectively [60]. Then, the obtained high and low temperatures were used to determine the continuous performance grades of each binder blend [61, 62].

The high and low temperature performance grades of VBR (blend of extracted binder from RAP and virgin binder) and each rejuvenated asphalt binder are shown in Figure 4-1. The results clearly show that addition of rejuvenators decrease both high and low temperatures of VBR. This observation confirms that the rejuvenators soften the aged asphalt binder [16, 44, 63, 64]. Figure 4-1(a) demonstrates that the high temperature PG controls the maximum allowable dosage of rejuvenator, while the low temperature PG limits the minimum required dosage (Figure 4-1 (b)). In addition, the range of dosage for each rejuvenator can be determined using the data presented in Figure 4-1 by targeting the PG of VBR binder to the desired PG such as 64-28 or 58-28. Test results indicate that the VBR binder requires lower amount of R1 and R3 compared to R2 to meet the target PG. It should be noted that the BBR results of VBR binder treated by 16% of R1 is not shown in Figure 4-1 (b) because of some technical issues occurred during the BBR tests and lack of material to redo the test. Table 4-1 summarizes resulting minimum and maximum dosage range of each rejuvenator to meet the target PG: either 64-28 or 58-28.



(a) **(b)**

Figure 4-1. PG test results of rejuvenated binders: (a) high end, (b) low end.

4.1.2 Glover-Rowe (G-R)

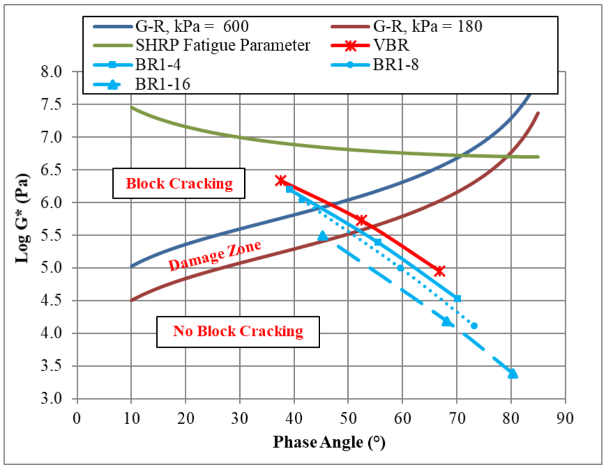
As discussed earlier, the aging has a direct relationship with the cracking resistance and durability of the asphalt mixtures. As a result, the aging characteristics of binders should rigorously be investigated. To this end, Glover Rowe (G-R) damage parameter tests were carried out using a dynamic shear rheometer with 8-mm diameter binder samples of original (unaged), RTFO + 1 PAV (20 hours), and (3) RTFO + 4 PAVs (80 hours). The complex modulus (G^*) and phase angle (δ) of each binder at 10 rad/s and 0.1% strain amplitude at 45 ˚C were recorded. Afterwards, using a black space diagram containing two G-R damage parameter curves: (1) $G^*(\cos^2 \delta/\sin \delta) = 180$ kPa and (2) $G^*(\cos^2 \delta/\sin \delta) = 600$ kPa, the measured G^* and δ values were plotted. Based on G-R parameter concept, if the G^*-δ value of a binder lands in the zone above the 600-kPa curve, it is assumed that the binder has severely been damaged. On the other hand, a binder with G^*-δ value below the 180-kPa curve is healthy and no crack is initiated in the binder [44].

Table 4-1. Selected rejuvenator dosage range.

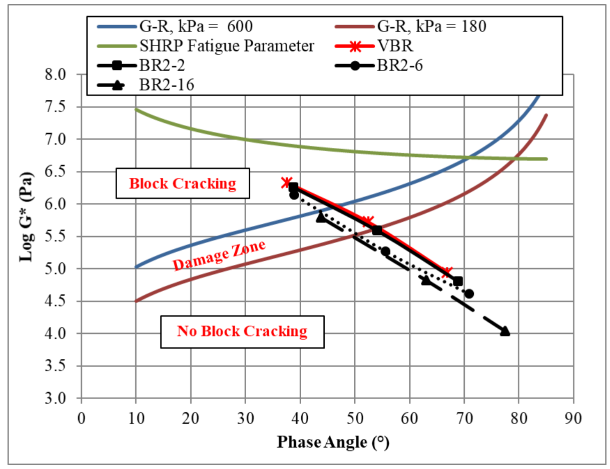
Rejuvenator	Minimum Dosage (%)	Maximum Dosage (%)
	Target PG: 64-28	
R1	3.7	7
R2	10.8	11.1
R3	7.5	8
	Target PG: 58-28	
R1	3.7	10.2
R2	10.8	16.1
R3	7.5	12

The results of G-R testes for VBR and rejuvenated VBR by adding different dosages of each rejuvenator at different aging conditions (i.e., unaged, RTFO + 1 PAV and RTFO + 4 PAVs) are presented in Figure 4-2 and Table 4-2. The results present that treated binders with higher dosages of rejuvenator (e.g., 16%) intersect G-R damage parameter curves (i.e., curve of 180-kPa and curve of 600-kPa) at lower modulus and phase angles compared to that of with lower dosage of rejuvenators (e.g., 4%). Furthermore, Figure 4-2 and Table 4-2 show that the rejuvenated binders cross the damage onset (initial) and severe cracking curves at longer aging time than the control blends. This implies that the cracking resistance of VBR binders improves when rejuvenators (i.e., R1, R2, and R3) are added. This improvement intensifies by increasing dosage of rejuvenators. For example, Figure 4-2(a) shows that VBR binder modified by 0% (control), 4%, 8% and 16% R1 reached damage onset after 16, 27.9, 47 and 74.6 hours of PAV aging (see Table 4-2), respectively. Similarly, they reached severe cracking state after 37.2, 54.0, 67.3 and 98.1 hours of PAV aging. The similar trend was observed from VBR binders treated by R2 and R3.

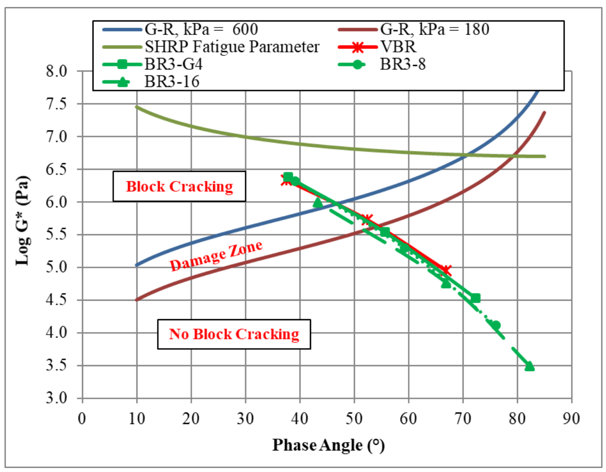
Table 4-2 shows the duration of aging (in hours) at which rejuvenated binders show onset of cracking and severe cracking. The results show that treated binders by R1 and R3 experience the severe cracking much faster than the binders treated by R2. In other words, the long-term performance (cracking resistance) of binders treated by R2 (aromatic extract) is expected to be better than that of modified by R1 (triglyceride/fatty acid) and R3 (tall oil).



(a)



(b)



(c)

Figure 4-2. G-R damage parameter: (a) R1, (b) R2, (c) R3.

Table 4-2. Measured aging time to induce onset (initial) and severe cracking.

Binder ID	Rejuvenator Dosage	Onset Cracking (hr)	Severe Cracking (hr)
		G-R, kPa =180	G-R, kPa =600
VBR	0	16.0	37.2
BR1-4	4	27.9	54.0
BR1-8	8	47.0	67.3
BR1-16	16	74.6	98.1
BR2-2	2	23.0	48.9
BR2-6	6	32.7	57.7
BR2-16	16	59.8	83.1
BR3-4	4	24.2	46.2
BR3-8	8	34.6	55.3
BR3-16	16	54.0	74.7

Table 4-3 summarizes the G-R parameters of rejuvenated binders at the two selected dosages (maximum and minimum found from the PG testing). The results reveal that in the optimum range of each rejuvenator, the estimated performance of rejuvenated binder with R2 is better than other rejuvenators, while addition of R2 and R3 to the binder accelerates the aging effect compared to R2.

Table 4-3. Estimated performance of rejuvenated binders based on G-R parameters at the optimum range of each rejuvenator.

Binder ID	Optimum Dosage	Onset Cracking (hr)	Severe Cracking (hr)
Minimum Dosage Based on Low End PG = -28			
BR1	3.7	29.0	51.7
BR2	10.8	45.8	69.7
BR3	7.5	33.4	54.5
Maximum Dosage Based on High End PG = 64			
BR1	7	41.4	64.2
BR2	11.1	46.6	70.5
BR3	8	34.6	55.7

4.1.3 Kinematic Viscosity

The kinematic viscosity characterizes flow behavior of the materials. The specifications are usually at temperatures of 60°C and 135°C. The time is measured for a fixed volume of the liquid to flow through the capillary of a calibrated glass capillary viscometer under an accurately reproducible head and at a closely controlled temperature. The kinematic viscosity is then calculated by multiplying the efflux time in seconds by the viscometer calibration factor [65].

Figure 4-3 shows the kinematic viscosity at 135 °C (typical compaction temperature of hot mix asphalt) of binders at varying dosages of each rejuvenator. The results show that there is an exponential relation between dosage and viscosity of asphalt binder regardless of type of rejuvenators. The performance of R1 and R3 is quite similar while the effect of R2 is a little different from others.

Table 4-4 summarizes the viscosity of rejuvenated binders at 135°C at the two selected dosages (maximum and minimum found from the PG testing).

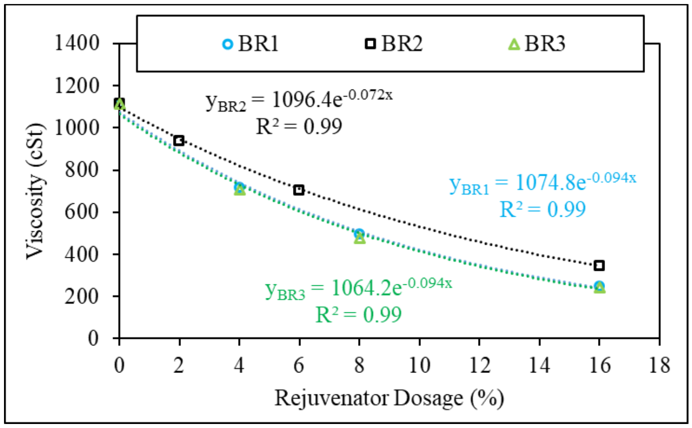


Figure 4-3. Viscosity of rejuvenated binders at 135 °C.

Table 4-4. Estimated viscosity of rejuvenated binders at 135 °C at the optimum range.

Binder ID	Viscosity (cSt) at minimum dosage	Viscosity (cSt) at maximum dosage
BR1	759	556
BR2	503	493
BR3	525	501

4.1.4 Fourier Transform Infrared (FT-IR) Spectroscopy

Fourier transform infrared (FT-IR) spectroscopy has been used by several researchers [64, 66-68] to investigate and quantify the chemical interactions that can occur within asphalt binder. The FT-IR spectrum of an altered binder due to chemical interactions resulting from the rejuvenation and aging processes will result in fluctuations in the absorbance spectrum of certain chemical groups. Two functional groups such as the carbonyl and sulfoxide have been used as potential indicators for understanding the oxidative aging process in asphalt binders. Relative increase in these functional groups can be used to quantify if a given binder is susceptible towards aging and consequently changes in their rheological properties. Hence, by using FT-IR test results, a better understanding of the interaction between the rejuvenator and binder can be established.

FT-IR spectroscopy analysis of the binders and rejuvenators used in the current study were performed using a Thermo Nicolet Avatar 380 FT-IR spectrometer. The FT-IR spectrum for each binder sample was captured using the attenuated total reflection (ATR) mode, for which a Smart Performer ATR accessory with a diamond crystal was used. Each spectrum for a given binder was obtained in the range of 500 to 4,000 cm^{-1} wave number at a resolution of 4 cm^{-1}. For the estimation of the areas under the peaks, the OMNIC 8.1 software was used. The background spectrum was subtracted from each sample spectrum. The diamond crystal was cleaned with ethanol after each sample analysis. Figure 4-4 exemplifies the FT-IR spectra of eleven binders without any laboratory aging condition.

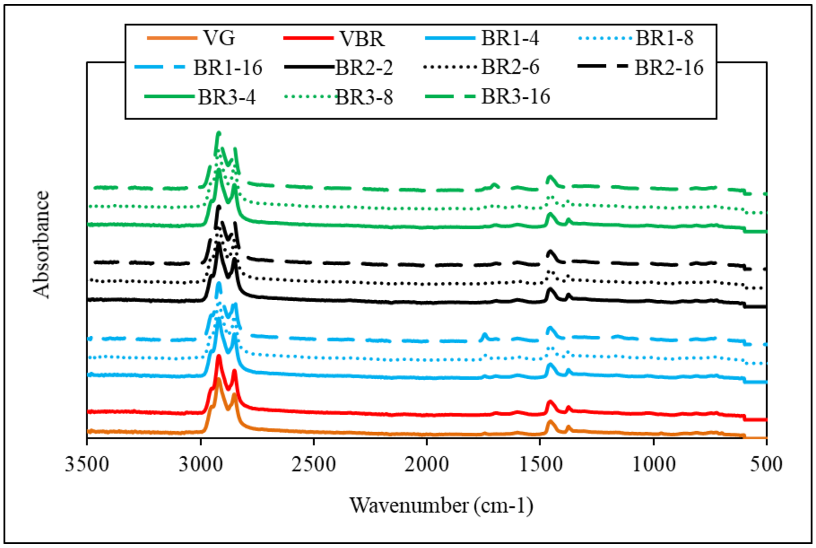


Figure 4-4. FT-IR spectra of all binders for unaged condition

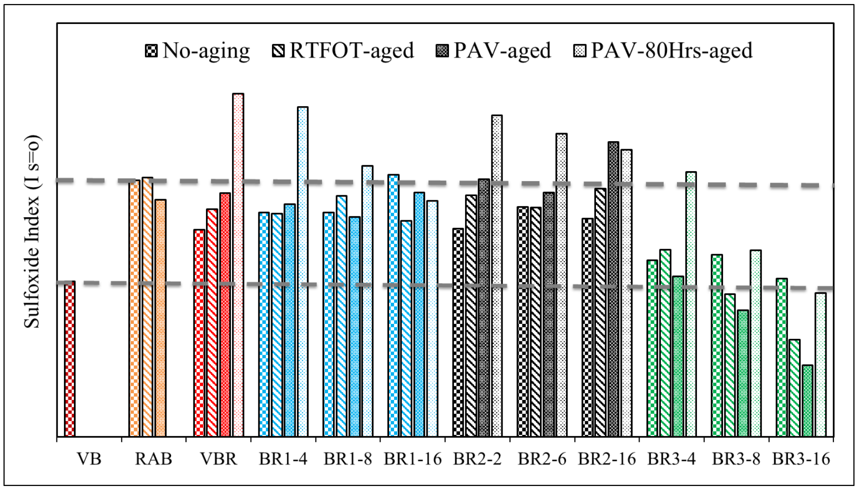
Two major functional groups (i.e., carbonyl (C=O) and sulfoxide (S=O)) are key to understand the influence of aging on the chemical properties of binder. Increase in these functional groups can result in strong intermolecular forces between the polar fractions within the binder there by resulting in increased viscosity of binder. The characteristic wavelength for the carbonyl and sulfoxide functional groups have been identified as 1,700 cm^{-1} and 1,030 cm^{-1} respectively. The area under each characteristic wave number (corresponding to functional groups) was then estimated for the determination of ratios of the area under a specific band to the overall area. These indices can be logically employed for comparative purposes. The calculation of structural indices adapted from Lamontagne et.al [66] and Feng et.al [68] can be described as follows:

Carbonyl Index ($I_{C=O}$): $A_{1700} / \Sigma A$

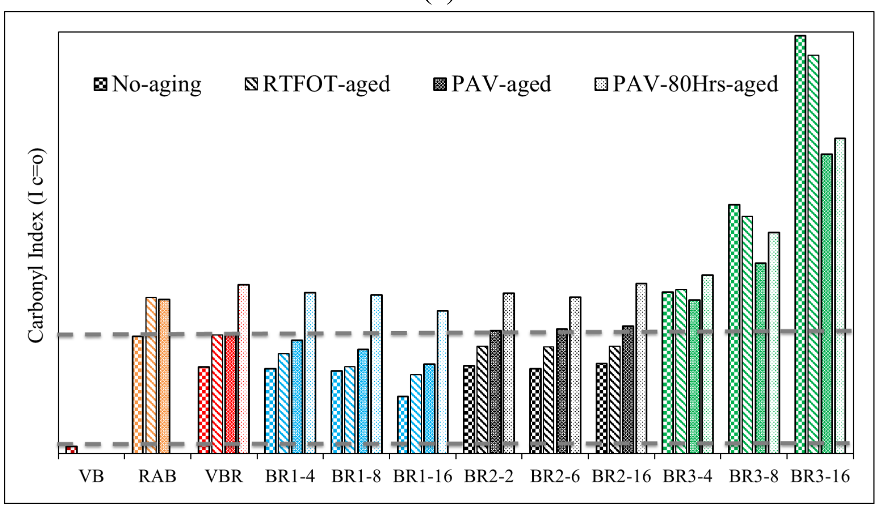
Sulfoxide Index ($I_{S=O}$): $A_{1030} / \Sigma A$

where $\Sigma A = A_{1700} + A_{1600} + A_{1460} + A_{1377} + A_{1030} + A_{956} + A_{866} + A_{814} + A_{723}$

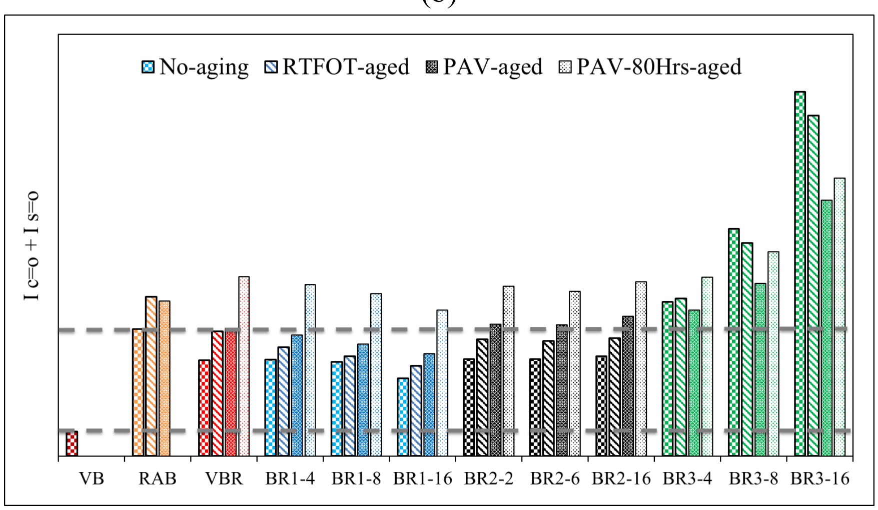
Figure 4-5 shows the carbonyl index, sulfoxide index and the sum of the two indices of each binder at different aging conditions such as no aging, laboratory short-term aging (RTFOT), and long-term aging (RTFOT + 1 PAV, RTFOT + 4 PAVs) conditions. It can be observed that increase in $I_{C=O}$ and $I_{S=O}$ indices as a clear consequence of aging when comparing the RAB with its control binder VB. More specifically, Figure 4-5(a) shows that there is no clear trend in the sulfoxide index due to the rejuvenators and aging conditions, while the carbonyl index of the binders dropped when rejuvenators R1 and R2 were added (Figure 4-5(b)). Furthermore, with increased dosage of R1 a decreasing trend of the carbonyl index was observed whereas there was no change for R2. Since both the carbonyl and sulfoxide contribute to the polar interactions, the sum of the two indices ($I_{C=O}$ + $I_{S=O}$) which is usually referred as the polar index was also used to examine the effect of rejuvenators. In Figure 4-5(c) it can be observed that R1 reduced the polar index substantially with increased dosage when compared to VBR with no rejuvenator. Moreover, the long-term performance of the R1 is within the desired range of polar index. Rejuvenator R2 did not change the polar index with an increased dosage and with different aging conditions compared to VBR. Figure 4-5(c) also indicates that rejuvenated binder with R3 is more susceptible to aging as it shows a substantial increase in polar index.



(a)



(b)



(c)

Figure 4-5. (a) sulfoxide index ($I_{S=O}$); (b) carbonyl index ($I_{C=O}$); and (c) $I_{C=O} + I_{S=O}$.

4.1.5 Saturate-Aromatic-Resin-Asphaltene (SARA) Analysis

In order to estimate the percentage of the asphalt binder component (i.e., SARA) in different asphalt binders, Iatroscan MK-6 was employed. The Iatroscan method is based on solubility and polarity. First, the asphaltenes are separated from the bulk asphalt as the materials are insoluble in n-heptane. It should be noted that normal heptane is non-polar and a linear saturated alkane and therefore the resin, aromatic structures are insoluble in this solvent. This is a separate test procedure and is not performed by the Iatroscan equipment. Once the n-heptane insoluble material is removed from the asphalt the remaining material (generally referred to as maltenes) are further separated based on their relative solubility in different solvents.

The Iatroscan uses a silica chromarod (chromatographic rod) assembly to separate the resins, aromatics and saturates. There are 10 small diameter rods set in a metal frame, and each rod is spotted with a small amount of the maltene solution onto the bottom of each rod and the solvent is allowed to evaporate from the rods leaving the maltenes on the bottom of the rods. The rods with the maltenes are set in a tank containing about 50 ml of normal pentane. The n-pentane elutes or carries the resins up the rod because the resins are soluble in the n-pentane. The rods are removed from the n-pentane tank, allowed to dry for 5 minutes, and then placed in a tank containing a 90%/10% blend of toluene/chloroform. The toluene chloroform solution elutes or carries the aromatics up the rod over a 7 minutes time period as the aromatics are soluble in the toluene. The saturate are left on the bottom of the rods after the other fractions have been eluted up the rods due to their solubility in the solvents described. The rods are removed from the tank, allowed to dry in a 90 °C oven for 15 minutes. The rods, which are in a holder, are placed in the Iatroscan and a small flame burns the chemicals off each rod simultaneously and the material burned is identified by what is known as a flame ionization detector (FID). The Iatroscan quantifies the amount of material burned off the rods in each section of the rod with the saturates being quantified first because they are on the bottom of the rods, the aromatics second because they are in the middle of the rods, and the resins last because they are the highest up on the rods.

Figure 4-6 presents the results of SARA tests for different binders in three different states (i.e., original, RTFO and PAV aged) and rejuvenators. As shown in the Figure 4-6(a), the SARA analysis of the VBR binder (the mixture of control binder VB and the recycle asphalt binder (RAB)) shows an increase in asphaltenes, resins, and saturates, and a decrease in aromatics compared to control binder (VB). As also shown in the Figure 4-6(a), addition of rejuvenators to VBR results in a decrease in asphaltene content, while changes in aromatics and resins are rejuvenator dependent. For instance, addition of R1 and R3 leads to an increase in amount of resins and a decrease in amount of aromatics whereas R2 changes those fractions (i.e., resins and aromatics) reversely. This trend escalates by increasing the dosage of rejuvenators. Figure 4-6(b) and (c) show the effect of short-term (RTFO) and long-term (PAV) aging on VBR and rejuvenated binders. As shown, the laboratory aging leads to an increase in asphaltenes and resins while the aromatics decreases.

With the SARA analysis results, the colloidal index (CI), which is defined as the ratio of "the sum of aromatics and resins" to "the sum of asphaltenes and saturates" [69], can be quantified as an indicator of aging and rejuvenation. The CI is generally used to identify the binder systems with deposit problems. In the aging process, a series of transformations occur due to the presence of oxygen; the aromatics to resins and then the resins to asphaltenes. During this process, the ratio of maltenes to asphaltenes reduces and consequently, lower amounts of maltenes are available for the dispersion of the asphaltenes [12]. This results in higher viscosity, lower ductility, and smaller value of CI. The CI values of VB, RAB, VBR, and rejuvenated binders are presented in Table 4-5. According to the results presented in the table, the blend of recycle asphalt binder (RAB) with control binder (VB), namely VBR, results in 8.2% decrease in CI value of VB. Due to the nature of the rejuvenators, where a high proportion of maltenes exist, the CI values generally increased in the presence of rejuvenators. This improvement in CI values intensifies by increasing dosage of rejuvenators. For instance, the CI of VBR is 3.27, and it increases to 3.39, 3.55, and 3.95 for BR1-4, BR1-8 and BR1-16, respectively. Same trend is observed in case of other rejuvenators. The CI results imply that, in general, aging decreases CI. However, the rate of changes in CI due to aging are somewhat rejuvenator-specific. Table 4-6 indicates that in the lower limit of selected range of rejuvenators, R2 shows a 3.2% and 18.8% reduction in CI during the short-term (RTFO) and long-term (PAV) laboratory aging process respectively, while the loss of CI is more pronounced when the R1 and R3 were used. Same trend is observed when the maximum dosage of selected range for each rejuvenator is added to the VBR. It can be inferred that the short-term and long-term performance of binders treated by R2 is better than other rejuvenators. SARA test results are generally in good agreements with G-R test results presented earlier.

Figure 4-6. Percentage of SARA components for each binder in different aging states.

Table 4-5. Values of colloidal index (CI) of original, short and long-term aged binders.

Binder ID	Rejuvenator Dosage	Original (Unaged)	Change relative of each binder to BC (%)	RTFO	Change relative of each RTFO binder to its original state (%)	PAV	Change relative of each PAV binder to its original state (%)
VB	0	3.57	-	-	-	-	-
RAB	0	3.08	-13.6	-	-	-	-
VBR	0	3.27	-8.2	2.96	-9.7	2.49	-23.8
BR1-4	4	3.39	-4.9	3.12	-8.1	2.45	-27.8
BR1-8	8	3.55	-0.6	3.09	-8.7	2.57	-24.1
BR1-16	16	3.95	10.8	3.59	5.8	3.02	-11.0
BR2-2	2	3.26	-2.3	2.78	-14.7	2.5	-23.3
BR2-6	6	3.42	4.9	3.03	-7.0	2.56	-21.4
BR2-16	16	3.75	13.2	3.33	2.3	2.74	-16.0
BR3-4	4	3.48	-8.7	3.08	-9.1	2.44	-28.1
BR3-8	8	3.74	-4.1	3.27	-3.4	2.44	-28.1
BR3-16	16	4.04	5.1	3.43	1.1	2.48	-26.8

Table 4-6. Estimated performance of rejuvenated binders based on CI reduction.

Binder ID	Optimum Dosage	Short-Term (RTFO)	Long-Term (PAV)
	Minimum Dosage Based on Low End PG = -28		
BR1	3.7	-10.7	-29.0
BR2	10.8	-3.2	-18.8
BR3	7.5	-5.3	-27.9
	Maximum Dosage Based on High End PG = 64		
BR1	7	-6.6	-24.3
BR2	11.1	-2.9	-18.6
BR3	8	-4.9	-27.9

4.1.6 Summary of Binder Tests

The results of Superpave performance grade (PG) tests showed that all rejuvenators soften (measured by G^* and δ, DSR tests) binders which increases the rutting susceptibility (measured by $G^*/\sin\delta$, DSR tests) and improves the low temperature cracking resistance (measured by $S(t)$ and m-value, BBR tests) of the blend of extracted binder from RAP and virgin binder. These trends intensified when the amount of rejuvenator increased. Furthermore, the results of PG tests showed that there is a specific dosage range for each rejuvenator (so called optimum/selected range) that can change the PG of resultant blend (i.e., VBR) into its target binder which is PG 64-28/PG 58-34. The optimum range of each rejuvenator was then selected for further evaluations using mechanical/rheological (i.e., viscosity, G-R) and chemical (i.e., FT-IR, SARA) tests and analyses. The results of viscosity tests at compaction temperature (135°C) showed that in the optimum range of each rejuvenator the viscosity of modified binders was fairly similar, except in case of R1 (triglyceride/fatty acid) in lower limit of selected dosage range which showed higher viscosity. In addition, the G-R, FT-IR, and SARA test results revealed that, at the optimum range of each rejuvenator, the short-term and long-term performance of binders treated by R2 (aromatic extract) was better than that of modified by other rejuvenators (i.e., R1 and R3). However, it is worthy of note that the short-term and long-term aging processes were simulated using laboratory procedures. The laboratory binder aging is limited to accurately examine the effects of rejuvenators on short-term and long-term field aging in pavements. Further studies that mimic the actual short and long-term field-aging process, such as the effects of ultraviolet light, moisture, and vehicular stresses, should be pursued.

4.2 Asphalt Concrete (AC) Tests and Results

From the mechanical/rheological and chemical tests of binders, the mixture-level testing program shown in Table 4-7 was adopted. The testing program was designed to investigate the effect of the rejuvenators selected, dosage, mixing and curing method on mixture performance. To target PG 64-28, dosages of 7%, 11% and 8% where selected for R1, R2 and R3, respectively. In addition, R2 was used to target PG 58-28 by using a dosage of 14%. It should be noted that for R3 mixtures, the selected dosage (i.e., 8%) was incorporated with an anti-stripping agent to check if the addition of anti-stripping agent can improve the moisture susceptibility of mixtures treated by R3. It should be noted that for every mixture (i.e., ID) prepared, three Superpave gyratory compacted (SGC) tall samples were compacted for the flow number and SCB tests.

Table 4-7 Final mixture-level testing program.

TEST GROUP	DOSAGE	MIXING AND CURING METHODS		MIXTURE ID
R1	7%	ADD R1 INTO RAP	UNCURED	R1-7-UNCURED
R2	11%	SHEAR MIX R2 INTO BINDER	UNCURED	R2-11-VBR2
	11%	ADD R2 INTO RAP	UNCURED	R2-11-UNCURED
	11%	ADD R2 INTO RAP	CURED	R2-11-CURED
	14%	ADD R2 INTO RAP	UNCURED	R2-14-UNCURED
R3	8%	ADD R3 INTO RAP	UNCURED	R3-8-UNCURED
	8%	ADD R3 AND ANTISTRIPPING INTO RAP	UNCURED	R3-8-AS-UNCURED
UNREJUVENATED	N/A	VIRGIN BINDER AND VIRGIN AGGREGATES		CONTROL-VIRGIN
	N/A	ADD PG64-28 BINDER AND AGGREGATES TO RAP		CONTROL-RAP

4.2.1 Flow Number

Two replicates were tested per mixture for flow number to assess their rutting potential. As aforementioned, testing was conducted at 54.0 °C and a cyclic loading was applied axially on the top of specimens. After each cycle, permanent deformation was recorded by the cross-head of the loading machine. The test stopped when the accumulated strain reached 5% (i.e., 50,000 $\mu\varepsilon$) or when the number of loading cycles reached 10,000. During testing, the rate of strain accumulation was being calculated in real-time. Figure 4-7(a) present results of flow number test as strains are plotted over loading cycles. As shown, test results between the two replicates were generally repeatable. Figure 4-7(b) presents averages of the two replicates of each mixture and it shows that, without exception, rejuvenator-treated mixtures were softer compared to untreated control mixture (i.e., CONTROL-RAP).

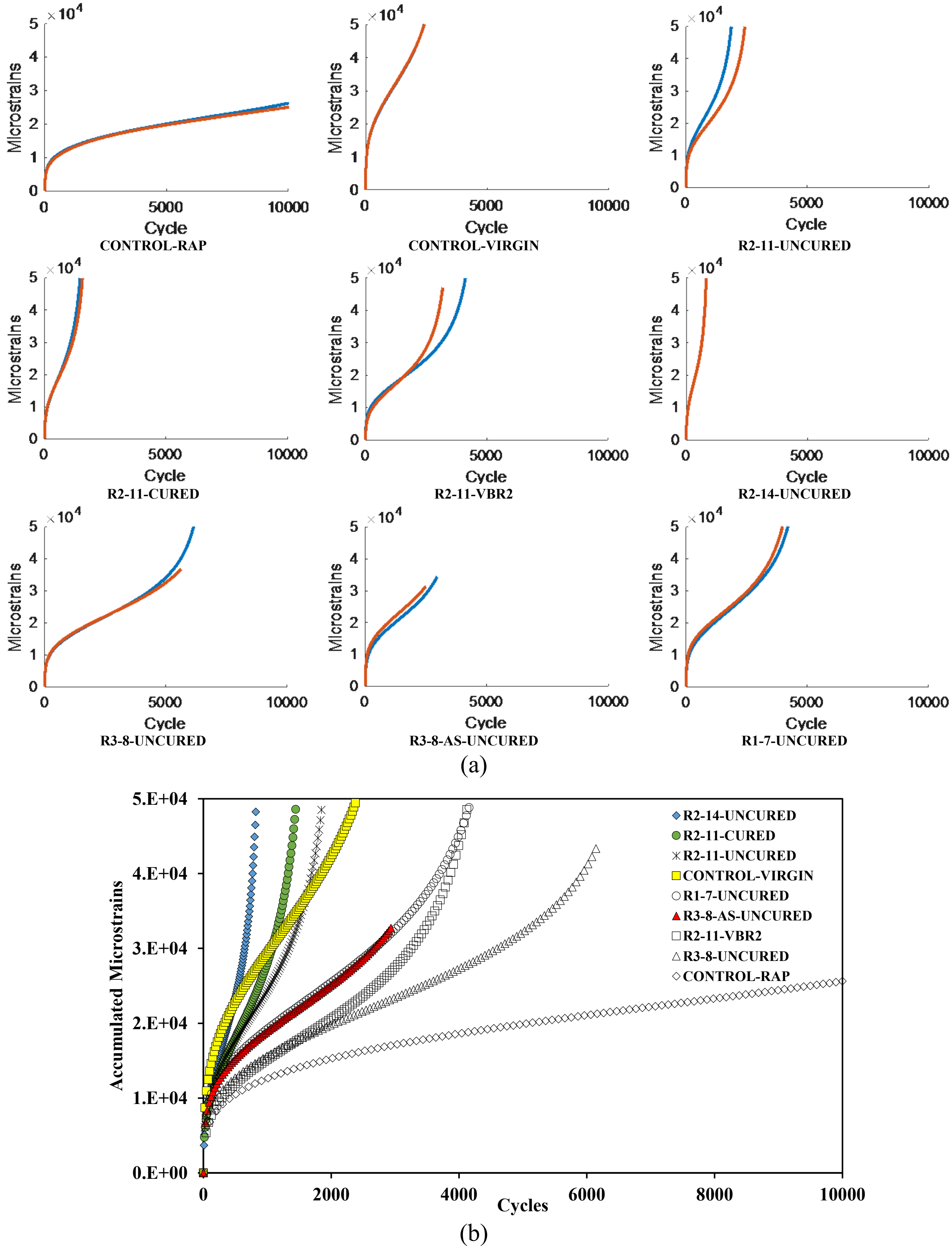


Figure 4-7. Flow number test results: (a) two replicates of each mixture; (b) averages of all mixtures.

From the results presented in Figure 4-7(b), it is seen that curing did not significantly affect the flow number test (R2-11-CURED vs. R2-11-UNCURED), while shear mixing of rejuvenator into virgin binder (i.e., R2-11-VBR2) affected more significantly the rutting performance. Compared to R2, R3 mixtures were generally stiffer in that they required more number of cycles to reach tertiary flow (Figure 4-7(b)). Tertial flow is defined as the region in which accumulated strains start to accelerate as loading continues. It is also noted that the R3 mixture with antistripping agent (i.e., R3-8-AS-UNCURED) was softer than the control (i.e., R3-8-UNCURED), which implies that the antistripping agent may help softening mixtures with RAP in addition to rejuvenators. Overall, mixture treated with 14% of R2 (i.e., R2-14-UNCURED) was the softest among all others tested.

For a more quantitative evaluation of the effect of rejuvenators on AC mixtures, a further data analysis was conducted. For each specimen, the flow number was found by taking a numerical derivation of results to capture the number of loading cycles at which the minimum strain rate was achieved (see Figure 4-8). The minimum strain rate occurs onset of the tertiary flow that is well correlated with field rutting of mixtures [55].

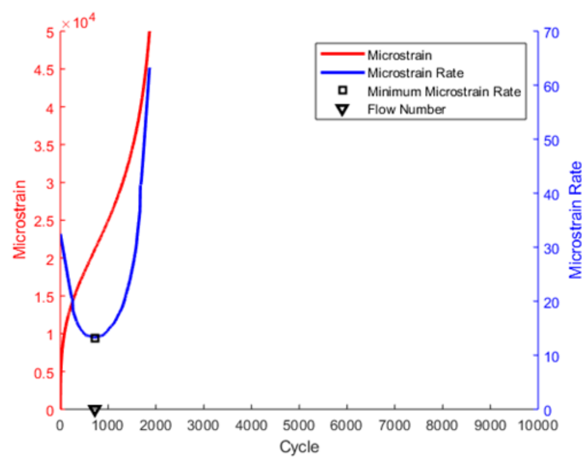


Figure 4-8. Determination of *FN* from test results.

Figure 4-9 shows the resulting flow number of each mixture. As shown, the control mixture with RAP (i.e., CONTROL-RAP) and the mixture with 14% of R2 (i.e., R2-14-UNCURED) presented the highest and lowest flow numbers, respectively. The highest flow number from the CONTROL-RAP mixture was expected due to aging. In general, rejuvenator treated mixtures effectively softened the CONTROL-RAP and reached the virgin mixture, CONTROL-VIRGIN.

It should be noted that based on company recommendation the R2 should be added to the hot virgin binder, however, in the mentioned cases (i.e., R2-11 CURED, R2-11 UNCURED) the R2 were added to the RAP materials without curing and with 5 min curing time. This modification may provide better blending between aged asphalt binders in the RAP with virgin binder compared to the situation that R2 was added to the virgin binder. In addition, the target binder in the R2-14-UNCURED mixture was PG58-34 (see Figure 3-6) which was different from the CONTROL-VIRGIN was prepared using PG64-34. As a result, the softer behavior of R2-14-UCURED was predictable.

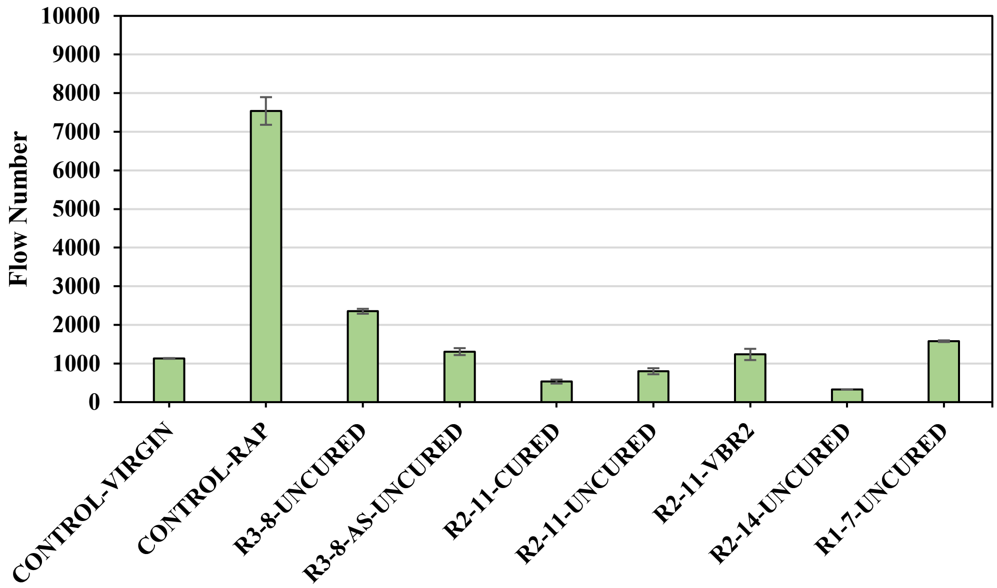


Figure 4-9. Average flow numbers with error bars.

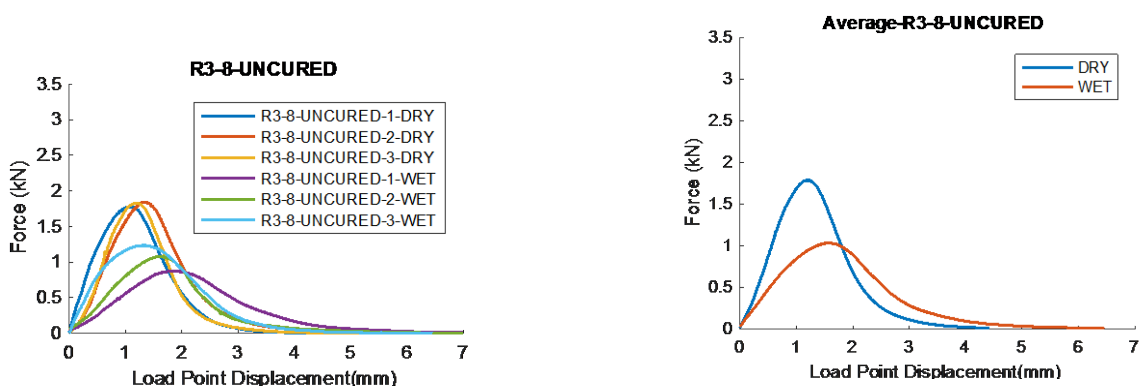
The effect of curing after adding rejuvenator can be observed by comparing R2 mixtures: R2-11-CURED to R2-11-UNCURED. Curing for 5 min after adding rejuvenator decreased the flow number by 33.5 % (i.e., from 800 to 532) which implies that curing may help rejuvenation to soften AC mixture. However, for practical purpose, curing may not be necessary since the mixture without curing is by itself softer than the virgin mixture. An increase in rejuvenator dosage (i.e., from 11% to 14%) resulted in a softer mixture (R2-11-UNCURED vs. R2-14-UNCURED).

The effect of blending method can be observed by comparing two R2 mixtures: R2-11-UNCURED (rejuvenator into RAP) vs. R2-11-VBR2 (rejuvenator into binder via shear mixing). It is seen that blending rejuvenator into binder may inhibit effective rejuvenating of RAP compared to adding the rejuvenator directly into RAP, as the flow number increased by more than 50% (i.e., from 800 to 1235).

Regarding mixtures with R3, the flow number was higher than R2 mixtures and somewhat further different from the target mixture CONTROL-VIRGIN. Adding an antistripping agent reduced flow number and made the R3 treated mixture closer to the target virgin mixture.

4.2.2 Semi Circular Bending (SCB) Fracture

SCB fracture test was performed with six replicates per each mixture by testing three specimens in dry condition and three others after moisture conditioning. Figure 4-10(a) exemplifies test results of R3-8-UNCURED for both dry and wet (i.e., moisture conditioned) cases. The test results were then averaged to obtained representative force-displacement curves for each test condition per mixture as shown in Figure 4-10(b).



(a) (b)

Figure 4-10. SCB test results for both dry and wet: (a) all replicates and (b) averages.

Averaged SCB results of all mixtures in both dry and wet conditions are presented in Figure 4-11(a). Overall, due to higher stiffness introduced by RAP, RAP-CNTRL showed the highest peak loads for both dry and wet conditions which were then noticeably reduced once rejuvenators were introduced. In addition, moisture conditioned specimens were in general more compliant than their dry counterparts in all test cases. The increased compliance is especially noticed in the post-peak regions of the test results. By comparing results of R3 with and without the antistripping agent, it is obvious that the agent improved moisture resistance of R3 mixtures by minimizing difference of results obtained from wet and dry condition (R3-8-AS-UNCURED-DRY vs. R3-8-AS-UNCURED-WET).

For a better comparison between mixtures, all averaged SCB curves were plotted together in Figure 4-11(b) and (c) for dry and wet condition, respectively. Clearly, the two control mixtures (i.e., CONTROL-RAP and CONTROL-VIRGIN) showed the highest and lowest peak load regardless of moisture conditioning. In general, rejuvenator treated mixtures effectively improved fracture resistance of the CONTROL-RAP and reached the virgin mixture, CONTROL-VIRGIN. This clearly demonstrates that the rejuvenator dosages found from the binder-level testing are appropriate for mixture-level fracture resistance.

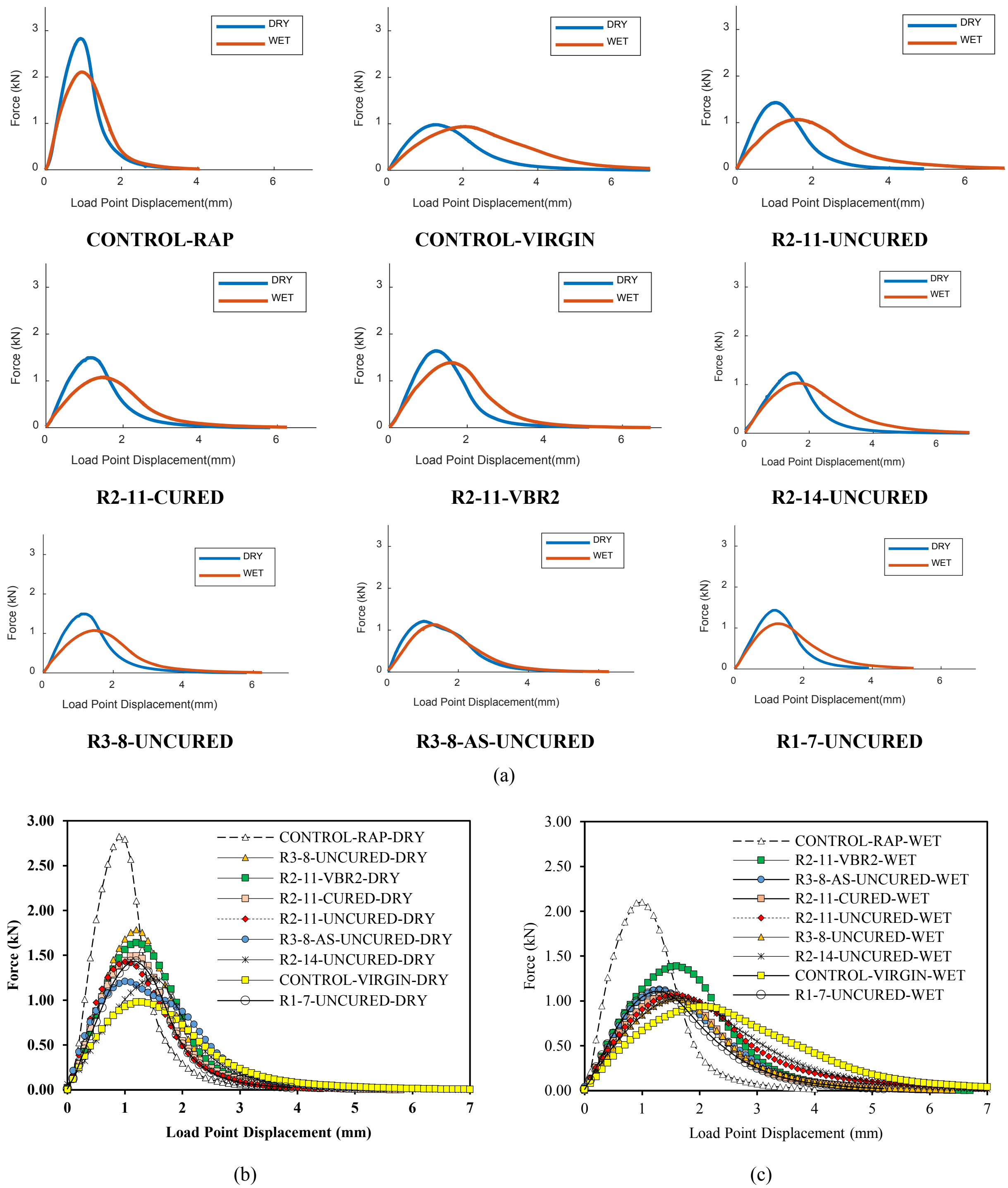
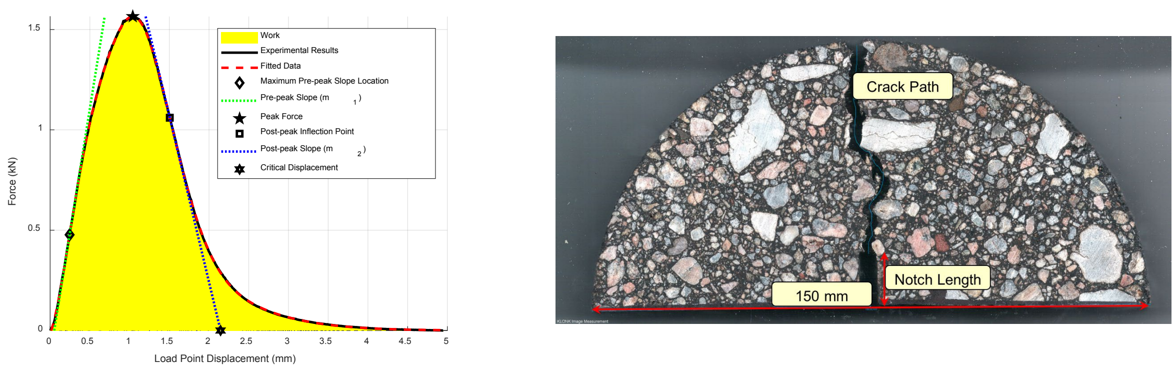


Figure 4-11. SCB test results: (a) dry and wet conditions for each case, (b) combined plots for dry condition and (c) combined plots for wet condition.

For a more quantitative evaluation of the effect of rejuvenators on AC mixtures, a further data analysis was conducted. As shown in Figure 4-12(a), several fracture-related indicators can be obtained from a load-displacement curve of SCB test result. The area underneath the curve represents the work required to completely fracture a specimen (Figure 4-12(b)). When diving the area by the ligament area can produce the fracture energy $\left(G_f\right)$ as such [70]:

$$G_f = \frac{W}{A_{lig}}$$

where W is work of fracture (i.e., the area underneath load-displacement curve), and A_{lig} is the ligament area.



(a) (b)

Figure 4-12. Analysis of SCB test results: (a) load-displacement curve where several fracture-related indicators can be found; (b) typical crack path after SCB test.

Ozer, Al-Qadi [71] proposed flexibility index (FI) which normalize fracture energy to the speed of cracking. The crack speed is considered into FI in a form of the post-peak slope (Figure 4-12(a)). The flexibility index (FI) is calculated as such:

$$FI = \frac{G_f}{m_2} * 10$$

where: FI (unitless), G_f (in kJ/m^2) and m_2 (in kN/mm) are the flexibility index, fracture energy and the post-peak slope, respectively. It is noted that m_2 is calculated at the inflection point in the post-peak region.

Figure 4-13 compares resulting *FI* of all mixtures. *FI* results obviously show differences between mixtures. Overall, all mixtures showed higher *FI*s from moisture-conditioned samples compared to dry samples with an exception of R3 mixture added with an antistripping agent. Currently, it is not clear why moisture conditioned mixtures presented higher *FI* values than dry mixtures. Similar to the observation in Figure 22, rejuvenator-treated mixtures increased *FI* of CONTROL-RAP and approached the *FI* of target mixture CONTROL-VIRGIN. It demonstrates the effect of rejuvenators to improve crack resistance by inducing more compliant nature into the RAP mixtures. R2 mixtures in dry condition performed similarly with a minimal effect of curing and blending method, while the increased content of rejuvenator improved the crack resistance. The antistripping agent in R3 mixture increased *FI* in dry case, but it was not effective when the mixture was moisture conditioned. Despite the improvement in fracture resistance due to rejuvenators, mixtures could not reach the level of fracture resistance of the target virgin mixture, which implies that mixture-level rejuvenation is partial, although the effect of rejuvenation in binder-level was optimum.

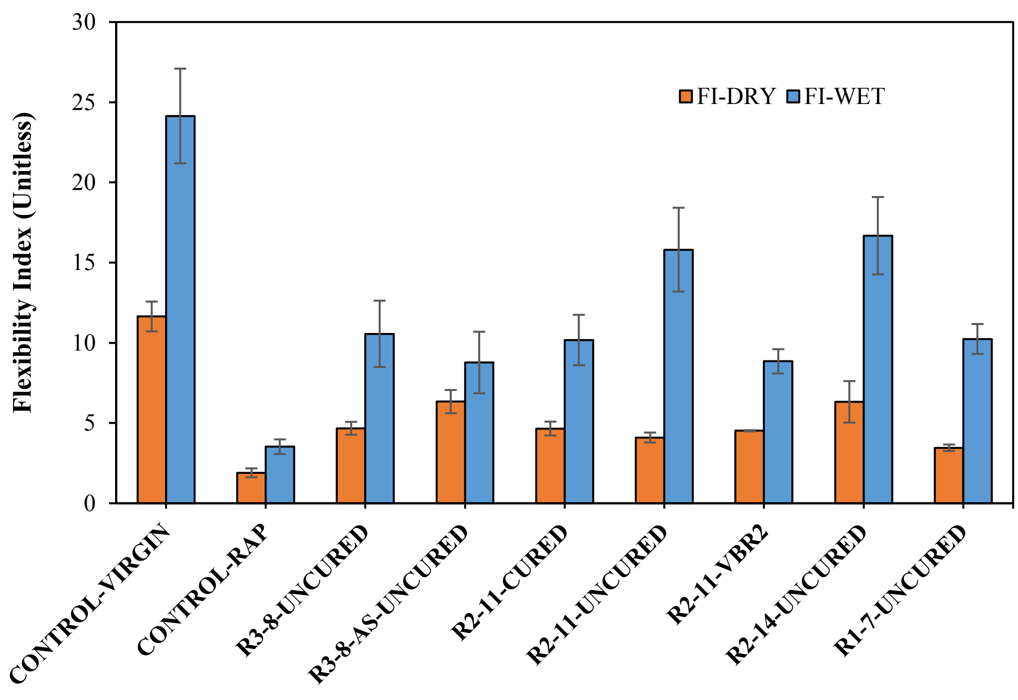


Figure 4-13. Flexibility index of each mixture.

Using the two AC performance measurements (flow number and *FI*), a performance space diagram (PSD) as shown in Figure 4-14 can be developed by relating a fracture indicator (i.e., *FI*) with a rutting indicator (i.e., *FN*). It is a typical way of the balanced mix design (BMD) of AC, as the two performance indicators (rutting and cracking) are primary distresses in AC pavement. A stiff mixture is preferable to minimize rutting while a soft and more compliant mixture is usually well suited for crack prevention in AC. Figure 4-14 demonstrates the contribution of rejuvenation by R2 and R3. The two rejuvenators softened mixtures (i.e., from *FN* of around 7,000 to *FN* less than 3,000), while increased FI more than twice. It can be noted that mixtures with higher *FN* and *FI* are desired for better performance of both rutting and fracture. It is also seen that curing of R2 mixtures resulted in a reduced *FN* while marginally improved *FI*. In contrast, shear mixing of R2 into virgin binder increased *FN* by more than 50% (from 800 to 1,200), while minimally affecting *FI*. This implies that shear mixing of R2 into binder could be an attractive way if the mixing process (i.e., mixing R2 with virgin binder, and then mixed with RAP and virgin aggregates) can be permitted without significant modification to the existing plant facility. PSD also shows that antistripping agent (AS) improved fracture resistance of R3 mixture, while rutting resistance was somewhat reduced. The increase of *FI* indicates an additional benefit of AS other than moisture damage mitigation.

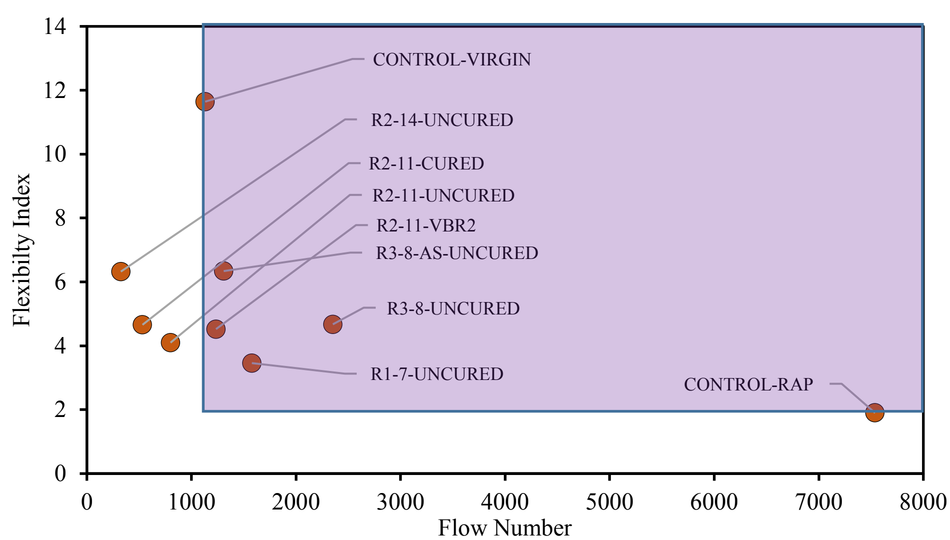


Figure 4-14. PSD of mixtures tested in dry condition.

Chapter 5 Summary, Conclusions, and Recommendations

Research outcomes and findings from the Phase I of this study [1] resulted in consequential research needs for more specific investigation of high-RAP mixtures with rejuvenators in order to achieve realistic implementation into future high-RAP paving projects. To this end, three different rejuvenators and an anti-stripping additive were added to the asphalt binder/mixture with 65% RAB/RAP content and 35% virgin materials (i.e., binder and aggregate). The results of various laboratory tests, including two AC performance tests (i.e., flow number and SCB fracture under dry and wet conditions) and four types of binder tests including the DSR (i.e., PG and G-R), kinematic viscosity, FT-IR, and SARA were carried out. The following conclusions can be made based on the test-analysis results:

- PG binder testing was successfully used to determine the proper dosage range of each rejuvenator. However, it is limited to assess the effects of rejuvenators in mechanical properties (stiffness-oriented) only. It appears that chemical restoration is quite limited and rejuvenator-dependent, which has been demonstrated by the FT-IR test results.
- From the SARA testing, it appears that the long-term performance of rejuvenated binders are rejuvenator-dependent, as R2 in this study better performed than binders modified by R1 and R3. This infers that selection of rejuvenators can be aided by examining chemical characteristics in addition to the mechanical (PG-oriented) properties and performance.
- AC mixtures treated with rejuvenators at the dosage levels selected from the binder PG testing showed improved fracture resistance compared to unrejuvenated mixtures.
- Rejuvenation methods (e.g., blending and/or curing) can alter performance of mixtures. For example, in this study, R2 softened mixtures more than enough when it was directly mixed into RAP, while its addition to binder (through shear mixing) improved both performance in a more balanced manner.
- Based on the PSD of the balanced mixture design concept, the four mixtures (7% R1, 8% R3 with and without AS, and 11% R2 mixed into binder) met the performance criteria. None of the four mixtures went through curing.

5.1 Recommended Future Research

The conclusions drawn in this study are mainly based on the tests carried out on the binder samples aged through laboratory processes, namely RTFO (short-term) and PAV (long-term). However, it should be noted that the current laboratory aging standards are limited to mimic actual aging processes occurred in field. As a consequence, further studies that mimic the actual field-aging process, such as the effects of ultraviolet light, moisture, and vehicular stresses, should be pursued. In addition, field-level validation of the findings from this study are recommended to examine if the dosages and mixing-blending methods can be implemented in production plants and field pavement sections with improved performance when the high amount of RAP is mixed.

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