FINAL REPORT

LONG-TERM AGING OF RECYCLED BINDERS Contract Number: BDV29 Two 977-01

Submitted to The Florida Department of Transportation Research Center 605 Suwannee Street, MS 30 Tallahassee, FL 32399

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Disclaimer:

The opinions, findings, and conclusions expressed in this publication are those of the authors and not necessarily those of the State of Florida Department of Transportation.

	SI* (MODERN	METRIC) CONV	ERSION FACTORS	
	APPROXI	MATE CONVERSIC	ONS TO SI UNITS	
Symbol	When You Know	Multiply By	To Find	Symbol
-		LENGTH		-
in	inches	25.4	Millimeters	mm
ft	feet	0.305	Meters	m
yd	yards	0.914	Meters	m
mi	miles	1.61	Kilometers	Km
		AREA		
in ²	square inches	645.2	Square millimeters	mm ²
ft ²	square feet	0.093	Square meters	m ²
vd ²	square vard	0.836	Square meters	m ²
ac	acres	0.405	Hectares	ha
mi ²	square miles	2.59	Square kilometers	km ²
		VOLUME		
floz	fluid ounces	29.57	Milliliters	ml
nal	gallons	3 785	Liters	1
ff ³	cubic feet	0.028	cubic meters	m ³
vd ³	cubic vards	0.765	cubic meters	m ³
yu	NOTE: VO	umes greater than 1000 L	shall be shown in m ³	
		MASS		
07	ouncos	29.25	Grams	
02	ounces	20.30	Giallis	y ka
T	short tons (2000 lb)	0.404	mogagrams (or "motric ton")	Ng (or "t")
0-		MPERATURE (exact	degrees)	10
۳F	Fahrenheit	5 (F-32)/9	Celsius	°C
		or (F-32)/1.8		
		ILLUMINATIO	N	
fc	foot-candles	10.76	Lux	Ix
fl	foot-Lamberts	3.426	candela/m ²	cd/m ²
	FOR	CE and PRESSURE	or STRESS	
lbf	poundforce	4.45	Newtons	N
lbf/in ²	poundforce per square inch	6.89	Kilopascals	kPa
	APPROXIM	ATE CONVERSION	IS FROM SI UNITS	
Symbol	When You Know	Multiply By	To Find	Symbol
Symbol	When You Know	Multiply By LENGTH	To Find	Symbol
Symbol	When You Know	Multiply By LENGTH	To Find	Symbol
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Symbol mm m km m ² m ² ha	When You Know millimeters meters kilometers square millimeters square meters square meters Hectares	Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47	To Find Inches Feet Yards Miles square inches square feet square yards Acres	Symbol in ft yd mi in ² ft ² yd ² ac
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Symbol mm m km km m ² m ² ha km ²	When You Know millimeters meters kilometers square millimeters square meters square meters hectares square kilometers	Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME	To Find Inches Feet Yards Miles square inches square feet square yards Acres square miles	Symbol in ft yd mi in ² ft ² yd ² ac mi ²
Symbol mm m km mm ² m ² ha km ²	When You Know millimeters meters meters kilometers square millimeters square meters square meters Hectares square kilometers	Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034	To Find Inches Feet Yards Miles square inches square inches square feet square yards Acres square miles	Symbol in ft yd mi in ² ft ² yd ² ac mi ²
Symbol mm m km m ² m ² ha km ² mL	When You Know millimeters meters meters kilometers square millimeters square meters square meters Hectares square kilometers Milliliters	Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264	To Find Inches Feet Yards Miles square inches square feet square yards Acres square miles fluid ounces Galloop	Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz
Symbol mm m km mm ² m ² ha km ² mL L m ³	When You Know millimeters meters meters kilometers square millimeters square meters square meters Hectares square kilometers Milliliters liters square meters	Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 25.214	To Find Inches Feet Yards Miles square inches square feet square yards Acres square miles fluid ounces Gallons guide feet	Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal gal gal
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Symbol mm m m km m ² m ² ha km ² mL L m ³ m ³ g kg	When You Know millimeters meters meters kilometers square millimeters square meters square meters square meters square meters square kilometers Milliliters liters cubic meters grams kilograms	Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202 1.02	To Find Inches Feet Yards Miles square inches square feet square yards Acres square miles fluid ounces Gallons cubic feet cubic feet cubic yards Ounces Pounds Pounds Pounds	Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ oz lb
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Symbol mm m m m ² m ² m ² ha km ² mL L m ³ m ³ g kg Mg (or "t") °C lx cd/m ² N	When You Know millimeters meters kilometers square millimeters square meters square meters square meters square meters square meters Hectares square kilometers Millilliters liters cubic meters grams kilograms megagrams (or "metric ton" Celsius lux candela/m² FOR	Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202 1.103 MPERATURE (exact 1.8C+32 ILLUMINATIOI 0.0929 0.2919 CE and PRESSURE 0.225	To Find Inches Feet Yards Miles square inches square feet square feet square yards Acres square miles fluid ounces Gallons cubic feet cubic yards Ounces Pounds short tons (2000 lb) Ehrenheit N foot-candles foot-Lamberts Or STRESS Poundforce Poundforce	Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ oz lb T °F fc fl lbf

Metric Conversion Table

*SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380 (Revised March 2003)

4. Title and Subtitle Long-Term Aging of Recycled		5. 3. Recipient's Catalog No.			
Long-Term Aging of Recycled		5. Report Date			
	Binders				
		6. Performing Organization Code			
7. Author(s)		8. Performing Organization Report No.			
Hesham Ali, Ph.D., PE					
Mojtaba Mohammadafzali, Ph.J	D. Candidate				
9. Performing Organization Nar	ne and Address	10. Work Unit No. (TRAIS)			
Florida International U	Iniversity				
	2	11. Contract or Grant No.			
		BDV29 Two 977-01			
12 Snoncoming A NI	nd Address	12 Trme of Depart and Demad Coursed			
12. Sponsoring Agency Name a	nd Address	13. Type of Report and Period Covered			
Florida Department of	Transportation	Draft Final Report			
		14 Sponsoring Agency Code			
		14. Sponsoring regency code			
15 Supplementary Notes					
Supported by a grant fi	rom the Florida Department of Transpo	rtation			
Supported by a grant h	Tom the Monda Department of Transpo.	Tation			
Asphalt pavement is America's more is recycled every year [1]. To effect continues to refine its maintenance financial and environmental benefit pavement. Aging of the recycled bit Asphalt binders harden with age an properties. Traditional methods the However, the recycled asphalt shot asphalt binder ages quickly. This is binders and compares it with that of Aging rates of recycled asphalt binder with the aging rate of the virgin bit power and other considerations.	st recycled material. Eighty million tons of a ectively maintain its 40,000 miles of paved and rehabilitation methods. Hot in-place re ts when used properly. The primary concern nder has a strong impact on the durability of d become prone to cracking. Rejuvenators a nat evaluate the effectiveness of binder re- puld have acceptable long-term performance research looks beyond early performance a f virgin asphalt. Hers, rejuvenated by five different commercia- nder. These rejuvenators were selected thro The Pressure Aging Vessel (PAV) was us emented to characterize the level of aging.	asphalt, nearly 80% of all milled asphalt pavement, I roads, the Florida Department of Transportation ecycling is a rehabilitation method with recognized n with this method is the longevity of the recycled f the pavement. are added to hard asphalt to enhance its rheological givenation only examine the early performance. ce. The pavement might crack prematurely if the and evaluates long-term aging of recycled asphalt ally available products, were studied and compared ough a screening process based on their softening sed to simulate aging, and the high temperature The cracking resistance of recycled asphalt mixes			

procedures can be used to modify the specifications so that the fonge (ity of the recycled asphart is considered.						
17. Key Word	18. Distribution State	ment				
Aging, Asphalt, Pavement, Recycling,	Public distribution					
Performance Grade, Long-term Performance						
19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price			
Unclassified	Unclassified	66	n/a			

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EXECUTIVE SUMMARY

Asphalt pavement is America's most recycled material. Eighty million tons of asphalt, nearly 80% of all milled asphalt pavement, is recycled every year [1]. To improve the maintenance of its 40,000 miles of paved roads, the Florida Department of Transportation continues to refine its maintenance and rehabilitation methods. Hot in-place recycling is a rehabilitation method with recognized financial and environmental benefits when used properly. The primary concern with this method is the longevity of the recycled pavement. Aging of the recycled binder has a strong impact on the durability of the pavement.

Asphalt binders harden with age and become prone to cracking. Rejuvenators are added to hard asphalt to enhance its rheological properties. Traditional methods that evaluate the effectiveness of binder rejuvenation only examine the early performance. However, the recycled asphalt should have an acceptable long-term performance. The pavement might crack prematurely if the asphalt binder ages quickly. This research looks beyond early performance and evaluates long-term aging of recycled asphalt binders and compares it with that of virgin asphalt.

Aging rates of recycled asphalt binders, rejuvenated by five different commercially available products, were studied and compared with aging rate of the virgin binder. These rejuvenators were selected through a screening process based on their softening power and other considerations. The Pressure Aging Vessel (PAV) was used to simulate aging, and the high temperature Performance Grade (PG) was implemented to characterize the level of aging. Also, the cracking resistance of recycled asphalt mixes was evaluated using the Texas Overlay Test. The change in the mix crack resistance over time was evaluated using the Accelerated Pavement Weathering System.

The results showed a significant difference in the long-term aging of samples recycled with different recycling agents. While some samples aged significantly slower than the virgin binder, others aged faster. Analysis of the aging rates of binders revealed that selecting a proper recycling agent can extend the service life up to nine years. In contrast with original binders, the aging rate of recycled binders did not decrease significantly after the first 20 hours of PAV exposure. Therefore, even if when recycled binders pass PG requirements, they might be less durable than virgin asphalt. Mix tests showed that rejuvenated mixes generally have a better initial fatigue and reflective cracking resistance than new mixes. However, their cracking resistance might drop faster over the pavement's life cycle.

The knowledge obtained from this study identified approaches to evaluate long-term aging of recycled binders. Performing a durability test, limiting the aging rate, and increasing PAV exposure time are the three proposed alternative procedures. These procedures can be used to modify the specifications so that the longevity of the recycled asphalt is considered.

CHAPTER 1: INTRODUCTION

1.1 Background

Florida has 120,000 miles of paved roads. This translates into billions of square yards of pavement to maintain. The continuing goal to refine cost-effective maintenance and rehab methods remains an important objective. The mainstream approach to pavement rehabilitation has been to mill the deteriorated asphalt and replace it with new asphalt mix. Pavement hot in-place recycling (HIR) is a process where a train of equipment heats the asphalt, mills it, adds aggregate and rejuvenating oils, mixes it, then adds more asphalt. When used in the right application, HIR may yield substantial cost and environmental benefits.

Recently, FDOT used hot in-place recycling under its developmental Specifications. This is in line with a national trend of expanding the use of in-place pavement recycling methods [2]. This trend is propelled by in-place recycling costs and environmental benefits. Recent research showed that initial construction cost savings can be up to 50%, and life-cycle cost savings can be up to 40% [3].

One of the most critical elements in hot in-place recycling (HIR) is binder rejuvenation. The asphalt binder aging process hardens asphalt and reduces its ductility. The rejuvenation process applies a recycling agent to enhance the binder's rheological properties. The traditional approach to evaluate the effectiveness of rejuvenators and determine the appropriate rejuvenator dosage is generally based on viscosity and penetration requirements. However, these criteria do not take the stability and durability of recycled asphalt binders into account. A recycling agent might effectively reduce the viscosity of asphalt, but it also adversely affects its durability. It is known that components that enhance the durability of recycled asphalt might be less effective in the reduction of the viscosity [4].

To achieve sustainable pavement recycling, the recycled asphalt should be at least as durable as virgin asphalt. More evaluation on the durability of recycled asphalt is needed. This study investigates the aging of recycled asphalt binders and compares it with that of virgin binders.

1.2 **Objectives**

The objective of this work was to evaluate long-term aging of recycled asphalt binders. In order to achieve longevity of recycle pavement, it is necessary to ensure that the recycled asphalt does not age quickly. For this purpose, the long-term aging of recycled binders was compared with that of virgin asphalt. The knowledge obtained from this study is beneficial for the development of performance-related specifications for the mix and the recycling agent.

Current specifications and previous research are reviewed in the next section of this chapter. Rejuvenating agents used in this study were selected through a screening process explained in Chapter 2. Superpave Performance Grade tests and procedures were used to simulate aging and characterize the level of aging, as described in the Chapter 3. The cracking resistance of recycled mixes and changes in the mixes over time was evaluated by the Texas Overlay Test and the Accelerated Pavement Weathering System (Chapter 4). Based on the knowledge obtained from this work, some modifications to the specifications for recycled asphalt binder are proposed to consider long-term aging and durability.

1.3 **Review of Literature**

1.3.1 Asphalt Aging and Rejuvenation

We can define rejuvenation as to restore, return to an original condition and cure from aging effects. A discussion on asphalt aging is needed prior to discussing the rejuvenation process. Asphalt aging, hardening, and embrittlement are well documented in literature. An excellent discussion of mechanisms contributing to aging is presented by Hanson et al. (2009) [5]. Their discussion of aging mechanisms is summarized below. Asphalt hardening can take place both in a reversible or permanent manner. Reversible changes basically are referred to as molecular associations like steric effects or wax crystallization.

Permanent changes, on the other hand, occur as a result of chemical reactions like oxidation, or physical changes such as loss of lighter molecules. Among all of the mechanisms contributing to asphalt hardening, the focus should be on those that most significantly influence the long-term performance of pavement.

Reversible Hardening

As the asphaltene weak attractions are destroyed over time, asphalt molecules change their orientation and become more tightly packed. These changes lead to an increase in the asphalt's density and stiffness. This process is accelerated by increased temperatures. Some of the reversible hardening mechanisms include the following processes.

Low-Temperature Physical Hardening

Some asphalts exhibit a substantial increase in stiffness when subjected to low temperatures over a period of time. The increase in BBR Stiffness directly correlates with a measured increase in asphalt density. Using a series of physico-chemical techniques, including Differential Scanning Calorimetry (DSC), phase contrast microscopy, and polarized light microscopy, Claudy and coauthors identified the cause of low-temperature physical hardening to be the reversible microcrystallization of long-chain aliphatic molecules, or waxes. As the waxes crystallize, both asphalt density and low-temperature stiffness increase [6].

Steric Hardening

Steric hardening is the process that describes asphalt hardening at ambient temperatures over a period of time during several weeks or months. This steric hardening effect leads to the gradual reorientation of polar molecules as they strive to reach thermodynamic equilibrium.

Thixotropy

The property of asphalt binder whereby it settles when unagitated, thixotropy is thought to result from hydrophilic suspended particles that form a lattice structure throughout the asphalt binder. This causes an increase in viscosity and thus, hardening. Thixotropic effects can be somewhat reversed by heat and agitation.

Irreversible Hardening

Irreversible hardening is a permanent change in the chemistry or composition of the asphalt, which can take place through one of the following mechanisms.

Loss of Lighter Molecules

As lighter oil fractions are lost, asphalt becomes harder. This is similar to the distillation process in vacuum towers as crude oil is refined. There are several mechanisms through which the smaller, less polar maltene oils are lost, including volatilization, selective adsorption, and syneresis. Volatilization is the evaporation of lighter constituents from asphalt. Selective Adsorption is the movement of smaller, mobile asphalt molecules into pores within the aggregate. Syneresis is the separation of less viscous liquids from the more viscous asphalt binder molecular network.

Increasing Molecular Size

Functional groups of different molecules can react with each other, linking different molecules together through covalent sigma bonds. Common reactions of this type include condensation, polymerization and vulcanization. Condensation is a reaction that joins two different functional groups. Polymerization is the combination of many smaller molecules to form high molecular weight polymers. Vulcanization is a chemical process by which elemental sulfur cross-links polymer molecules to make them larger.

Asphalt Oxidation

Oxidation is the chemical reaction of asphalt with oxygen, such that individual carbon or sulfur atoms within asphalt molecules increase in the oxidation state. Asphalt oxidation is commonly recognized to be the dominant cause for long-term age hardening. The most conclusive evidence comes from lab and field research that consistently reports a very high correlation between carbonyl content and the various rheological measures of hardening.

Asphalt Rejuvenation

There has been a long-standing belief that the principle function of recycling agents is to replace asphalt molecules that oxidized, evaporated, or adsorbed into the porous aggregate. This is achieved by adding a calculated amount of the recycling agent to bring back physical properties of the binder to its original state. Physical properties that have been used for this purpose are viscosity, penetration and/or performance grade. The most likely cause of cracking in the recycled mix is related to additional asphalt aging. Such cracking most likely initiates near the surface, where ongoing oxidation causes embrittlement of the asphalt.

1.3.2 Current Specifications

A proper mix design is one of the keys to a successful pavement recycling practice. Currently, there is no nationally accepted method for creating a mix design for HIR and agencies use different methods [7]. Generally, HIR mix design methods aim to enhance the properties of the old asphalt mixes to make them similar to those of a new mix. Therefore, the process involves methods that are usually based on standard Hot Mix Asphalt (HMA) mix design methods. Type and gradation of new asphalt binder and aggregates and their proportion are elements to be identified in a new HMA mix design. Mix designs for HIR are more complex. Old aggregate and asphalt binder from existing pavement should be identified, and type and amount of new asphalt, new aggregate and recycling agent should also be determined.

One of the most important parts of the HIR process is rejuvenation of the asphalt binder. A proper recycling agent with/without new asphalt binder is typically added to the aged asphalt to enhance its rheological properties. There are four major approaches to rejuvenating asphalt binder for HIR [7]:

- Use of a rejuvenator to restore properties of the existing aged asphalt.
- Use of a soft virgin asphalt binder.
- Use of a virgin asphalt binder, rejuvenator and new aggregate altogether.
- Use of recycled mix properties rather than asphalt binder properties to determine the mix design.

We consider the first approach in this study. A mix design for HIR consists of the following steps:

- Evaluation of the properties of existing pavement material, including asphalt binder and aggregates.
- Determination of the type and amount of the recycling agent/rejuvenator.
- Testing of trial rejuvenated binder and mix samples to obtain optimum mix design.

As mentioned earlier, the mix design for HIR is usually processed based on HMA mix design methods. The NCHRP synthesis 421 titled "Recycling and Reclamation of Asphalt Pavements Using In-Place Methods" summarizes mix design methods for in-place recycling methods implemented by agencies and contractors, as shown in Table 1-1 [8]. This shows that while many agencies do not use any mix design, among those that implement the mix design, the Superpave and Marshall methods are most popular. Florida uses Superpave for its HIR mix designs, though it was not listed in the Table 1-1 reference.

Mix Design	Recycling Method						
Method	Hot In-Place Recycling	Full Depth Reclamation					
Do not do mix design	CA, ID, IA, MO, VT, WA	CA, DE, ID, IA, NC, NH, NV, RI, SD, VT, WA, WI	CT, DE, ID, MN, MT, NC, NH, NV, NY, SD, VT, WI				
Marshall	AZ, KY, NE	AZ, MN, NE, OR, VA, WY	VA				
Superpave	CO, KS, MO, ND, UT, VT	CO, KS, MO, ND, UT, VT	MD, MO, UT, VA				
Wirtgen		V A	AK, CA, IA, VA				
Other	NY, TX	CT, MT, NY	AL, CO, GA, NE, NY, SD, WY				

Table 1-1 Mix design methods implemented by agencies and contractors

Blending Charts

Blending charts are used to determine the amount of the recycling agent/rejuvenator based on properties of aged asphalt binders, recycling agent and target asphalt binder viscosity. These charts can be created according to penetration grade, viscosity grade or performance grade (PG). Figure 1-1 shows an example of a blending chart [8]. In this chart, the left vertical axis is viscosity or $G^*/\sin\delta$ of the RAP binder, and the right vertical axis shows viscosity or $G^*/\sin\delta$ of the new asphalt or recycling agent (RA). If a horizontal line is plotted from the target viscosity until it intersects the line that connects the viscosity of the old and new asphalt/RA, and then a vertical line is plotted from that intersection to the horizontal axis, the required percentage of new asphalt/RA will be obtained. Alternatively, if the percentage of the RA is already known, the viscosity of it may be determined through the reverse route.

The ASTM D4887 [9] standardizes the preparation of viscosity blends for hot recycled asphalt materials. Two charts are displayed in Figures 1-2 (viscosity) and 1-3 (PG). Figure 1-2 is a blending chart based on viscosity and should be used similarly to Figure 1-1, except that two inclined lines are plotted for the two new asphalt/RAs (one softer than the other), and the horizontal line indicates the percentage of RAP (referred to as Reclaimed Asphalt Material or RAM in this case) rather than soft asphalt.



Figure 1-1 Blending chart used to select the percent of new asphalt or RA needed to provide the desired binder properties (based on FHWA 1997) [8].



Figure 1-2 Allowed percentage of RAM (RAP) binder based on viscosity (ASTM D4887).

Blending Charts Based on Performance Grade System

With an increasing use of the Superpave Performance Grade system for the HMA mix designs, mixes containing RAP (such as HIR mixes) are also increasingly being designed by the PG system. Figure 1-3 shows an ASTM 4887 blending chart for use with PG. The procedure to find the RAP content to meet the PG criteria in the desired grade is as follows:

- 1. Point the RA high PG temperature on the left vertical axis (A) and RAM(RAP) binder high PG temperature on the right vertical axis (B), and then connect points A and B with a straight line.
- 2. Draw a horizontal line from the target high PG to the line (AB), then draw a vertical line from the intersection to the horizontal axis (Point G).
- 3. Repeat steps 1 and 2 for intermediate and low temperatures. The projection between points G and H is the allowable RAP content needed to pass PG requirements.



Figure 1-3 Allowed percentage of RAM (RAP) binder based on PG measurements.

Blending Equations

The proper dosage of recycling agent needed to achieve the target performance grade can also be determined through blending equations. If the percentage of the old asphalt binder and recycling agent is already known, the high, intermediate and low PG critical temperatures of RA, which can be a fresh asphalt binder softened with a rejuvenator, is determined as follows:

$$T_{virgin} = \frac{T_{blend} - (\% RAP \times T_{RAP})}{(1 - \% RAP)}$$
(Eq. 1-1)
Where:

Where:

 T_{virgin} = Critical temperature of virgin asphalt or recycling agent (high, intermediate, or low). T_{Blend} = Critical temperature of desired blended asphalt binder (high, intermediate, or low). %RAP = Percentage of RAP expressed as a decimal.

 T_{RAP} = Critical temperature of recovered RAP binder (high, intermediate, or low). In the scenario where the performance grade of the recycling agent is known, the percentage of the old asphalt binder is:

$$\% RAP = \frac{T_{Blend} - T_{virgin}}{T_{RAP} - T_{virgin}}$$
(Eq. 1-2)

Classification of Recycling Agents

Recycling agents are classified based on their properties in ASTM D4552 and ASTM D5505 (for the emulsified recycling agent). Tables 1-2 and 1-3 show the necessary classifications and property requirements.

	ASTM	RA	1	BA	5	BA 1	25	BA.	75	BA 2	50	RA 4	500
Test	Test			1143		1174		114	/0	11/12	00	11/4 4	
	Method	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Viscosity • 60°C [140°F], mm²/s	D2170 or D2171	50	175	176	900	901	4500	4501	12500	12501	37500	37501	60000
Flash Point, COC, °C [°F]	D92	219 [425]		219 [425]		219 [425]		219 [425]		219 [425]		219 [425]	
Saturates,wt,%	D2007		30		30		30		30		30		30
Tests on Residue from RTFO or	D2872 or												
TFO oven 163°C [325°F]	D1754												
Viscosity Ratio ^A	44	122	3		3		3		3		3		3
Wt Change,±,%	**		4		4		3		3		3		3
Specific Gravity	D70 or D1298	Report		Report		Report		Report		Report		Report	

Table 1-2 Classification of Recycling Agents (ASTM 4552)

AViscosity Ratio= Original Viscosity 60°C [140°F], cSt

Table 1-3 Classification of Recycling Agents (ASTM 5505)

	200			3 1990 1975				
Tests	Test Method	ER-1		ER-2		ER-3		
		min	max	min	max	min	max	
On emulsion								
Viscosity, 50°C, SFS	D244		100	20	450	20	450	
Sieve, %	D6933		0.1		0.1		0.1	
Storage stability,	D6930		1.5		1.5		1.5	
Residue, by distillation. %	D6997	65		65		65		
Dilution			report ^A					
Specific gravity	D70		report		report		report	
Compatibility ^B	varies		report		report		report	
On residue from distillation			20		20			
Kinematic Viscosity, 60°C, mm ² /s	D2170	50	200					
Saturates, %	D2007		30		30		30	
Solubility in Tri- chloroethylene	D2042	97.5		97.5		97.5		
On residue from distillation after RTFO ^C								
Penetration, 4°C, 50 g. 5 s	D5			75	200	5	75	
RTFO, weight	D2872		4		4		4	

A ER-1 shall be certified for dilution with potable water.

⁹ This specification allows a variety of emulsions, including high-float and cationic emulsions. The engineer should take the steps necessary to keep incompatible materials from co-mingling in tanks or other vessels. It would be prudent to have the chemical nature (float test for high float emulsions, particle charge test for cationic emulsions, ^c RTFO shall be the standard. When approved by the engineer, the Thin Film Oven Test (Test Method D1754) may be substituted for compliance testing.

1.3.3 Performance Grade of Recycled Asphalt Binders

Performance-based properties of rejuvenated binders and asphalt mixtures were investigated by Shen, Amirkhanian, and Tang (2006) [12]. A Pen 40/60, a Pen 60/80, and three types of rejuvenator at different percentages (0, 6, 9 and 14 percent) were tested. Each binder was evaluated at different aging stages: the original binder before aging, the RTFO-aged binder to simulate construction aging, and the pressure aging vessel (PAV)-aged RTFO residue to represent long-term aging of the binder. The optimum rejuvenator content to reach target PG was obtained. The PG was determined using the Dynamic Shear Rheometer (DSR) and Bending Beam Rheometer (BBR) tests according to AASHTO M320 criteria.

Results revealed that performance properties of the aged binder significantly affect rejuvenator content required to reach a target PG. The optimum percentage of rejuvenator can be presented with the use of blending charts. As expected, an increase in the amount of rejuvenator leads to the improvement of fatigue resistance and shrinkage parameters, but decreases rutting resistance.

A Hot Mix Asphalt (HMA) that used original and rejuvenated binder was also compared in this research. Mixtures using rejuvenated aged binder performed as well as those with original asphalt binder and in some cases had better performance. Dynamic Stability (DS) of the mixtures was observed to decrease with the increased amount of rejuvenator. However, at the same time, fracture properties were improved.

Polymer modified binders also need to be recycled and rejuvenated. Tao MA et al. (2010) performed a comprehensive study, including penetration and PG tests for polymer modified asphalt binders (PMB) [13]. The effects of using a common recycling agent on aged PMB. Along with a new modifying additive called TPSTM, were investigated. A styrene-butadiene-styrene (SBS) block copolymers modified asphalt binder was tested in this study.

Penetration grade tests, softening point, ductility and elastic recovery, and Superpave PG tests including DSR, BBR and a direct tension test (DDT) were performed on original asphalt binder, a recycling agent rejuvenated binder and a TPSTM modified binder. Results showed that aging leads to deterioration of pavement surface and destruction of SBS modified binder microstructure. In addition, the rejuvenation of this modified binder with some common recycling agents that were tested in this study did not lead to any significant improvements. However, modifying the aged binder with the TPSTM improved the performance of recycling the polymer modified asphalt remarkably. This modified recycled binder showed even better road performance and durability than the original PMB.

A procedure to determine the rejuvenator dose to be mixed with RAP to satisfy PG requirements was determined by Martins Zaumanis et al. (2014) [14]. According to this study, high, intermediate and low PG critical temperatures decrease linearly when a dose of a rejuvenator is added to the mix. It was also found that the PG sum (sum of high and low temperature PG) of the RAP binder is often higher than that of the virgin binder. Adding rejuvenators usually decreases it slightly, but it still remains higher than that of the virgin binder. Based on these results, the following criteria are proposed to determine the minimum and maximum rejuvenator dose:

- The maximum rejuvenator dose should be determined to meet the requirements of the targeted high PG temperature.
- The minimum rejuvenator dose should be determined to meet the requirements of the targeted low PG temperature and intermediate PG temperature.

According to these criteria, a sample of RAP can be restored by a rejuvenator only if the dose required to meet the intermediate and low PG temperatures does not cause failure in meeting high temperature requirements. Figure 1-4 shows the minimum rejuvenator dose needed to meet the high and low PG temperatures.



Figure 1-4 Minimum Rejuvenator Dose to Reach PG and Penetration of PG64-22 binder [14]

1.3.4 Effectiveness of Rejuvenation

The effectiveness of rejuvenators for production of high RAP content pavement was evaluated by Zaumanis and co-authors (2013) [15]. Nine recycling agents with different origins (plant oil, waste motor oil, refinery based oils and engineered oils) and with different volume percentages of rejuvenator were tested. Kinematic viscosity and penetration tests at two different temperatures were used to evaluate the softening effectiveness. Temperature susceptibility was addressed by the consistency of results at different temperatures. This was obtained using the Penetration Index (PI), Penetration-Viscosity Number (PVN) and Bitumen Test Data Chart (BTDC) of the softened binders.

According to this study, the PI is a good and simple measure for elevuating the effectiveness of rejuvenation. However, results from the PVN were found to be non-dependable due to the difference in the PI range. Among nine rejuvenators tested, five were evaluated as effective at improving low-temperature performance of the pavement. They succeeded in maintaining or increasing indirect tensile strength, fracture energy and low-temperature creep compliance. Also, organic blend, refined tallow, aromatic extract and distilled tall oil rejuvenators were able to reduce the viscosity of RAP to the level of the virgin binder.

Influence of the use of the stiff RAP binder on the properties of the RAP mixture might be a challenging issue in high RAP content pavement. Hajj et al. (2013) evaluated a bio-rejuvenating agent called "BituTech" RAP through laboratory work [16]. The impact of this product on the viscoelastic properties of mixtures containing up to 50% RAP were evaluated for any possible moisture damage or thermal cracking problems that may arise from the use of RAP in the wet-freeze environment of Manitoba, Canada.

Results showed that loss modulus (E") of mixtures is significantly increased by using Bitu-Tech. Mixtures with 50% RAP and Bitu-Tech have a performance similar to those with just 15% of RAP and no Bitu-Tech. Hence, the product was concluded to be effective to make the use of high RAP content possible. Adding RAP also improved moisture resistance. According to TSRST test results, Bitu-Tech was effective in improving the low-temperature performance of RAP mixtures.

Rejuvenator Seal Material

Juntao Lin et al. (2012) investigated the effects of the rejuvenator seal material (RSM) on aged binders and asphalt mixtures [17]. To study aged binders, RFTO and Ultra-Violet (UV) light were implemented to age asphalt. Then, the aged binder was rejuvenated using two types of RSM, and the performance of the resulting asphalt was evaluated by a creep recovery test, fatigue test, viscosity test, temperature sweep test, and component analysis. Both tested RSMs were successful in reducing viscosity and complex modulus and phase angle of aged asphalt. They also decreased rutting and fatigue parameters. The rejuvenator sealer material also had a higher creep recovery, compared to the control binder.

In order to study the performance of asphalt mixtures when treated by the rejuvenator seal material, the mixtures were subjected to a frequency sweep test, wheel tracking test, static creep test, indirect tensile strength test, raveling test and skidding resistance test. After running all of these tests, it was concluded that RSM decreases the rutting resistance of hot mix asphalt. RSM also causes a decrease in tensile strength and an increase in creep strain. RSM is found to be effective in decreasing raveling, but at the same time, it decreases skidding resistance.

Rejuvenation is also studied through rheological techniques [18]. A dynamic Mechanical Thermal Analysis was implemented to determine the thermal transition due to the collapse of the compact structure constituted by asphaltene. As the binder ages, the transition shifts to higher temperature grades. Rejuvenation, on the other hand, shifts the transition back to lower temperatures. The rutting factor, $G^*/\sin \delta$, defines the highest temperature the binder can stand without permanent deformation. Implementation of the rejuvenator was shown to postpone permanent deformation, in comparison with the original binder.

Moreover, according to viscosity function results, high temperatures above 200 °C are required to mix the hardened binder on appropriate fluid-like material. However, the use of rejuvenators decreases the abovementioned temperature.

Diffusion

In order to recycle asphalt, new binder or rejuvenator is added to the old aged asphalt. Diffusion, the process of mixing old and new binders, is affected by the old asphalt's degree of aging. Karlsson & Isacsson (2002) studied rejuvenator diffusion using the Fourier Transform Infrared Spectroscopy by Attenuated Total Reflectance (FTIR-ATR) [19]. The rate of diffusion is generally affected by parameters such as viscosity of the media, shape and size of the molecules of diffusing material and temperature. Hence, the increase in viscosity caused by aging influences the rate of diffusion.

Based on results of the mentioned study, while RTFO artificial aging does not affect the diffusion of rejuvenator significantly, an increase in molecular size and polarity of the maltene caused by distillation of binder reduces the rate of diffusion. Results from implemented Stoke-Einstein equations properly fit diffusion data. In general, it is concluded that aging does not have a major effect on diffusion rates.

Effect of HIR Heating

Asphalt is heated during the process of hot in place pavement recycling (HIR). Hot air and infrared radiation are the major mechanisms that heat asphalt for HIR. The effects of heating asphalt pavements on the effectiveness of recycling were investigated by Mallick et al. [20]. Finite element multiphysics modeling, using heat transfer/chemical engineering modules of COMSOL software, was implemented to simulate asphalt heating during HIR. It was found that the intense heat on the surface dissipates rapidly along the depth of pavement. Hence, heating the surface cannot ensure a desired temperature, even at a 25mm depth of pavement. Using hot air, however, results in a more uniform temperature profile along the depth. The temperature affects the extent of rejuvenation. An effective rejuvenation cannot take place in temperatures below 100°C. This temperature normally cannot be achieved at any depth more than 30 to 50mm. Effectiveness of rejuvenation is also dependent on time of mixing. Therefore, the designing process of HIR and choosing of the rejuvenator should consider depth of mix, temperature and time of mixing.

Waste Oils as Rejuvenators

Using waste oils as rejuvenators can help with sustainable development by reducing environmental pollution that comes from waste oil disposal and also reduces the cost of road system maintenance. Subsequently, there is a trend when it comes to evaluating different types of waste oil as bitumen rejuvenators.

Dedene et al. (2011) Studied waste engine oil as an aged asphalt softening agent. $G^*/sin(\delta)$ was measured for aged bitumen mixed with waste engine oil and also aged bitumen mixed with original asphalt binder [21]. Adding waste oil was shown to successfully decrease the viscosity of the aged binder and subsequently improve thermal cracking resistance, and it makes mixing, handling and compaction of the asphalt mix easier.

Used motor oil was again considered a rejuvenator by Oliveira et al. (2013) for 100% RAP content recycled 100% RAP) HMA [22]. In this work, the optimum amount of used motor oil was obtained through penetration grade tests. The minimum percentage of used motor oil that changes the grade of the aged binder from a 10/20 to 20/30 penetration grade is considered the optimum amount.

Rheological tests were then conducted to evaluate the performance of used motor oil rejuvenated with recycled HMA. Results confirmed that tested used motor oil was an effective rejuvenator for a 100% RAP mixture and can provide an effective sustainable paving solution.

Waste cooking oil (WCO) was evaluated as a rejuvenation agent [23]. Softening point, penetration, Brookfield viscosity, DSR and Fourier transform infrared spectroscopy tests were conducted on virgin, aged and WCO rejuvenated binder. Results revealed that 3–4% of waste cooking oil content can successfully rejuvenate group Pen 40/50 aged bitumen and restore original Pen 80/100 bitumen performance. The rejuvenated bitumen was less severely affected by short-term aging, compared to the original binder. Hence, using WCO can be considered a new green and economical solution in pavement preservation.

Rejuvenation in Surface Treatments

Asphalt rejuvenators are used in fog seals as a surface treatment technique. This method works well as a low-cost practice to maintain asphalt pavement. During a study conducted by Tricor Refining, LLC, pavement condition was improved by a fog seal as demonstrated in testing thousands of core samples [24]. Rejuvenation restored maltenes in aged and oxidized binder and softened the asphalt in the top 1/4" to 3/8" portions of pavement surface. This reversed the aging caused by air, moisture and time. Rejuvenation also prevented intrusion of air and water, and

postponed the deterioration of the pavement. The chemical composition of aged asphalt was also improved by rejuvenation.

On the other hand, a simple fog seal treatment is not very effective, according to FHWA research conducted at Texas A&M University [25]. Fog sealed and untreated pavement sections were cored and evaluated in terms of susceptibility to permanent deformation and permeability. Results suggest significant improvement neither in softening of the asphalt nor in permeability. Even minor variations between performance of treated and untreated cores were believed to be due to original asphalt variability rather than rejuvenation. Also, no relief in oxidation aging was observed. Generally, it was concluded that fog sealing has no significant effect on improving the durability of pavement, while rejuvenator fog seals are shown to be effective.

Performance of rejuvenation treatment applied on US-40 pavement in Henry County, Indiana was evaluated and reported by Jusang Lee et al.(2012) [26]. Four performance tests, friction, permeability for the pavement and DSR, and Contact Angle (CA) for the asphalt were conducted. Results showed that the rejuvenator reduced the dynamic shear modulus, but increased the phase angle and water drop contact angle. However, no remarkable improvement of permeability was observed. The influence depth of the treatment was also found approximately to be 0.5 inches. A significant reduction (app. 56%) of friction numbers was observed due to rejuvenating treatment. However, these reduced friction values still met the INDOT minimum requirement.

1.3.5 Aging and Durability of Recycled Asphalt

One important consideration regarding the use of recycled asphalt is the durability of pavement containing RAP. The recycled binder is expected to achieve a long life, as long a life as that of the original binder or longer. Few studies were found that shed light on the durability and aging behavior of recycled asphalt binder.

An early effort to compare the durability of RAP and virgin asphalt binder was shown in a laboratory study by D. Fritchen (1997) [27]. A new asphalt mix and two rejuvenated old pavements were tested. Moisture damage to asphalt concrete was simulated by a vacuum-submerged conditioning procedure, followed by several freeze-thaw thermal cycles. Performance of the pavement was monitored by non-destructive resilient modulus tests. Results showed that recycled asphalt concrete usually performed as well as new asphalt samples.

Superpave binder and mix tests were used to compare the performance of rejuvenated and virgin asphalt. The Rolling Thin Film Oven (RTFO) and Pressure Aging Vessel (PAV) were used to simulate short-term and long-term aging, and the Dynamic Shear Rheometer (DSR) and Bending Beam Rheometer were used to test asphalt binder performance. Results showed that the performance of rejuvenated samples were similar to or better than that of virgin asphalt [28].

In a study by Ohio State University and FHWA, the durability of mixes containing RAP was evaluated [29]. This work was aimed at determining the maximum RAP content that does not adversely affect the durability of the mix. DSR, BBR and Moisture Damage tests (AASHTO T283) were performed on four mixtures with RAP percentages between 0 and 30. To quantify the durability of HMA, samples were aged through heating in an oven, and absorbed energy at failure was determined before and after aging. No recycling agent was added to the mixes containing RAP. Results showed a higher creep stiffness for samples containing RAP. Samples containing 30% RAP had the best performance in terms of absorbed energy at failure.

Recycled asphalt was aged during an experiment that investigated the intermingling process between recycling agents and aged asphalt binders [30]. An 80 to 90 percent RAP mixture was tested, which was prepared by millings at a specific pavement section, mixed with 0.5% and 1% of a commercial recycling agent. Also, two control mixtures were prepared with the virgin asphalt binder, one with burnt aggregate, and the other with a heated RAP aggregate. A dynamic modulus test was conducted. An accelerated aging protocol was also used to evaluate the intermingling or diffusion of a recycling agent into aged asphalt binder material. An inert gas oven was used to eliminate oxidation of the asphalt binder. While the mix exposed to the conventional oven showed a significant change in the dynamic modulus, those exposed to the inert gas oven did not experience a major change in dynamic modulus values over time. Therefore, a long-term increase in the stiffness of the binder seems to be related to binder oxidation rather than diffusion. However, as seen from the minor changes that occurred in the inert mix, it can be concluded that long-term diffusion takes place.

Singh, Zaman and Commuri studied the durability of recycled asphalt by using long-term oven (LTO) aging of HMA mixes containing RAP, and by conducting dynamic modulus ($|E^*|$) tests [31]. Two samples were tested in this study: Mix 1 contained PG 64-22, an unmodified binder with 25% RAP, and Mix 2 consisted of a Styrene-Butadiene-Styrene (SBS) modified binder mix and 15% RAP. The asphalt content and type and gradation of the aggregate were the same for both samples. Samples were compacted by a Superpave Gyratory Compactor (SGC) and were subjected to LTO-aging in accordance with AASHTO R30. Dynamic modulus tests were conducted before and after aging at six different loading frequencies, and at four different temperatures. Results showed an increase between 42% and 60% in $|E^*|$ due to LTO-aging. An important finding from this study was that mixes with higher RAP content aged at a slower rate.

In a recent study, impacts of aging and RAP percentages on the effectiveness of recycling agents were investigated [32]. Several samples of asphalt mixtures containing different percentages (25% and 45%) of RAP materials, proper dosages of six different recycling agents, and PG 76-22 virgin asphalt binder were prepared. The control was the PG 76-22 virgin binder. First, mixtures were put in a 135° C heated oven for two hours (short-term), and then for six more hours (long-term). Then, the asphalt binder was recovered from the mixture through AASHTO T 164 and ASTM D5404 procedures. The high- and low-temperature performance grade of the asphalt was determined by DSR and BBR tests. Figure 1-5 shows true temperature performance grade results for control and rejuvenated binders extracted from aged mixtures. These results showed the following:

- All recycling agents were able to decrease the levels of low and high PG.
- Aging of the asphalt mixtures did not have a significant impact on the recycling agents' ability to decrease the low and high PG.
- Recycling agents had the same effectiveness in rejuvenating the aged RAP binder with 25% and 45% RAP percentages. This shows that increasing the percentage of RAP to 45% did not impact the durability of the binder.
- Analysis of shear modulus master curves showed that these curves were lower in rejuvenated binders than those in the control binder. It can be concluded that using recycling agents in mixtures containing RAP improves fatigue cracking resistance without adversely affecting rutting resistance.



Figure 1-5 True Temperature PG Results for Control and Rejuvenated Binders Extracted from Mixtures Containing 25% RAP; (a) High Temperature PG with 25% RAP, (b) Low Temperature PG with 25% RAP (c) High Temperature PG with 45% RAP, (d) Low Temperature PG with 45% RAP, [32].

1.4 Summary

Rejuvenation is the process of adding some recycling agent to the hard asphalt to enhance its rheological properties. One of the requirements for a successful rejuvenation is that the recycled binder be at least as durable as the virgin asphalt. The research introduced in this chapter looks beyond initial performance and aims to study the long-term aging of recycled asphalt binder.

Previous research on asphalt binder aging and rejuvenation was reviewed, and standard specifications related to asphalt recycling were reported. None of the specifications were given any procedure to control the durability of the recycled binder. Also, limited research was performed on the aging and durability of recycled asphalt. Generally, previous studies show desirable performance of recycled asphalt. To the authors' knowledge, no previous work was done beforehand to quantify the aging rate of recycled binders and compare it to that of virgin asphalt.

CHAPTER 2: REJUVENATOR PROPERTIES AND SCREENING

This chapter describes the process of identifying rejuvenating agents for the binder long-term aging study.

2.1 **Process for obtaining rejuvenators**

The project team obtained ten rejuvenators from various vendors, as presented in Table 2-1. Florida International University (FIU) solicited participation from various vendors and producers from the Asphalt Recycling and Reclaiming Association. Five-gallon samples of all products were received. The SAE 10W30 motor oil was also evaluated for asphalt softening effectiveness.

Product Name ¹	Tag ¹	Product Description ²				
Naphthenic Base Oil – Low Viscosity	NOL	These two recycling agents restore select maltenes that have oxidized from asphalt binder to rebalance the chemical composition of the aged asphalt. Refined from a naphthenic wax-free crude source in California's San Joaquin Valley, these products				
Cationic Water-based Emulsion	CWE	offer excellent solvency, fluxing and mixing capabilities with the asphalt. NC asphalt-free, meaning that it contains 0% asphaltene and is composed of maltenes, saturates, and acidiffins to restore the aged binder.				
Anionic Emulsion 1	AE1	These are emulsion-containing polymers. AE2 contains double the polymer amount included in AE1. Products are generic and most Kenses HIP specification. Division				
Anionic Emulsion 2	AE2	1200.				
Bio-Rejuvenator, Oil base Fluid	BOF	This is a mixture of long-chain and tricyclic organic acids, resin acids, fatty a esterified fatty acids and vegetable oils. These products are manufactured				
Bio-Rejuvenator, Oil base Semi-fluid	BOS	renewable raw materials and can be used as a viscosity cutting agent or as a powerful penetrating oil and co-mingling agent for Recycled Asphalt Pavement.				
Heavy Paraffinic Distilled Solvent Extract	HPE	Asphalt modifiers with high aromatic content. The manufacturer produces 19 different products with various viscosity, flash point and other properties. Two				
Residual Oil Solvent Extract	ROE	types were selected based on previous use by HIR contractors. HPE is a lighter product than ROE.				
Petroleum Neutral Distillate	PND	PND is an oil extract that contains about half aromatic and half naphthenic molecules to maintain compatibility between the asphalt and the rejuvenator oil.				
Arizona Pine Oil	APO	A Polyol ester pine chemical derived from a co-product of the pulp and paper industry; a light yellow oil.				
Conventional Motor Oil	СМО	The SAE 10W30 conventional motor oil was evaluated as a rejuvenator.				

Table 2-1 Products Obtained for Evaluation

¹Assigned by the authors

² Claimed by manufacturers

2.1.1 Specifications provided by manufacturers

Table 2-2 presents some key specifications of the products, as provided by the products' suppliers and/or other suppliers. The viscosity is reported at various temperatures for these products. All available viscosity measurements are reported in Table 2-2.

	Viscosity at 77 ℉(25℃), SFS	Viscosity at 104 °F(40 °C), cSt	Viscosity at 140 ℉(60℃), cSt	Viscosity at 212 F(100 °C), cSt	Flash Point, COC, F(C)
ASTM Standard	D-445	D-445	D-2170	D-445	D-92
NOL	-	-	200-500		400 (204) min
CWE	100-200	-	200-500	-	-
AE1	-	-	-	-	212 (100) min
AE2	-	-	-	-	212 (100) min
BOF	-	-	100 max	-	425 (218) min
BOS	105 @80°F	-	50 @150°F	-	500 (260) (Closed Cup)
HPE	-	-	104	-	410 (210) min
ROE	-	-	-	52.2	559 (282)
PND	-	92.2	-	7.40	420 (216)
APO		43		9	563 (295) min

 Table 2-2 Viscosity and Flash Point of the Products, as declared by Manufacturers

2.2 Laboratory Evaluation

In order to select proper rejuvenators for long-term performance studies, all products were tested at the FIU Green Paving Lab. Laboratory studies included the following tests:

- Softening effectiveness through Rotational Viscosity Test at 135 °C
- Rolling Thin Film Oven (RTFO) mass loss
- Open Cup Cleveland Flash Point Test
- Physical properties observations (appearance, freezing, odor and smoke)

2.2.1 Softening Properties, Rotational Viscosity Test

The softening powers of the rejuvenators were evaluated by establishing softening curves based on viscosity measurements using a rotational viscometer (AASHTO T316-06) [33]. Softening curves were established for a sample of very hard Reclaimed Asphalt Pavement (RAP) binder.

The asphalt was softened by mixing 3%, 6% and 9% of the weight of each product. Rejuvenators were mixed with asphalt in the thermosel chamber. A sample of hard asphalt, heated to 135 °C, was poured into the chamber and determined quantities of recycling agents were added. Then, the mixture was hand-stirred using a spatula for two minutes. It was allowed a total of one hour between the sample preparation and the viscosity test so as to provide enough time for the emulsions to break. This mixing procedure was implemented for all products and all rejuvenator contents. In order to overcome the variability of initial viscosity values, a viscosity reduction percentage was considered as the criterion to evaluate the softening effectiveness.

Tables 2-3 to 2-13 present the results of the viscosity tests. Table 2-14 summarizes viscosity test results, and Table 2-15 shows viscosity reduction effectiveness for tested products. Figure 2-1 presents softening curves of rejuvenators. Viscosity reduction effectiveness of the products are presented and compared in Figure 2-2.

Rejuvenator : Naphthenic Base Oil – Low Viscosity (NOL)							
Test Tempera	ature: 135 °C		Date:07/23/2013	3	Spindle	: SC4- 27	
Rejuvenator Content	Speed (RPM)	Factor	Torque Percentage	Viscosity Reading (cP)	Average Viscosity (cP)	Viscosity Reduction (%)	
			82.50%	103125			
0%	2	125000	82.30%	102875	102750	NA	
			81.80%	102250			
	2	125000	48.90%	61125	61500	40.15%	
3%			49.30%	61625			
			49.40%	61750			
			29.60%	37000			
6%	2	125000	29.60%	37000	36958	64.03%	
			29.50%	36875			
9%			20.90%	26125	26250		
	2	125000	21.00%	26250		74.45%	
			21.10%	26375			

Table 2-3 Viscosity Test Results, NOL

Table 2-4 Viscosity Test Results, CWE

Rejuvenator : Cationic Water-based Emulsion (CWE)							
Test Tempera	ature: 135 °C		Date:07/24/2013	3	Spindle	Spindle : SC4- 27	
Rejuvenator	Speed (DDM)	Factor	Torque	Viscosity	Average	Viscosity	
Content	Speed (KI MI)	Factor	Percentage	Reading (cP)	Viscosity (cP)	Reduction (%)	
			85.60%	107000			
0%	2	125000	85.90%	107000	107000	NA	
			85.80%	107000			
	2	125000	54.60%	68250	68042	36.41%	
3%			54.40%	68000			
			54.30%	67875			
			38.40%	48000			
6%	2	125000	38.50%	48125	48083	55.06%	
			38.50%	48125			
9%			27.60%	34500			
	2	125000	27.90%	34875	34792	67.48%	
			28.00%	35000			

Table 2-5 Viscosity Test Results, AE1

	Rejuvenator : Anionic Emulsion 1 (AE1)								
Test Tempera	ature: 135 °C		Date:07/24/201	Spindle : SC4- 27					
Rejuvenator Content	Speed (RPM)	Factor	Torque Percentage	Viscosity Reading (cP)	Average Viscosity (cP)	Viscosity Reduction (%)			
			45.80%	114000					
0%	1	250000	45.90%	115000	114667	NA			
			46.10%	115000					
		125000	71.20%	89000	88958	22.42%			
3%	2		71.00%	88750					
			71.30%	89125					
			52.70%	65875	65958				
6%	2	125000	52.80%	66000		42.48%			
			52.80%	66000					
9%			46.20%	57750	57708				
	2	125000	46.10%	57625		49.67%			
			46.20%	57750]				

Rejuvenator : Anionic Emulsion 2 (AE2)								
Test Tempera	ture: 135 ℃		Date:07/25/2013	3	Spindle :	Spindle : SC4- 27		
Rejuvenator Content	Speed (RPM)	Factor	Torque Percentage	Viscosity Reading (cP)	Average Viscosity (cP)	Viscosity Reduction (%)		
0%	1	250000	44.70% 44.80%	111000 112000	111667	NA		
			45.00% 67.00%	112000 83750				
3%	2	125000	67.40% 67.10%	84250 83875	83958	24.81%		
6%	2	125000	50.60% 50.70% 50.80%	63250 63375 63500	63375	43.25%		
9%	2	125000	44.00%	55000 55125	55083	50.67%		
270	_		44.10%	55125		2 2 . 5770		

Table 2-6 Viscosity Test Results, AE2

Table 2-7 Viscosity Test Results, BOF

	Rejuvenator : Bio-Rejuvenator, Oil base, Fluid (BOF)								
Test Tempera	ature: 135 °C		Date:07/25/2013	3	Spindle	Spindle : SC4- 27			
Rejuvenator Content	Speed (RPM)	Factor	Torque Percentage	Viscosity Reading (cP)	Average Viscosity (cP)	Viscosity Reduction (%)			
			78.40%	98000					
0%	2	125000	78.20%	97750	97875	NA			
			78.30%	97875					
		2 125000	28.10%	35125	35375	63.86%			
3%	2		28.20%	35250					
			28.60%	35750					
			62.00%	31000					
6%	5	50000	61.90%	30950	31000	68.33%			
			62.10%	31050					
			80.80%	20200					
9%	10	25000	80.90%	20225	20225	79.34%			
			81.00%	20250					

Table 2-8 Viscosity Test Results, BOS

	Rejuvenator : Bio-Rejuvenator, Oil base, Semi-fluid (BOS)								
Test Tempera	ature: 135 °C		Date:07/23/2013	3	Spindle	: SC4- 27			
Rejuvenator Content	Speed (RPM)	Factor	Torque Percentage	Viscosity Reading (cP)	Average Viscosity (cP)	Viscosity Reduction (%)			
0%	2	125000	76.80%	96000 96500	96000	NΔ			
070	2	125000	76.40%	95500	90000	1171			
3%	2	125000	46.20% 46.90%	57750 58625	58458	39.11%			
<u> </u>	2	125000	47.20% 36.90%	59000 46125	45017	50.170/			
0%	2	125000	36.60% 36.70%	45750 45875	45917	52.17%			
9%	2	125000	35.30% 35.40% 34.80%	44125 44250 43500	43958	54.21%			

Rejuvenator : Heavy Paraffinic Distilled Solvent Extract (HPE)								
Test Tempera	ature: 135 °C		Date:07/24/2013	3	Spindle	Spindle : SC4- 27		
Rejuvenator Content	Speed (RPM)	Factor	Torque Percentage	Viscosity Reading (cP)	Average Viscosity (cP)	Viscosity Reduction (%)		
			89.10%	111000				
0%	2	125000	89.00%	111000	111000	NA		
			89.50%	111000				
	2	125000	52.30%	65375	65167	41.29%		
3%			52.00%	65000				
			52.10%	65125				
			33.10%	41375				
6%	2	125000	33.00%	41250	41667	62.46%		
			33.90%	42375				
9%			24.60%	30750	30458	72.56%		
	2	125000	24.30%	30375				
			24.20%	30250				

Table 2-9 Viscosity Test Results, HPE

Table 2-10 Viscosity Test Results, ROE

	Rejuvenator : Residual Oil Solvent Extract (ROE)								
Test Tempera	ature: 135 °C		Date:07/22/2013	Spindle	Spindle : SC4- 27				
Rejuvenator Content	Speed (RPM)	Factor	Torque Percentage	Viscosity Reading (cP)	Average Viscosity (cP)	Viscosity Reduction (%)			
			84.80%	106000					
0%	2	125000	84.10%	105000	105333	NA			
			84.40%	105000					
		2 125000	50.80%	63500	63167	40.03%			
3%	2		50.60%	63250					
			50.20%	62750					
			42.70%	53375	53250				
6%	2	125000	42.50%	53125		49.45%			
			42.60%	53250					
			35.20%	44000	44042				
9%	2	2 125000	35.30%	44125		58.19%			
			35.20%	44000					

	Rejuvenator : Petroleum Neutral Distillate (PND)									
Test Tempera	ture: 135 °C		Date:07/22/201	3	Spindle	Spindle : SC4- 27				
Rejuvenator Content	Speed (RPM)	Factor	Torque Percentage	Viscosity Reading (cP)	Average Viscosity (cP)	Viscosity Reduction (%)				
			81.60%	102000						
0%	2	125000	81.90%	102000	102000	NA				
			81.80%	102000						
			45.80%	57250	57583	43.55%				
3%	2	125000	46.00%	57500						
			46.40%	58000						
			79.20%	39600						
6%	5	50000	79.30%	39650	39633	61.14%				
			79.30%	39650						
			90.30%	22575	22617	77.83%				
9%	10	25000	90.50%	22625						
			90.60%	22650						

Rejuvenator : Conventional Motor Oil (CMO)								
Test Tempera	ature: 135 °C		Date:08/26/201	Spindle	Spindle : SC4- 27			
Rejuvenator Content	Speed (RPM)	Factor	Torque Percentage	Viscosity Reading (cP)	Average Viscosity (cP)	Viscosity Reduction (%)		
			88.50%	111000				
0%	2	125000	88.40%	111000	111000	NA		
			88.50%	111000				
	2	125000	68.80%	86000	86083	22.45%		
3%			69.10%	86375				
			68.70%	85875				
			58.30%	72875				
6%	2	125000	58.40%	73000	72833	34.38%		
			58.10%	72625				
9%		125000	48.40%	60500	60667	45.35%		
	2		48.60%	60750				
			48.60%	60750				

Table 2-12 Viscosity Test Results, Motor Oil

Table 2-13 Viscosity Test Results, APO

		Rejuvo	enator : Arizona	Pine Oil			
Test Tempera	ature: 135 °C		Date:07/02/201	5	Spindle : SC4- 27		
Rejuvenator Content	Speed (RPM)	Factor	Torque Percentage	Viscosity Reading (cP)	Average Viscosity (cP)	Viscosity Reduction (%)	
			48.80%	122000			
0%	1	250000	48.50%	121250	121833	NA	
			48.90%	122250			
		125000	56.50%	70625		41.96%	
3%	2		56.50%	70625	70708		
			56.70%	70875			
			35.20%	44000			
6%	2	125000	35.30%	44125	44042	63.85%	
			35.20%	44000			
			53.20%	26600			
9%	5	50000	53.00%	26500	26517	78.24%	
			52.90%	26450			

Table 2-14 Summary of viscosity test results

Rejuvenator Content	PND	HPE	ROE	BOS	BOF	NOL	CWE	AE1	AE2	Motor Oil	APO
0%	102000	111000	105333	96000	97875	102750	107000	114667	111667	111000	121833
3%	57583	65167	63167	58458	35375	61500	68042	88958	83958	86083	70708
6%	39633	41667	53250	45917	31000	36958	48083	65958	63375	72833	44042
9%	22617	30458	44042	43958	20225	26250	34792	57708	55083	60667	26517

Table 2-15 Summary of viscosity reduction effectiveness of rejuvenators

Rejuvenator Content	PND	HPE	ROE	BOS	BOF	NOL	CWE	AE1	AE2	Motor Oil	APO
3%	43.55%	41.29%	40.03%	39.11%	63.86%	40.15%	36.41%	22.42%	24.81%	22.45%	41.96%
6%	61.14%	62.46%	49.45%	52.17%	68.33%	64.03%	55.06%	42.48%	43.25%	34.38%	63.85%
9%	77.83%	72.56%	58.19%	54.21%	79.34%	74.45%	67.48%	49.67%	50.67%	45.35%	78.24%



Figure 2-1 Softening Curves of Rejuvenators



Figure 2-2 Viscosity Reduction of Rejuvenators

As shown in Table 2-1, CWE, AE1 and AE2 are emulsified rejuvenators that contain water. Therefore, their effectiveness per unit weight is different from those of non-emulsified rejuvenators. Emulsified rejuvenators were compared only to emulsified rejuvenators. One emulsion product was selected for long-term performance evaluation.

According to the results, BOF, APO and PND are the most powerful recycling agents among tested non-emulsified products. Motor oil, on the other hand, was found to be the least effective. CWE performed significantly better than other tested emulsions.

2.2.2 Rolling Thin Film Oven (RTFO) Mass Loss

Construction heating can cause loss of rejuvenator volatiles. This can affect the effectiveness of the recycling agents in real application. The RTFO mass loss test (according to AASHTO T240-09 standard test method) [34] was implemented to evaluate resistance of the products against mass loss during construction.

By definition, emulsions (CWE, AE1 and AE2) contain nearly 40% water. Therefore, the RTFO mass loss values of emulsions would reflect the mass of evaporated water rather than that of lost volatiles. Construction heating is not expected to cause a major loss of volatiles until after the water evaporates. As such, a RTFO mass loss was not reported for emulsions.

The RFTO mass loss is mostly related to the loss of volatiles and is a measure of the product's vulnerability to construction heat. BOS showed mass gain rather than mass loss. This is due to oxidative products formed during the test [35]. ROE had just a 0.17% mass loss, and this value ranged between 2%-7% for other products. Table 2-16 presents results from RTFO mass loss tests.

Rejuvenator	Empty Bobble Weight (gram)	Weight Before RTFO (gram)	Weight After RTFO (gram)	Initial Mass (gram)	Mass Change (gram)	Mass Change (%)	Average Mass Change (%)	Standard Deviation	Allowable Deviation
NOI	166.256	201.221	200.184	34.965	-1.037	-2.966%	-3.050%	0.00110	0.00721
NOL	165.352	200.451	199.351	35.099	-1.100	-3.134%	-3.03078	0.00119	0.00721
PND	166.932	201.543	199.377	34.611	-2.166	-6.258%	6 128%	0.00184	0.00832
IND	167.374	202.004	199.927	34.630	-2.077	-5.998%	-0.12070	0.00164	0.00832
BOE	168.719	203.649	202.028	34.930	-1.621	-4.641%	1 657%	0.00023	0 00770
BOT	165.889	200.599	198.977	34.710	-1.622	-4.673%	-4.03770	0.00025	0.00779
BOS	165.348	200.114	200.200	34.766	0.086	0.247%	0.280%	0.00046	0.00600
BOS	169.156	203.985	204.094	34.829	0.109	0.313%	0.20070	0.00040	0.00000
Motor Oil	167.526	202.462	201.361	34.936	-1.101	-3.151%	3 1380/	0.00020	0.00724
	167.333	202.450	201.353	35.117	-1.097	-3.124%	-3.13070	0.00020	0.00724
DOF	165.357	200.221	200.166	34.864	-0.055	-0.158%	0.1609/	0.00016	0.00616
KOL	165.224	200.223	200.160	34.999	-0.063	-0.180%	-0.10970	0.00010	0.00010
LIDE	169.163	204.103	203.416	34.940	-0.687	-1.966%	1.0220/	0.00061	0.00680
nre	167.526	202.418	201.762	34.892	-0.656	-1.880%	-1.923%	0.00001	0.00080
ABO	166.90	201.91	201.17	35.01	-0.74	-2.11 %	2 210/	0.00141	0.0060
APO	168.62	203.70	202.89	35.07	-0.81	-2.31%	-2.21%-	0.00141	0.0060

Table 2-16 Results from RFTO Mass Loss Test

2.2.3 Cleveland Open Cup Flash Point Test

The high temperature during mixing and construction may cause material to enflame and emit excessive smoke. To avoid safety hazards and heat damage to rejuvenating agents, a good rejuvenator should have a flash point higher than construction temperatures. The flash point of a material is defined as the lowest temperature at which it can vaporize to form an ignitable mixture in air. The Cleveland Open Cup (COC) method was implemented to obtain the flash point of the products. The flash point was determined only for non-emulsified rejuvenators.

Results from the COC flash point test are presented in Table 2-17 and are compared to those offered by manufacturers. Minimum flash point values declared by manufacturers were expected to be lower than those obtained from our tests. This was true for NOL, ROE, HPE and BOF, but was not the case for PND and BOS. However, this cannot be considered a failure of these products. The acceptable difference between flash point values obtained by different operators at different laboratories is determined as $18 \,^{\circ}$ (ASTM D-92). The difference between an observed flash point value and the declared minimum for PND is only $10 \,^{\circ}$, which falls in the acceptable range. Also, the value declared by the BOS manufacturer is a close cup flash point and cannot be compared with COC values.

Product	Flash Point AASHTO T48-06	Manufacturer Declared Flash Point							
NOL	224°C	Min 204°C							
PND	206°C	216°C							
ROE	284°C	Typical 289°C ; Min 276°C							
HPE	216°C	Min210°C							
BOF	318°C	Min 218°C							
BOS	188°C	260°C (Closed Cup)							
Motor Oil	212°C	-							
APO	304°C	Min 295°C							

Table 2-17 Open Cup Cleveland Flash Point Test Results

2.2.4 Physical Properties Observations

The appearance of the rejuvenators was observed at room temperature, as well as after being kept in a freezing temperature for 18 hours. In addition, products were heated, and the intensity of the smoke and odor released were watched and rated subjectively. Proper workability and low smoke and odor emission are considered important characteristics for an appropriate rejuvenator. While these qualitative observations can be useful to select preferred products, we did not consider these criteria in the selection of products for further evaluation. Table 2-18 presents results from the physical observation.

Product	Appearance	Smoke	Odor	Appearance after Cooling down to -18°C for 18 hours
NOL	Green, heavy oil	Low	Low	Frozen, no ice crystals
CWE	Red, light emulsion	Moderate	High	Semi-Frozen with ice crystals
PND	Dark, light oil	Moderate	Low	Liquid
ROE	Dark yellow heavy and relatively coarse liquid	Moderate	Moderate	Frozen, no ice crystals
HPE	Dark yellow, light oil	Moderate	Moderate	Frozen, no ice crystals
AE1	Dark brown, sticky emulsion	High	Moderate	Frozen with ice crystals
AE2	Dark brown, sticky emulsion	High	Moderate	Frozen with ice crystals
BOF	Dark amber oil	Low	Moderate	Liquid
BOS	Light yellow nontransparent semi-fluid oil	Low	Low	Frozen with ice crystals
Motor Oil	Transparent yellow oil	Low	Moderate	Liquid
АРО	Transparent, yellow oil	Low	Moderate	Liquid

Table 2-18 Physical Properties Observations

2.3 Summary and Final Ranking

Ten rejuvenators and one type of conventional motor oil were studied and tested to determine suitability of product for further studies. Data sheets provided by manufacturers were studied, and the appearance of products and the smoke and odor emitted when heated were observed. The effectiveness of the rejuvenators in softening hard asphalt was evaluated by measuring rotational viscosity with different rejuvenator contents. The ability of non-emulsion products to withstand construction heating was evaluated by RTFO mass loss and the Cleveland Open Cup Flash Point Test.

The results from the tests are summarized in Table 2-19. Softening effectiveness was considered the major criterion for selection. Each product was ranked based on its softening effectiveness (viscosity reduction), RTFO mass loss and flashpoint performance. An overall score was computed based on 70% weight for softening rank, 15% for RTFO mass loss rank and 15% for flash point rank. Emulsions were ranked based on softening effectiveness only. This resulted in CWE being the selected emulsified rejuvenator among the three tested.

The top four non-emulsified rejuvenators were BOF, APO, PND and NOL. However, CWE is the emulsified version of NOL, and it was selected for evaluation. To eliminate duplication, we included the next ranked product, HPE, instead of NOL. The five rejuvenators selected for further study are BOF, APO, HPE, PND and CWE.

Product	Viscosity reduction with 9% rejuvenator content	Softening Rank (1 is best)	RTFO Mss Loss	Mas Loss Rank	Flash point	Flash Point Rank	Overall score (Rank) (Lower is better)	Remarks
NOL	74.45%	4	-3.05%	5	224°C	4	3.15	Selected then replaced by HPE**
PND	77.83%	3	-6.13%	7	206°C	7	3.35	Selected
ROE	58.19%	6	-0.17%	2	284°C	3	4.1	
HPE	72.56%	5	-1.92%	3	216°C	5	3.85	Selected
BOF	79.34%	1	-4.66%	7	318°C	1	1.75	Selected
BOS	54.21%	7	0.28%	1	188°C	8	5.4	
АРО	78.24%	2	- 2.21 %	4	304°C	2	2.3	Selected – Added to the tests later
Motor Oil	45.35%	8	-3.14%	6	212°C	6	6.4	
CWE (Emulsion)	67.48%	1			-		1	Selected
AE1 (Emulsion)	49.67%	3			-		3	
AE2 (Emulsion)	50.67%	2			-		2	
*Overall Rank is b	based on70% wei	ght for Softer	ning, 15% n	ass loss and	d 15% Flashpo	oint; For em	ulsions, based on S	oftening only.

Table 2-19 Summary of Test Results

** NOL ranked in the top 3; however, since CWE is the emulsified version of the product and will be included for further evaluation, it was decided to omit NOL and replace it by the next ranked product.

CHAPTER 3: LONG-TERM BINDER AGING STUDY

The aging behavior of recycled asphalt binders was studied through Superpave Performance Grade tests and extended PAV aging. Five rejuvenators were selected in the screening process, as described in Chapter 2, namely: CWE, HPE, PND, BOF and APO, all of which were used in this stage of the study.

3.1 Material and Testing Procedures

3.1.1 Material

Two asphalt binder samples and five rejuvenators were used in this study. Both virgin asphalt samples were graded as PG 67-22, which is a common asphalt grade in Florida. Rheological performance grade tests were conducted on asphalt samples in accordance with standard specifications for performance-graded asphalt binder [36]. Tables 3-1 and 3-2 summarize the results from the Dynamic Shear Rheometer (DSR) and Bending Beam Rheometer (BBR) tests for asphalt samples.

Test Method	Sample	Aging Level	Test Temperature	G*/sin δ (kPa) (G*.Sinδ for PAV)	AASHTO M320 Criterion	Status
		Original	67°C	1.15	$G^*/\sin \delta > 1.0 \text{ kPa}$	Pass
	Binder1	RFTO	67°C	3.17	$G^*/\sin \delta > 2.2 kPa$	Pass
AASHTO		RFTO+PAV	26.5°C	3514.4	G*.Sinδ <5000 kPa	Pass
T315		Original	67°C	1.70	$G^*/\sin \delta > 1.0 \text{ kPa}$	Pass
	Binder2	RFTO	67°C	5.21	$G^*/\sin \delta > 2.2$ kPa	Pass
		RFTO+PAV	26.5°C	3670.4	G*.Sind <5000 kPa	Pass

 Table 3-1 DSR test results for asphalt samples

Test Method	Sample	Aging Level	Test Temperature	Time (s)	Stiffness (MPa)	m-Value	Status
AASHTO	Binder 1	RTFO+PAV	-12°C	60	190	0.309	Pass
T313	Binder 2	RTFO+PAV	-12°C	60	159	0.313	Pass

Table 3-2 BBR test results for asphalt samples (at -12 °C and 60 seconds)

Five rejuvenators selected through the screening process were used to soften the aged asphalt. These products are often known by their commercial names. However, these names cannot be revealed in this report. Instead, abbreviated name tags were assigned to rejuvenators for identification. Table 3-3 presents a brief description of rejuvenators, their flash point and their viscosity at 140 °F (60 °C).

3.1.2 Testing Procedure

Long-term aging of asphalt binders is studied through performance grade (PG) tests. The performance grading system is developed to test asphalt binders in conditions simulating critical stages during the life of an asphalt binder [37].
Product Tag	Brief Description	Viscosity at 140 F(60 °C), cSt	Flash point F(°C)
CWE	-Water-base emulsion from wax-free naphthenic crude -Measured Residue content: 60%	200-500 (Residue, at 140 °F)	-
HPE	-Heavy paraffinic distilled solvent extract ; dark yellow oil -High aromatic content	104 (at 140°F)	420 (216)
PND	-Petroleum neural distillate; dark, light oil -Containing about half aromatic and half naphthenic molecules	92.2 (at 140°F)	403 (206)
BOF	-Oil-based bio-rejuvenator ; dark amber -Mixture of long-chain and tricyclic organic acids, resin acids, fatty acids and vegetable oils	100 Max (at 140°F)	604 (318)
АРО	- A Polyol ester pine chemical derived from a co-product of the pulp and paper industry; light yellow oil	43 (at 104°F)	560 (293)

Table 3-3 Brief description and properties of rejuvenators

The testing program consisted of three steps: Aging, Rejuvenation and Re-aging. Asphalt samples were aged by PAV until they reached a high temperature grade of 95°C (Aging). Then, the samples were softened with the addition of rejuvenators to reach their initial grade again (Rejuvenation), and finally, the rejuvenated asphalt samples were aged again to compare the aging rate of virgin and rejuvenated asphalt (Re-aging).

Performance grade rheological tests, DSR and BBR, were used to determine the high and low temperature grade of asphalt in all three steps and at various aging conditions. The Rolling Thin Film Oven (RTFO) was used to simulate the short-term or construction aging. The long-term aging was simulated by a Pressure Aging Vessel (PAV). Table 3-4 shows tests and aging procedures implemented, as well as the corresponding standards used, and the purpose of using each of procedure and standard.

Test	Standard	Application	Remarks
Dynamic Shear Rheometer (DSR)	AASHTO T315	High temperature rheological properties	
Bending Beam Rheometer (BBR)	AASHTO T313	Low temperature creep stiffness and stress relaxation properties	
Rolling Thin Film Oven (RTFO)	AASHTO T240	Simulating short term aging	
Pressure Aging Vessel (PAV)	AASHTO R28	Accelerated long term aging	@ 100℃ &2.1MPa

Table 3-4 Tests and aging procedures

As a part of performance grade testing procedure, asphalt samples should be aged by PAV for 20 hours to simulate in-service aging due to oxidation of the binder. In this study, the PAV exposure time was extended up to 60 hours in order to simulate further aging. An aging temperature of 100 °C and pressure of 2.1MPa was applied in all steps and aging times. PAV is designed for simulating the in-service aging of asphalt; it is well recognized that the PAV is not very successful in representing the field aging condition since the aging mechanism in PAV (due to high temp and high pressure) is different than the field aging mechanism. However, it can still be used as a tool to estimate the binder's behavior over time, and it is being used for this purpose in the PG system. A study conducted by Bahia and Anderson [38] showed that the standard PAV aging procedure (20 hours) was equivalent to eight years of field aging, as tested in a pavement section in Florida.

Based on this experience, it is assumed in the current study that every hour of PAV aging corresponds to 0.4 years of field aging. The time each sample is exposed to PAV aging is called "PAV time," and the corresponding service time is called "Service Years" in this report.

Grading in performance grade systems is based on several requirements, including: minimum DSR stiffness at higher temperatures for virgin and RTFO-aged binders; maximum DSR stiffness at intermediate temperatures, and maximum creep stiffness and minimum creep rate at bottom temperatures for RTFO and PAV aged binders. However, in this study, only the high temperature grade criterion (G*/sin $\delta > 1.0$ kPa) is used as the indicator of aging levels, and low temperature aging rate was evaluated at 2 aging stages determined by high temperature grade.

In this study, reaching a high temperature grade of 95 °C was considered a failure criterion. This assumption was based on Florida's experience with the RAP PG grade. The aging time it took each sample to reach this point is regarded as an indication of longevity.

3.2 **Test Results**

3.2.1 Step 1: Aging

In this step, after the continuous grade of samples was determined, the binders were aged by PAV. The aging time was increased in 10-hour intervals until the high temperature grade exceeded 95 °C. Determination of the continuous grade was done through logarithmic interpolation or extrapolation using Equation 1 [37].

$$T_{c} = T_{1} + \left[\frac{Log(1.0) - Log(G_{1}^{*} / \sin \delta_{1})}{Log(G_{1}^{*} / \sin \delta_{1}) - Log(G_{2}^{*} / \sin \delta_{2})} \times (T_{1} - T_{2})\right]$$
(Eq. 3-1)

In which T_C is continuous grade, T_1 and T_2 are two testing temperatures and G^*_1 , δ_1 , G^*_2 , δ_2 are DSR complex modulus and phase angles at temperature T_1 and T_2 respectively. Tables 3-5 and 3-6 display the test results for Step 1.

Asphalt Sample	PAV Time (Hours)	Test Temperature (°C)	δ (°)	G*/sin δ (kPa)	High Temperature Grade (°C)
	0	67	83	1.15	(9.36
	0	76	86	0.45	08.30
	10	76	86	1.31	79.01
	10	82	88	0.63	/8.21
	20	82	86	1.02	<u>92 10</u>
		88	88	0.50	82.18
	30	82	84	1.65	87.06
	50	88	85	0.91	07.00
	40	82	80	2.71	80.00
	40	88	82	1.28	09.99
	50	82	79	4.00	02.00
		88	82	1.87	92.90
		82	79	5.94	06.61
	00	88	80	2.86	90.01

Table 3-5 DSR test results for asphalt samples aged at increasing PAV times – Binder 1

Asphalt Sample	PAV Time (Hours)	Test Temperature (°C)	δ (°)	G*/sin δ (kPa)	High Temperature Grade (℃)	
	0	67	85	1.70	71.62	
	0	76	88	0.60	/1.03	
	10	67	79	6.85	82.67	
	10	82	85	1.07	02.07	
	20	82	84	1.52	85.46	
		88	86	0.74		
Dindon 2	30	82	81	2.54	89.72	
Dilider 2		88	84	1.23		
	40	82	79	3.48	02.00	
	40	88	82	1.72	92.00	
	50	82	75	5.59	07.02	
	30	88	77	2.92	97.92	
	60	82	70	11.77	101 34	
	00	88	74	5.48	101.34	

Table 3-6 DSR test results for asphalt samples aged at increasing PAV times – Binder 2

Figures 3-1 and 3-2 show variations of high temperature grades with PAV time. The accurate PAV time to age asphalt samples to PG 95-XX is determined to be 55 hours for Binder 1 and 44 hours for Binder 2 through the linear interpolation of results.

Binder 2 is generally harder than Binder 1. So, as expected, it reached the grade of 95 °C in a shorter aging time. Both samples experienced faster aging in first 10 hours. Then, the rate of aging decreased, and the high temperature grade-PAV time curves became almost linear at an approximate rate of 0.36 to 0.38 °C/hour.



Figure 3-1 Variations of high temperature Grade with PAV time - Binder 1



Figure 3-2 Variations of high temperature Grade with PAV time - Binder 2

The RTFO was not performed for re-aged samples. Therefore, in order to estimate PAV time that causes aging similar to the standard AASHTO M320 aging procedure (RTFO+20 Hours PAV), samples that underwent the mentioned aging process were tested to determine their high temperature grade. Results showed that the RTFO+ 20 hours PAV ages asphalt similarly to the 32 to 36 hours of PAV aging (Table 3-7).

At least 1 kg of each sample was aged at determined times to supply the PG 95-XX hard asphalt for the next steps: rejuvenation and re-aging. To assure accuracy of the grade of aged samples, a DSR test was performed. As shown in Table 3-8, aged samples had a high temperature grade of $95^+_{-}1^{0}$ C.

Sample	Aging Level	Test Temperature	δ (°)	G*/sin δ (kPa)	High Temperature Grade (°C)	Equivalent PAV Time (Hours)	
Dindon 1	RTFO	82	83	1.91	CT T9	22	
Binder I	+20hr PAV	85	84	1.36	01.12	32	
Pindon 2	RTFO	82	80	3.32	01 27	26	
Binder 2	+20hr PAV	88	82	1.54	91.57	30	

Table 3-7 DSR test results for RTFO+PAV aged samples

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Sample	Aging Condition	Test Temperature	δ (°)	G*/sin δ (kPa)	High Temperature Grade (°C)			
Binder 1	55 hr PAV	82	77	5.10	94 61			
Dinuci 1	55 m 1 A v	88	81	2.35	74.01			
Binder 2	44 hr PAV	82	75	6.16	95.55			
		88	79	2.75				

Table 3-8 DSR test results for aged samples

The low temperature grade of asphalt samples was determined by BBR at two aging conditions: M320 requirement (RFTO+20hr PAV) and ultimate aging time (60hr PAV). Results are shown in Table 3-9. Critical temperatures based on creep stiffness and the m-value are determined using Equations 3-2 and 3-3 respectively [37]. The more critical (higher) temperature should be considered the low temperature grade.

$$T_{c} = T_{1} + \left[\frac{Log(300) - Log(S_{1})}{Log(S_{1}) - Log(S_{2})} \times (T_{1} - T_{2})\right] - 10$$
 (Eq. 3-2)

$$T_c = T_1 + \left[\frac{0.3 - m_1}{m_1 - m_2} \times (T_1 - T_2)\right] - 10$$
 (Eq. 3-3)

In which T_C is continuous grade, T_1 and T_2 are two testing temperatures, and S_1 , m_1 , S_2 , m_2 are BBR stiffness and m-value at 60 seconds, measured at temperatures T_1 and T_2 respectively.

Critical bottom temperatures at failure PAV times were determined by linear interpolation between two aging conditions. According to these calculations, the critical low temperature is -17.07°C for Binder 1 at 44 hours PAV time, and -22.09°C for Binder 2 at 55 hours.

Binder	Aging Level	Temperature (°C)	Stiffness at 60 Seconds (MPa)	m-value at 60 Seconds	Critical Temperature (Stiffness)	Critical Temperature (m-value)	
	RTFO +	-12	190	0.309	25.36	22.67	
	20hr PAV	-18	430	0.228	-23.30	-22.07	
Binder 1		-6	143	0.299			
	60 hr PAV	-12	263	0.258	-23.34	-15.85	
		-18	475	0.202			
	RTFO +	-12	159	0.313	27 47	24.17	
	20hr PAV	-18	319	0.277	-27.47	-24.17	
Binder 2		-6	105	0.31			
	60 hr PAV	-12	191	0.279	-26.03	-17.94	
		-18	374	0.223			

Table 3-9 BBR test results for original binders

3.2.2 Step 2: Rejuvenation

Aged asphalt was softened by mixing it with rejuvenators. Table 3-10 shows the required rejuvenator contents to be mixed with aged asphalt samples in order to soften them to their original grade. Softening curves were established based on a high temperature performance grade. The target grade for rejuvenation was determined as the original continuous grade of each asphalt sample: $68.36^+_{-}1^{0}$ C for Binder 1 and $71.63^+_{-}1^{0}$ C for Binder 2. Tables 3-11 and 3-12 show DSR test results for two asphalt samples, mixed with four rejuvenators at various proportions. These tables are followed by softening curves (Figures 3-3 and 3-4). The rejuvenator CWE is an emulsion, so its softening curves were established considering the residue content of the emulsion. The aged asphalt prepared in Step 1 was mixed with proper amounts of rejuvenators in order to obtain rejuvenated samples.

Asphalt Binder 1						Binder 2				
Rejuvenator	CWE	HPE	PND	BOF	APO	CWE	HPE	PND	BOF	APO
Dosage	33% Emulsion	27%	20%	15%	13%	30% Emulsion	22%	18%	13%	13%

Table 3-10 required rejuvenator content to soften aged asphalt samples to their original grade

Sample	Rejuvenator	Content	Test Temperature	δ (°)	G*/sin δ (kPa)	High Temperature Grade (°C)
	Origin	al Binder	67	83	1.15	68 36
	Origin		76	86	0.45	00.50
	Hard	Asphalt	82	77	5.10	94.61
		Aspilan	88	81	2.35	94.01
		10%	76	83	1.06	76 52
	1070	82	86	0.52	70.32	
		14%	67	79	1.49	70.34
	BOE		76	83	0.51	70.34
	DOI	150/	67	83	1.20	68 65
		1370	76	87	0.44	00.05
		160/	67	83	1.10	67.91
		10%	76	86	0.41	07.04
		10%	76	80	2.39	83.43
		1070	82	83	1.20	03.43
		20%	67	81	2.21	73 50
LIDE	20%	76	85	0.75	15.39	
	TIFE	25%	67	82	1.45	60.06
			76	86	0.47	09.90
Dimilar 1		270/	67	83	1.19	(0.20
Binder 1		27%	76	87	0.39	00.30
		100/	67	76	2.22	01 5 0
	DND	10%	76	77	0.93	81.30
	PND	2007	67	83	1.13	68.06
		20%	76	86	0.39	
		20%	76	84	1.18	
		(12% residue)	82	86	0.59	//.44
	CWE	30%	67	83	1.50	70.46
	CWE	(18% residue)	76	87	0.52	/0.40
		33%	67	84	1.22	(0.70
		(19.8% residue)	76	87	0.42	08.70
		50/	82	81	1.63	05 70
		3%	88	83	0.75	03.70
		100/	67	78	2.65	75 20
		10%	76	83	0.93	13.38
	APU	12%	67	80	1.77	71.75
			76	84	0.6	
		14%	64	80	1.39	66 40
			67	82	0.98	66.48

Table 3-11 DSR tests to establish softening curve – Binder 1

Sample	Rejuvenator	Content	Test Temperatur e	δ (°)	G*/sin δ (kPa)	High Temperature Grade (°C)	
	Origin	Bindor	67	85	1.70	71.63	
	Origina	ai Dilidei	76	88	0.60	/1.03	
	Hard	Asphalt	82	77	6.16	05 55	
		Asplian	88	81	2.75	95.55	
		10%	67	79	2.69	74.20	
			76	84	0.78	74.20	
	BOF	12%	67	81	2.21	73.86	
	DOI	1270	76	84	0.78	75.00	
		13%	67	82	1.74	71 74	
		1370	76	85	0.61	/1./4	
		10%	82	82	1.26	83.88	
		1070	88	84	0.61	05.00	
		20%	67	80	2.32	74.05	
	HPF		76	84	0.79	74.05	
	III L	2.2%	67	81	1.70	71 46	
		2270	76	85	0.58	/1.40	
		27%	67	83	1.14	68.04	
			76	86	0.38	00.04	
		10% 16%	76	80	1.82	80.87	
Binder 2			82	82	0.87	00.07	
			67	78	2.43	74 10	
	PND		76	83	0.79	7 1.10	
	IND	17%	67	79	2.11	73 10	
		1770	76	83	0.70	75.10	
		18%	67	80	1.81	72.05	
		1070	76	84	0.63	12.05	
		20%	76	83	1.25	77 85	
	CWF	(12% residue)	82	85	0.61	11.05	
	CWL	30%	67	83	1.60	71.06	
		(18% residue)	76	86	0.56	71.00	
		5%	82	80	1.79	86.92	
		570	88	83	0.88	00.72	
	F	10%	76	81	1.11	76.93	
	APO	1070	82	83	0.57	10.25	
	110	12%	67	78	2.65	74 70	
		12%	76	82	0.86	/4./9	
		14%	67	80	1.53	70.61	
		14%	76	83	0.53	/0.61	

 Table 3-12 DSR tests to establish softening curve – Binder 2



Figure 3-3 Rejuvenator Softening Curves -Binder 1



Figure 3-4 Rejuvenator Softening Curves -Binder 2

3.2.3 Step 3: Re-aging

Rejuvenated samples prepared in Step 2, which had high temperature grades similar to original binders, were aged again to compare their aging rate together and with those of virgin asphalts. High temperature grades were determined at 20, 40 and 60 hours PAV time. BBR tests were performed for samples aged by RTFO and 20-hour PAV, as well as at the ultimate aging condition (60 hours PAV). Detailed results from the DSR tests are tabulated in Tables 3-13 and 3-14.

Binder	Reiuvenator	PAV	Test	δ (°)	G*/sin ð	High Temperature
Diffact	Rejuvenator	Time	Temperature	0()	(kPa)	Grade (°C)
		0	67	83	1.15	68 36
		0	76	86	0.45	00.50
		20	82	86	1.02	82.18
	Original	20	88	88	0.50	02.10
	Originar	40	82	80	2.71	80.09
		-10	88	82	1.28	07.77
		60	82	79	5.94	96.61
		00	88	80	2.86	50.01
		0	67	84	1.21	68 63
		0	76	87	0.42	00.05
		20	76	83	1.20	77 56
	CWF	20	82	85	0.59	77.50
	CWL	40	82	80	1.39	84 75
		-10	88	83	0.68	04.75
		60	82	74	4.09	02 07
		60	88	78	1.89	92.91
		0	67	83	1.28	60.11
		0	76	87	0.44	09.11
		20	76	82	1.38	78.83
	LIDE	20	82	84	0.70	/0.05
	nre	40	82	79	1.72	96 19
		40	88	82	0.83	00.40
		60	82	74	3.72	02.42
		00	88	78	1.87	95.42
Binder 1		0	67	82	1.31	60.20
			76	86	0.45	09.29
		20	76	77	1.90	01 20
	DND		82	80	0.93	01.50
	FND	40	82	72	3.29	02.04
		40	88	76	1.62	92.00
		60	82	63	12.09	00.72
		00	88	68	5.20	99.72
		0	67	81	1.30	60.21
		0	76	87	0.45	09.21
		20	76	81	1.63	90.40
	DOE	20	82	84	0.84	80.40
	БОГ	40	82	77	3.28	01.71
		40	88	79	1.57	91./1
		(0	82	71	10.76	101.76
		60	88	79	5.23	101.70
		0	67	80	1.25	69.00
		U	76	84	0.44	08.92
		20	76	80	1.55	70.56
		20	82	82	0.74	/9.30
	APO	40	82	77	1.95	00.00
		40	88	79	1.01	88.09
		60	82	62	8.82	00.04
			88	73	4.06	98.84

 Table 3-13 DSR test results for Re-aged Samples - Binder 1

Rinder	Rejuvenator	PAV	Test	8 (°)	G*/sin δ	High Temperature	
Dilluer	Rejuvenator	Time	Temperature	0()	(kPa)	Grade (°C)	
		0	67	85	1.70	71.63	
		0	76	88	0.60	/1.05	
		20	82	84	1.52	95 16	
	Original	20	88	86	0.74	03.40	
		40	82	79	3.48	02.66	
		40	88	82	1.72	92.00	
		60	82	70	11.77	101.34	
		00	88	74	5.48	101.34	
		0	67	82	1.90	72.20	
		0	76	85	0.64	12.29	
		20	76	81	1.75	80.72	
	CWE	20	82	83	0.86	80.72	
	CWE	40	82	78	2.17	88 24	
		40	88	81	1.03	00.24	
		60	82	73	5.22	06.30	
		60	88	76	2.61	90.30	
		0	67	80	1.86	72.60	
		0	76	84	0.69	72.00	
		20	82	80	1.17	83.18	
	LIDE	20	88	83	0.53		
	ПГС	40	82	77	2.50	80.40	
		40	88	80	1.19	<u> </u>	
Binder		60	82	74	4.23	04.61	
2		00	88	77	2.13	94.01	
		0	67	79	1.77	71 77	
			76	84	0.60	/1.//	
		20	82	77	1.58	86.43	
	PND	DND 20	88	80	0.85	00.15	
	IND	40	82	68	5.73	06.11	
		40	88	73	2.73	90.11	
		60	82	62	11.69	103 63	
		00	88	66	5.91	105.05	
		0	67	80	1.85	72.61	
		0	76	84	0.69	72.01	
		20	82	80	1.60	86.07	
	BOF	20	88	82	0.80	00.07	
	DOI	40	82	73	5.59	97 71	
		40	88	76	2.90	27.71	
		60	88	76	9.99	110 73	
		00	91	69	7.37	110.75	
		0	67	80	1.65	71.46	
		0	76	77	0.60	/ 1.70	
		20	76	77	2.09	82 13	
	APO	20	82	80	1.05	02.75	
	110	40	82	73	3.13	91.22	
		40	88	77	1.49	11.22	
		60	82	65	12.02	102 33	
		00	88	70	5.77	102.33	

Table 3-14 DSR test results for Re-aged Samples - Binder 2

Table 3-15 summarizes the high temperature grading for the Step 3 (re-aging). Generally, samples rejuvenated by CWE and HPE aged slower when compared with the original binder, while those rejuvenated by PND, BOF and APO aged faster. Another general trend that can be observed is that re-aging curves of rejuvenated binders are more linear than those of original asphalt; while aging rates of fresh binders dropped considerably after the first 20 hours, those of the rejuvenated binders did not decrease much. For instance, samples rejuvenated by PND, BOF and APO had aging rates close to or slower than those of the original binders in the first 20 hours. However, a significant difference can be seen from 20 to 60 hours. Table 3-16 presents aging rates of different samples in two aging phases: the first 20 hours, and in between 20 and 60 hours. Results are also shown in the form of high temperature grade vs. aging time curves in Figures 3-5 and 3-6.

		High Temperature Performance Grade (°C)											
PAV Time (Hours)			Bind	er 1		Binder 2							
	Original	CWE	HPE	PND	BOF	APO	Original	CWE	HPE	PND	BOF	APO	
0	68.36	68.63	69.11	69.29	69.21	68.92	71.63	72.29	72.60	71.77	72.61	71.46	
20	82.18	77.56	78.83	81.38	80.40	79.56	85.46	80.72	83.18	86.43	86.07	82.43	
40	89.99	84.75	86.48	92.06	91.71	88.09	92.66	88.24	89.40	96.11	97.71	91.22	
60	96.61	92.97	93.42	99.72	101.76	98.84	101.34	96.30	94.61	103.63	110.73	102.33	

Table 3-15 Summary of Aging Behavior of Original Rejuvenated Samples

	Tuble 5-10 Hurdening rates of Original Refuventied Samples											
		Hardening Rate based on High Temperature Performance Grade ($^{\circ}C/hr$)										
Aging Phase		er 1			Binder 2							
	Original	CWE	HPE	PND	BOF	APO	Original	CWE	HPE	PND	BOF	APO
0 - 20 Hours	0.69	0.45	0.49	0.60	0.56	0.48	0.69	0.42	0.53	0.73	0.67	0.55
20-60 Hour	0.36	0.39	0.36	0.46	0.53	0.48	0.40	0.39	0.29	0.43	0.62	0.50

Table 3-16 Hardening rates of Original Rejuvenated Samples



Figure 3-5 Re-aging of rejuvenated binder samples - Binder 1



Figure 3-6 Re-aging of rejuvenated binder samples - Binder 2

The longevity of rejuvenated and original asphalt samples was evaluated. Reaching a high temperature grade to 95 °C was considered a typical failure point, and the PAV time it took each sample to reach this grade was called PAV failure time. The service life of samples was calculated from PAV times, assuming that every hour of PAV aging corresponds to 0.4 years of field aging.

As reflected in Table 3-17and Figure 3-7, service life analyses showed that selecting the proper rejuvenator has a significant effect on the durability of recycled asphalt binder.

Longevity		Binder 2										
Measure	Original	CWE	HPE	PND	BOF	APO	Original	CWE	HPE	PND	BOF	APO
Failure PAV Time (Hours)	55	65	65	48	47	52	44	57	61	38	35	47
Failure Service Years	22	26	26	19	19	21	18	23	24	15	14	19

Table 3-17 Longevity of Rejuvenated and Virgin Asphalt Samples



Figure 3-7 Longevity of Rejuvenated Binders Based on (a) Failure PAV Time and (b) Failure Service Years

Low temperature grades of rejuvenated samples were determined based on creep stiffness (S) and stress relaxation parameters (m-value) from BBR tests. The tests were performed at two aging stages: After aging by RFTO and 20 hours of exposure to PAV, and at the ultimate aging condition (60 hours PAV). Tables 3-18 and 3-19 present results from the BBR tests. Determination of the low temperature grade was achieved by considering the use of both the BBR parameters (S and m-value at 60 seconds) and interpolation of results, which used Equations 3-2 and 3-3 to arrive at their results. In all cases, the m-values were more critical and resulted in a higher low temperature grade. Figures 3-8 and 3-9 show the low temperature grades of samples at two aging stages based on the BBR m-value at 60 seconds graphically.

All rejuvenated binders passed the M320 low temperature requirements for PG 67-22 (S \leq 300 MPa and m \geq 0.300 at 60 seconds -12 °C). Rejuvenated samples were significantly softer (with a lower creep stiffness) at low temperatures, when compared to the original binders. APO samples were particularly very soft at low temperatures. However, in some cases, the stress relaxation parameters (m-values) of rejuvenated samples were more critical than those of the originals. More specifically, Binder 1 rejuvenated by PND and BOF, and also Binder 2 mixed with PND, had higher low temperatures after 60 hours of PAV aging, when compared with the original binder.

			Tomporatura	Stiffnoor		Critical	Critical	
Binder	Rejuvenator	Aging	$(^{\circ}C)$	(MPa)	m-value	Temperature	Temperature	
			(0)	(1)11 u)		(Stiffness)	(m-value)	
		$PTFO \perp 20hr PAV$	-12	190	0.309	-25.36	-22.67	
			-18	430	0.228	-23.30	-22.07	
	Original		-6	143	0.299			
		60 hr PAV	-12	263	0.258	-23.34	-15.85	
			-18	475	0.202			
		$PTFO \pm 20hr PAV$	-12	81.1	0.346	31 30	26.76	
		$K11^{\circ}O + 2011^{\circ}FA^{\circ}$	-18	187	0.288	-31.39	-20.70	
	CWE		-6	55.3	0.311			
		60 hr PAV	-12	112	0.289	-31.26	-19.00	
			-18	212	0.241			
		DTEO 1 20hr DAV	-12	72.8	0.353	21.50	75.92	
		K1FO + 2011 FAV	-18	178	0.27	-31.50	-23.83	
	HPE		-6	46.1	0.319			
		60 hr PAV	-12	89.8	0.29	-33.45	-19.93	
Dindon 1			-18	169	0.258			
billuer 1			-12	52.1	0.319	26.60	24.00	
		KIFO + 20nr PAV	-18	107	0.262	-30.00	-24.00	
	PND		-6	37.3	0.295			
		60 hr PAV	-12	69	0.205	-37.93	-15.67	
			-18	120	0.206			
		DTEO + 20hr DAV	-12	66.4	0.36	20.44	24.29	
		K1FO + 2011 PAV	-18	194	0.209	-30.44	-24.38	
	BOF		-6	78.1	0.292			
		60 hr PAV	-12	134	0.267	-30.61	-14.08	
			-18	235	0.208			
		PTEO + 20hr DAV	-12	29.3	0.385	41.61	34.14	
		K110 + 2011 PAV	-18	59.7	0.343	-41.01	-34.14	
	APO		-6	33.4	0.333			
		60 hr PAV	-12	73.1	0.258	-37.56	-18.64	
			-18	126	0.243			

Table 3-18 Low Temperature Grading of Samples Based on BBR- Binder 1

		^	Tammanatama	C4:ff-aga		Critical	Critical	
Binder	Rejuvenator	Aging	(°C)	(MPa)	m-value	Temperature (Stiffness)	Temperature (m-value)	
		RTFO + 20hr	-12	159	0.313	27 47	24.17	
		PAV	-18	319	0.277	-27.47	-24.17	
	Original		-6	105	0.31			
		60 hr PAV	-12	191	0.279	-26.03	-17.94	
			-18	374	0.223			
		RTFO + 20hr	-12	80.3	0.332	33 17	27 82	
		PAV	-18	160	0.299	-55.47	-27.02	
	CWE		-6	58.4	0.309			
		60 hr PAV	-12	112	0.282	-32.38	-18.00	
			-18	198	0.265			
		RTFO + 20hr	-12	62.3	0.342	20.80	24.22	
		PAV	-18	180	0.229	-30.89	-24.23	
	HPE		-6	36.5	0.372			
		60 hr PAV	-12	79.8	0.309	-29.42	-22.52	
Dim Jan 3			-18	233	0.205			
Binder 2		RTFO + 20hr	-12	44.5	0.322	25.64	34.60	
		PAV	-18	103	0.273	-35.64	-24.69	
	PND		-6	64.2	0.296			
		60 hr PAV	-12	119	0.269	-32.02	-15.11	
			-18	207	0.221			
		RTFO + 20hr	-12	88.5	0.314	25 29	26.00	
		PAV	-18	153	0.293	-35.38	-26.00	
	BOF		-6	47.7	0.308			
		60 hr PAV	-12	113	0.286	-32.17	-18.18	
			-18	201	0.258			
		RTFO + 20hr	-12	40.8	0.33	40.15	21.96	
		PAV	-18	78.9	0.316	-40.15	-34.86	
	AOP		-6	33.5	0.325			
		60 hr PAV	-12	67.3	0.29	-34.62	-20.29	
			-18	137	0.217			

Table 3-19 Low Temperature Grading of Samples Based on BBR- Binder 2



Figure 3-8 Low Temperature Grade Based on BBR m-value – Binder 1

A study on the effects of BBR parameters on thermal stress properties of asphalt binders showed that stiffness (s) is the factor that primarily controls low temperature thermal stress development [39]. Results from this study showed that the stiffness of rejuvenated asphalt is significantly less than that of original binders. This means that the magnitude of low temperature thermal stresses developed in rejuvenated binders is smaller than those developed in virgin asphalt.



Figure 3-9 Low Temperature Grade Based on BBR m-value – Binder 2

3.3 Summary

Two asphalt binder samples were aged by PAV until their high temperature performance grade reached 95±1°C. Each of these aged asphalt samples were softened by adding five rejuvenators (CWE, HPE, PND, APO and BOF) until their high performance grade dropped to their initial grade. Rejuvenated samples were aged by PAV again, and their aging behavior was compared together and with that of the original binders. The following general trends were identified:

- Different rejuvenators cause different aging rates. Two rejuvenators out of five (CWE and HPE) slowed down aging, and the three others (PND, BOF and APO) accelerated it.
- While the slope of aging curves dropped significantly after 20 hours for virgin asphalt samples, the aging curve of recycled binders were close to linear.
- The service life of recycled asphalt is highly dependent on the rejuvenator. Selecting the proper rejuvenator was observed to increase the service life up to nine years, as compared with rejuvenating with a less effective product.
- Almost the same trend experienced for high temperature PG was observed for low temperature PG.

Conclusions from these observations are discussed in Chapter 5.

CHAPTER 4: MIX AGING STUDY

The study reported in the Chapter 3 focused on long-term aging of recycled binders. This chapter investigates the aging and cracking resistance of recycled asphalt mixes. The resistance of mixes against fatigue and reflective cracking was measured by the Texas Overlay Test, and the Accelerated Pavement Weathering System (APWS) was employed to apply accelerated weathering.

Reclaimed Asphalt Pavement (RAP), obtained from the SR-15 HIR project, was used to prepare recycled asphalt samples. These samples consisted of the hot milled mix from SR-15, mixed with proper dosages of two rejuvenators and sand screenings. Two control samples were tested and compared with the recycled samples. The Control I sample was prepared by extracting all of the asphalt from RAP and replacing it with virgin asphalt, and the Control II sample was a randomly selected new FDOT-approved mix.

Rejuvenated and control samples were exposed to the APWS for 0, 1,000, and 3,000 hours. In order to study the effects of aging on cracking resistance, the Texas Overlay Test was performed at 0, 1,000, and 3,000 hours in APWS.

4.1 Accelerated Pavement Weathering System (APWS)

The long-term aging of asphalt binder is affected by environmental factors such as temperature, ultraviolet radiation and water acting on exposed surfaces and voids. Neither of the Superpave binder aging protocols, the Rolling Thin Film Oven (RTFO) nor the Pressure Aging Vessel (PAV), are capable of capturing the effects of all of these parameters. In addition, the reality is that the intensity of aging of asphalt pavement material varies by pavement depth. While surface layers experience higher aging, less aging occurs in deeper layers [40].

The APWS ages full-depth asphalt pavement specimens by simulating rain, sunshine and temperature changes. Grzybowski et al. [41] explained the development of this system and showed that the aging profile is similar to that observed in real pavement. Figure 4-1 shows the APWS at PRI Asphalt Technologies, Inc. General specifications of this system are presented in Appendix A.



Figure 4-1 Accelerated Pavement Weathering System (APWS)

4.2 **Sample Preparation and Testing Procedure**

4.2.1 Material

One hundred and forty pounds (140 lbs.) of RAP were sampled from the SR-15 HIR project. The mix was obtained by heating the asphalt mat to an average of 250°F, then milling the material to a one inch depth. The material was collected from the windrow, prior to the introduction of the rejuvenator. Thus, this mix represents only the RAP material from the project. The HIR mix design is presented in Appendix B.

Among five rejuvenating agents used in Task 3, two of the best aging performers were selected, CWE and HPE. Table 4-1 presents a brief description of these rejuvenators and some of their properties.

Product Tag	Brief Description	Viscosity at 140 °F(60 °C), cSt	Flash point F(°C)
CWE	Water-based emulsion From wax-free naphthenic crude Measured residue content: 60%	200-500 (Residue)	-
HPE	Heavy paraffinic distilled solvent extract High aromatic content	104	420 (216)

 Table 4-1 Brief description and properties of rejuvenators

4.2.2 Sample Preparation

Two samples of rejuvenated mixture and two control samples were prepared.

Control Samples (Control I and Control II)

The Control I used the aggregate extracted from the mix, blended with a new asphalt binder. The aggregate was extracted from the mix and the asphalt content was determined using NCAT Ignition Oven in accordance with AASHTO T 308 using the provided calibration factor of 0.1. Then the extracted aggregated was mixed with a PG 67-22 binder at the same binder content as determined by extraction.

Control II samples were FDOT-approved mixes of Type SP-9.5 and FC-9.5. These samples represented common asphalt mixes used in Florida with gradations similar to the original mix. The mix design and quality control testing of each mix is presented in Appendix B.

Rejuvenated Samples (CWE and HPE)

The two rejuvenated samples were prepared using the RAP mixture, mixed with CWE and HPE rejuvenators. To characterize the binder and establish softening curves, 180 grams of binder was recovered in accordance with ASTM D5404. The PG was determined in accordance with AASHTO M320 as presented in the Table 4-2. The sampled mix had experienced heating during hot milling. Therefore, the criterion for the RTFO residue (G*/sin δ <2.2 kPa) was used to determine its high temperature grade.

Since the sampled mix had experienced construction aging, the criterion for the RTFO residue $(G^*/\sin\delta < 2.2 \text{ kPa})$ was used to determine the high temperature PG.

A softening curve was established for each rejuvenating agent and the recovered binder. These curves were used to determine the dosage needed to reduce the high performance grade temperature to 67 $^{\circ}$ C. Figure 4-2 shows the softening curves, and Table 4-3 presents rejuvenator

percentages of the mixtures and their high temperature PG. All percentages are reported as the Total Weight of Mixture (TWM).

Rejuvenated samples were prepared by mixing the RAP with appropriate amounts of rejuvenator and 3% Screening Sand. The sand screening was added to the mixture to account for the breakdown in the ignition oven (that the Control aggregate would experience).

Rejuvenated mixtures and also the Control I, were evaluated for their design at 50 gyrations, and the maximum specific gravity was determined in accordance with ASTM D 2041.

	Property	AASHTO Test Method	Specifications	Results	
Recovered Bind	er				
		70 °C			6.11
Dynamic Shear G*/sin& 10 rad/s	k Pa	76 ℃	T 315	2.2 min.	2.86
G / Sino, 10 1au/ S	82 °C			1.38	
PAV RESIDUE	(100°C, 300 psi, 20 hr.)				
Dynamic Shear		22 °C	Т 215	5000 may	5800
G*•sino, 10 rad/s	s, kPa	25 °C	1 515	5000 max.	4110
	Stiffness, MPa (60 s)	12.00		300 max.	143
Dan din a Daam	m-value		T 212	0.300 min.	0.334
Stiffness, MPa (60 s)		10.00	1 313	300 max.	279
	-18 °C		0.300 min.	0.288	
AASHTO M 320	Superpave Binder Grade, PC	д :			76-22

 Table 4-2 Performance grading of the recovered binder



Figure 4-2 Softening curves of rejuvenators mixed with recovered RAP

Mixture	Rejuvenator % (TWM)	High Temperature PG
SR-15 RAP	0	78.2 °C
CWE	1	68.8 °C
HPE	0.5	68.8 °C
Control I	0	67.3
Control II	0	67.0 "Design value"
	0	(expected to be 70.0 after plant mixing)

Table 4-3 Rejuvenator percentage and high temperature grade of samples

Specimen Preparation for Overlay Tests

For each test cell, three replicate slabs were made to obtain the target air voids. The method recommended for the overlay test (Tex 248 F) calls for casting gyratory compacted pills, and then saw cutting its core to obtain the specimens for testing. Since some specimens were aged and weathered in the APWS for a long time before running the overlay test, the project team decided that saw cut specimens would have exposed aggregate without a protective asphalt film, and would therefore exhibit exaggerated and unrealistic aging. The project team decided to use an alternative method to prepare specimens that are not saw cut, but rather, cast in the manner described below.

- The Gmm for the control and the rejuvenated mixes was obtained.
- Weight of the slab was obtained by calculating area of the slab × height × Gmm × 0.93 (7% air voids). Area of slab: 260 mm × 460 mm. Sample height: 51.7 mm.
- A correction factor is applied based on experience (1.01).
- The 260 mm × 460 mm slabs were compacted to a fixed height of 51.7 mm, targeting a 7% air void content. The compaction effort was generated by means of a kneading roller from the compactor.
- Specimens were trimmed to meet Tex 248-F test method.

Table 4-4 displays the properties of the mixtures.

		Sample							
Property	Test Method	Cont	rol II	Control I	CWE	UDE			
		SP-9.5	FC-9.5	Control I	CWE	ΠFL			
Asphalt Content, %	AASHTO T 308	6.5	7.5	6.3	6.3	6.3			
Maximum Specific Gravity	ASTM D 2041	2.362	2.359	2.375	2.361	2.368			
Air Voids %	ASTM D 3203	4.36	4.58	5.8	2.8	2.6			

Table 4-4 Properties of Mixtures

4.2.3 Testing Procedures

The Texas Overlay Test was performed on at least three replicates of each sample to compare their cracking resistance before any aging occurred. Tests were done in accordance with the Tex 248-F specifications [42].

Then, rejuvenated samples and the Control I specimens underwent APWS aging. Three replicates of CWE and HPE mixes, as well as two replicates of the Control I group were aged in the APWS for 1,000 hours; the remaining specimens were aged for 3,000 hours. A 3,000-hour APWS exposure can simulate aging that occurs in the field in 7 to 10 years [43]. The Texas Overlay Test was also performed on all aged samples.

4.3 **Results and Discussions**

Table 4-4 displays the results from the Texas Overlay Test before APWS aging. At the initial stage, significant difference was observed in the cracking resistance of samples made with new and rejuvenated asphalt. The average number of cycles to failure was considered an indication of susceptibility of mixtures to fatigue and reflective cracking. Both rejuvenated samples performed much better than both control samples, which were made with virgin asphalt. These observations show that RAP binder can even enhance cracking performance of the pavement if be rejuvenated appropriately. Table 4-5 presents the results at different aging levels and Figure 6 shows the variations of Texas Overlay Test results with APWS aging time.

The results show that the average number of cycles to failure decreases with increased APWS aging time. This trend confirms that weathering of the pavement makes it more susceptible to cracking. The rate of decrease in the number of cycles to failure with APWS time was considerably faster for rejuvenated mixes than for the control I. This means that cracking susceptibility, which is an indication of aging, increased significantly faster in mixes containing rejuvenated asphalt than in those made with virgin binder. However, even at the end of 3,000 hours, rejuvenated samples had an equally good or better resistance to cracking when compared with un-aged control samples. It can be concluded that although rejuvenated samples can have an overall better cracking performance, they might lose their resistance faster than virgin asphalt mixes. This trend needs further investigation with more samples and longer aging time.

Mintuno	Donligato	Test	Air	Starting	Final Load,	Decline in	Cycles to	Average	
wiixture	Replicate	Method	Voids,%	Load, kN	kN	Load, %	Failure	Cycles	
	1		7.2	2.185	0.153	93	55		
Control I	2		8.9	1.724	0.117	93.2	72	71	
	3		7.6	2.325	0.159	93.2	86		
Control	1 (SP)		4.5	4.230	0.282	93.3	104		
Control	2 (SP)		4.2	0.155	0.008	94.7	62	63	
11	3 (FC)	Toy 248 E	4.6	2.582	0.175	93.2	24		
	1	10x 240-r	4.1	1.653	0.112	93.2	384		
HPE	2		4.7	1.759	0.12	93.2	145	239	
	3		4.9	1.797	0.119	93.4	189		
	1		3.9	1.576	1.109	93.1	347		
CWE	2		3.6	1.742	0.118	93.2	144	267	
	3		3.7	1.707	0.118	93.1	310		

Table 4-4 Texas Overlay Test Results before APWS Aging

It should be noted that there was some significant variability between the Overlay Test results from similar replicates. A large number of replicates are needed to achieve an acceptable reliability. Testing such a large number of specimens was not practical due to material and budget limitations. In addition, the air voids of the control were higher than those of rejuvenated mixes. In order to confirm that the higher cracking susceptibility of the control mix was not due to increased air voids, Control II specimens were tested at an air-voids level similar to recycled mixes prior to aging. The Overlay Test results did not change and confirmed the low crack resistance values obtained in the Control I specimens.

CWE samples yielded better cracking resistance than HPE samples. The average number of cycles to failure of CWE samples were 10% to 27% higher than HPE samples. It is noted that CWE is a naphthenic crude emulsion and HPE is a heavy paraffinic distilled solvent extract. The rate of drop in the average number of cycles to failure with aging time was almost similar for the two rejuvenated mixtures.

Mixture	Replicate	Test Method	Air Voids,%	Starting Load, kN	Final Load, kN	Decline in Load, %	Cycles to Failure	Average Cycles		
0 Hours										
	1		7.2	2.185	0.153	93	55			
Control I	2		8.9	1.724	0.117	93.2	72	71		
	3		7.6	2.325	0.159	93.2	86			
	1		4.1	1.653	0.112	93.2	384			
HPE	2	Tex 248-F	4.7	1.759	0.12	93.2	145	239		
	3		4.9	1.797	0.119	93.4	189			
	1		3.9	1.576	1.109	93.1	347			
CWE	2		3.6	1.742	0.118	93.2	144	267		
	3		3.7	1.707	0.118	93.1	310			
1000 Hours										
Control I	1		9	2.435	0.167	93.1	36	59		
Control I	2		8.6	2.438	0.168	93.1	79	50		
	1		5.9	2.213	0.151	93.2	186			
HPE	2	Toy 248 E	5	2.135	0.147	93.1	98	186		
	3	1ех 240-г	4.7	2.386	0.167	93	275			
	1		4.9	2.53	0.174	93.1	153			
CWE	2		4.2	2.721	0.19	93	256	253		
	3		5	2.526	0.174	93.1	349			
3000 Hours										
нрб	1		4.9	2.987	0.23	93.2	75	71		
IIIE	2	2 Tox 248 E	6.4	2.55	0.17	93.4	66	/1		
CWF	1 Iex 248	1 СЛ 240-Г	5.8	2.927	0.199	93.2	58	08		
CWE	2		6.8	2.663	0.18	93.3	137	70		

Table 4-5 Texas Overlay Test Results before and after APWS Aging



Figure 4-3 Variations of average number of Texas Overlay cycles to failure with APWS aging time

4.4 **Summary**

The cracking susceptibility of rejuvenated asphalt mixes was compared with that of virgin asphalt mixes using the Texas Overlay Test. The following observations were made:

- When rejuvenated properly, recycled asphalt mixes can be more resistant to fatigue and reflective cracking than virgin asphalt mixes.
- The resistance of recycled pavement to cracking might decrease faster over during pavement's life cycle when compared with the new asphalt. However, according to this experience, even after 7-10 years in service, properly recycled asphalt can have a better cracking resistance than new asphalt.
- The crack resistance of recycled mixes is dependent on the rejuvenator used. In this experiment, the CWE rejuvenated mix performed better than the HPE with a larger number of cycles to failure. The CWE rejuvenated samples had 10% to 27% more load cycles to failure, compared to HPE rejuvenated samples. Both mixes, however, exhibited a very similar rate of crack resistance reduction with aging.

4.5 Limitations

It should be noted that there are several factors that limit the generalization of the trends observed above. We encourage others to investigate and confirm these trends. These include:

- 1. The Texas Overlay Test variability,
- 2. The variability of the air voids between the control and recycled samples, and
- 3. The relatively small size of this experiment.

CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS

5.1 Summary and Conclusions

The durability of recycled asphalt pavement was investigated by studying the long-term aging of recycled binders and cracking resistance of recycled asphalt mixes over time. Eleven rejuvenators were nominated, and the five best were selected for binder testing. The two that caused the slowest aging of the binder were selected for mix tests. The conclusions are listed below:

- Eleven rejuvenators were tested for softening power, flash point, RTFO mass loss and physical properties, and were ranked accordingly. The five best were selected for binder testing, namely BOF, PND, CWE, HPE and APO.
- There is a significant difference between various long-term aging rates of samples rejuvenated by different recycling agents. Compared to the aging rate of the virgin binder, two rejuvenators out of five (CWE and HPE) slowed down aging, while three others (PND, BOF and APO) accelerated it.
- In contrast to the original binder, it seems that aging of rejuvenated asphalt does not slow down over time. While the aging rate of original asphalt (in terms of high temperature grade rise for each hour of PAV aging) was observed to decrease with aging time, it remained almost constant for rejuvenated binders.
- Even when the aging behavior of rejuvenated binders was similar to that of original binders in the performance grade system, as determined by the first 20 hours of PAV aging, some had a faster long-term aging. For instance, binders rejuvenated with PND and BOF had high temperature grades very close to those of the original binders after the first 20 hours. However, their PAV failure times were 6 to 9 hours less than that of the original binders. This observation confirms the importance of studying long-term aging beyond performance grade system requirements.
- The service life of recycled asphalt is highly dependent on the rejuvenator. Selecting the proper rejuvenator was observed to increase the service life up to 9 years, as compared to rejuvenating with a less effective product. This observation does not explain early premature failures of HIR mixes. It is believed that early premature failures may be caused by improper HIR application such as insufficient mixing, not enough or too much rejuvenator applied, rejuvenator contamination with water or other foreign agents, and rejuvenator heat damage. Also, factors during the service life, such as rejuvenator susceptibility to water damage, can cause premature failures.
- BOF, which is a bio-rejuvenator, caused the fastest aging. Also, samples that rejuvenated the other bio-based rejuvenator, APO, experienced fast aging during the last 20 hours. These observations show that bio-based rejuvenators may cause fast aging, especially in latter stages of the pavement life.
- Low temperature creep stiffness of rejuvenated binders is significantly lower than that of the original binder. Stress relaxation (BBR m-value) was the parameter that controlled the low temperature grade of rejuvenated asphalt. Similar to that observed in high temperature grading, samples rejuvenated by CWE and HPE showed lower low temperature grades, while those rejuvenating by PND and BOF did not improve low temperature aging rates, and in some cases, worsened it. APO samples were very soft at bottom temperatures. They had a

very low bottom temperature PG after 20 hours, but that value increased rapidly from 20 hours to 60 hours.

- When rejuvenated properly, recycled asphalt mixes can be more resistant to fatigue and reflective cracking than virgin asphalt mixes.
- The resistance of recycled pavements to cracking might decrease faster over the pavement's life cycle, compared with the new asphalt. However, according to this experience, even after 7-10 years in service, properly recycled asphalt can have a better cracking resistance than new asphalt.
- The crack resistance of recycled mixes is dependent on the rejuvenator used. In this experiment, the CWE rejuvenated mix performed better than the HPE with a larger number of cycles to failure. The CWE rejuvenated samples had 10% to 27% more load cycles to failure, compared to HPE rejuvenated samples. Both mixes, however, exhibited a very similar rate of crack resistance reduction with aging.

5.2 Suggestions for Modifying Specifications

In this section, the findings obtained in this study were implemented to propose procedures to prevent premature aging of the recycled binder. These procedures might be used to modify specifications.

The Superpave PG system fully characterizes asphalt binders for different environmental and climatic conditions. Therefore, to ensure proper performance of the recycled asphalt binder, the PG system is preferred over penetration and viscosity requirements which are empirical measures at a single temperature and condition. The following steps are proposed to determine the proper dose of rejuvenator for the HIR process:

- Recover the asphalt binder in the existing pavement and determine the high, intermediate and low critical PG temperatures.
- Create blending charts according to high temperature PG requirements and determine the maximum dose needed in order to meet the PG requirements at the targeted high temperature.
- Select a rejuvenator dose not higher than the maximum dose determined in the previous step, and test the rejuvenated asphalt blend to make sure it meets the intermediate and low temperature requirements.

Based on the outcomes from the binder testing, as described in the Chapter 3, the standard PG system is not necessarily sufficient to ensure proper long-term durability of recycled binders. After 20 hours of PAV aging, which is the time specified in AASHTO R 28, the aging behavior is different for virgin and recycled binders. The aging rate of virgin binders usually drops significantly after the first 20 hours, yet this does not happen for recycled binders. This difference can be seen in Figure 5-1, which shows the aging curves presented in Chapter 3. The specifications for recycled binders need some modifications to ensure durability of recycled asphalt binders. In sections 5.2.1 through 5.2.3, we propose three alternatives for such modifications.



Figure 5-1 Aging of Recycled Binders - Binder1

5.2.1 Alternative 1: Limiting High and Low PG Grade Increase by Additional PAV Aging

An extended aging test can be performed to ensure durability. Table 5-1 shows an estimation of the increase in the top and bottom temperature PG that occurs due to different aging times for virgin binders, based on the results of the binder testing. It should be noted that for the bottom temperature PG, only the increase that occurs from standard aging for the BBR test (RTFO+20 hours PAV) to 40 hours is considered. Since the recycled asphalt should not be allowed to age faster than virgin asphalt, limits should be specified for the increase in PG measurements after 40 hours of PAV aging.

Aging	PG Measurement	Increase for Binder 1 (°C)	Increase for Binder 2 (°C)	Proposed allowable increase (°C)
40 Hours	High Temperature PG	21.63	21.03	22
40 11001 5	Low Temperature PG	1.92	1.04	2

Table 5-1 Increase in top and bottom PG after 40 hours of PAV aging

Therefore, the following procedure is proposed for the durability test:

The true grade of the recycled asphalt is determined. Then is aged in the PAV for 40 hours at a temperature of 100 °C. Afterward, the following requirements should be met:

- The critical high temperature PG (G*/Sin $\delta \ge 1.0$) should not be increased more than 22 °C.
- The critical low temperature PG (S < 300 MPa & m-value > 0.300) should not be increased more than 2 °C.

5.2.2 Alternative 2: Limiting Aging Rate

Another way to ensure durability is by limiting the aging rate. Table 5-2 shows aging rates of virgin and recycled asphalt samples observed in the binder testing. It can be seen that the decreases in aging rates after the first 20 hours of aging is significantly higher for virgin asphalts. Therefore, for recycled binders, in order to prevent fast aging rates throughout the entire lifetime of the

pavement, a slower aging process is needed during the first 20 hours of aging. The average aging rate of virgin binders from 0 to 60 hours is approximately 0.5 °C/hr. If it is conservatively assumed that recycled binders have a constant aging rate, the value of 0.5 °C/hr can be considered a limit for that rate. Figure 5-2 shows three different schematic aging curves that are labeled by their aging rates. The first curve has an aging rate of 0.7 °C/hr in the first 20 hours of aging, and 0.4 °C/hr in the next 20 hours of aging. This is similar to the observations made in the virgin binders. The two others have constant rates of 0.50 and 0.56 °C/hr.

	Aging Phase	Hardening Rate based on High Temperature Performance Grade ($^{ m C}$ /hr)											
		Binder 1					Binder 2						
		Original	CWE	HPE	PND	BOF	APO	Original	CWE	HPE	PND	BOF	APO
	0 - 20 Hours	0.69	0.45	0.49	0.60	0.56	0.53	0.69	0.42	0.53	0.73	0.67	0.55
	20-60 Hours	0.36	0.39	0.36	0.46	0.53	0.48	0.40	0.39	0.29	0.43	0.62	0.50
	0-60 Hours	0.47	0.41	0.41	0.51	0.54	0.51	0.55	0.41	0.37	0.53	0.64	0.51

 Table 5-2 Aging Rates of Original and Rejuvenated Samples

The desirable side of recycled binders is that due to their slower aging rates at the early stages of a pavement's life, perhaps less damage will occur earlier in the pavement's life compared to pavements with virgin binders. Although the "0.5" and "0.7 - 0.4" curves in Figure 5 reach the same point after 60 hours, the "0.5" curve is softer before this point. Therefore a recycled asphalt with such an aging curve experiences less cumulative damage during the life of the pavement. It can be estimated that a binder with a slightly higher aging rate, like the 0.56 curve in Figure 6, causes damage almost similar to that of the virgin binder.

Based on these estimations, a maximum aging rate of $0.56 \, {}^{\circ}C/hr$. is recommended. This corresponds to a 20 ${}^{\circ}C$ increase in high temperature grade after standard aging (RTFO +20 hour PAV). Therefore, a maximum raise in high temperature of 20 ${}^{\circ}C$ after standard aging is recommended.



Figure 5-2 Schematic aging behavior of virgin and recycled binders

5.2.3 Alternative 3: Increasing PAV Aging Time

Based on the data presented in the Table 7, 20 hours of PAV causes almost 14 ^oC increase in the high temperature grade of virgin asphalts. As mentioned earlier, a constant aging rate of 0.56 ^oC/hr. causes aging related damage similar to virgin binders tested in the Task 3 (same area under the curve). With this rate, 25 hours of PAV aging is needed to reach a 14 ^oC increase in the high temperature. Therefore, it can be estimated that if a recycled binder is PAV aged for 25 hours it will have a similar grade to a virgin binder aged for 20 hours and will experience almost the same aging related damage as a virgin binder in the long-term.

It is proposed to require a recycled binder to be PAV aged for 25 hours instead of 20 hours and still meet the PG requirements. The rationale behind this approach is the fact that virgin asphalt typically ages faster in the first 20 hours than the rejuvenated binder, then it ages slower. In this approach we are looking for the number of aging hours for the rejuvenated binder that would yield the same high temperature grade. From Figure 5.2, assuming the green line represents the line which yields same cumulative aging rate as the original binder (same area under the curve) and has a slope of 0.56 $^{\circ}$ C/hour. Drawing a horizontal line at the top temperature grade of the virgin binder at 20 hours intersects the green line at 25 hours. This represents the PAV aging time that would result in same top temperature grade as the virgin binder.

5.3 Summary of Suggested Procedures

FDOT's current specifications do not address recycling agents. Based on the research conducted in this project, any specification developed for recycled asphalt would need to account for longterm aging. To ensure proper longevity of recycled binders it is necessary that they do not age faster than virgin binders in the long-term. For this purpose, three alternative procedures have been proposed:

- 1. **Durability Test:** Age recycled asphalt for 40 hours in PAV; the increase in high and low temperature grade should not exceed 22 ^oC and 2 ^oC, respectively.
- 2. Limiting Aging Rate: Limit the aging rate to 0.56 ^oC/hr. This is corresponding to 20 ^oC increase in the high temperature grade after the standard aging procedure (RTFO+ 20 hour PAV)
- 3. **Extending Aging Time:** The PAV aging time should be increased from 20 to 25 hours for recycled binders and meet the PG requirements. The aging rate for a recycled binder should not exceed that of a virgin binder that is PAV aged for 20 hours.

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APPENDICES

Appendix A: Asphalt Pavement Weathering System (APWS)

Asphalt Pavement Weathering System (APWS)

Attributes:

- Designed to accommodate "full depth" pavement specimens (25 ft.² max. area)
- Simulates natural pavement wathering top→down
- Provides accelerated weathering exposure
- Fully monitored and recorded for temperatures, RH and rain
- Controlleable Cycles
 - o Rain
 - Sunlight (UV Exposure)
 - Temperature
- Modeled after ASTM International Standards
- Designed as a product development/improvement tool
- Designed for pavement product research and development
- Designed to assess pavement performance
- Provides cores or slabs of sufficient size for complete evaluations after weathering

Materials, Pavements and Systems Evaluations:

- Conventional HMA, OGFC & SMA
- WARM Mix Pavement Technologies
- PMA Binders evaluated in-situ
- GTR & Hybrid Binder evaluated in-situ
- Evaluation of Maintenance & Preservation
 - Rejuvenator/Sealer
 - Slurry Seals & Micro-Surfacing
 - Pavement Sealers
 - Thin lift overlays
- Evaluation of new binder modifiers and additives in-situ
- Study of Binders Aggregate interaction(s)
- Pavement Performance
 - Fatigue Crack Resistance
 - Lift Cycle Cost Analysis
 - Water Sensitivity
 - WARM Mix Technologies
 - o Mixes w/RAP
 - Mixes w/GTR
 - Permeability
 - Associated Products
 - Crack & Loop Sealants
 - Stripping Paints





Asphalt Pavement Weathering System (APWS)

Accelerated Weathering with Temperature Flexibility

- Chamber 50-160°F
- Specimen Surface 50-180°F

Water

0 – 1.0+ inches per time interval

Cycles: typical combination

- Sunlight only
- Sunlight + Rain
- Rain Only
- Dark Only
- Dark + Rain
- Sunlight/Dark + Rain
- Operation

• 24/7

Specimen Exposure Elapsed Time

- Per evaluation design
 - typical 1,000 5,000 hours

Specimen Size

- Up to 25 ft.² x 12" thickness
- Any shape (cores, slabs)

Special Conditioning Available

- Leachate (Water) Collection
- Water Options (fresh, salt, deionized)
- Varied specimen mounting

 partially sub-merged
- Companion Sample Outdoor Weathering
- 10 Channel Temperature Monitoring & Recording
- Water Spray System Flexibility

 mist to 1.0+"/hour
- Chamber Partitioning (water only)
- Permeability (rate determinations)

Technical: The APWS uses lamps containing quartz discharge tubes with tungsten filaments which provide a mix of radiation similar to natural sunlight (UVA, UVB, visible and infrared radiation). The special glass of the bulb also filters out the portion of the generated radiation which is not contained in natural sunlight.

	The Concept	Top→Down	Weathering
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The ability to slice pavement specimens into layers for composition and performance analysis before and after accelerated weathering.

Appendix B : Mix Design of the Control Samples (Chapter 4)

B-1 Mix Design for Control I

Rework Pavement Recycling SR-15 Mix Design

Road Surface Recycling Inc. (RSR) follows a standard process to control pavement recycling and establish quality assurance targets. The process includes the following activities:

- 1. Obtain cores
- 2. Analyze the properties of the existing mix
- 3. Establish the asphalt softening curve
- 4. Batch a trial mix and establish QC targets

The following sections describe the process and results for FDOT SR-15.

Road Section History

This roadway was last resurfaced in 2004.

This section of road has been overlaid many times due to poor sub-grade conditions.

Coring Data

On June 10, 2013, 6-inch cores were obtained from various locations along SR-15 within the job limits. The thickness of all bituminous layers ranged between 4 and over 10 inches, placed in multiple lifts. All the lifts appear to be a limestone product. The top 3.0 inches of the cores had a maximum aggregate size of 9.5 mm to 12.5 mm. There are some areas of moderate cracking; however, the majority of the roadway is heavily cracked with extensive distressed areas and deep rutted areas. Cores showed that the top lift is approximately 1" to 1.5" in thickness. The second lift is approximately 1.5 inches, and the third lift varies in thickness but appears to have similar properties to the second lift. The layer below is not identified. Cores were taken to supplement the amount of material needed for testing.

Laboratory Testing

The cores were transported to the RSR Lab where the top first inch was removed. Testing was performed on the next 2 inches that were trimmed from the cores. Tests include AC content, gradation, air voids and penetration. The following Table 4B-1 summarizes the existing asphalt mix test results.

Property	Value
Nominal Maximum Aggregate Size	9.5mm
Rock Type	Limestone
Asphalt Content (%)	6.3
Lab Air Voids (%)	6.1
Penetration (dmm)	22

Table 4B-1. In-place properties of the Existing Mix

After the removal of the first top inch, the next two inches of the cores were heated and trimmed from the cores. The loose mix sample created from the cores was used for the mix design. Table 4B-2 lists all of the test results performed, including gradation, AC content, lab air voids, and binder penetration. The first column shows properties of the existing mix. The following two columns are for two trial batches.

To determine the amount of rejuvenator and screenings needed, two trial batches were prepared. One sample contains 4% MNE Rejuvenator and 3% Screenings (Table 5 code F20 screenings). The second sample contains 6% MNE Rejuvenator and 6% screenings. The objective is to achieve rejuvenation (Penn near 50 dmm) and lab air voids in the 3.5% to 4.0% range. It is noted that these two objectives are conflicting. Prior to adding the Rejuvenating oil, the air voids were in the 6% range. After adding the Rejuvenating oil, air voids dropped to 1.3%. Screening helped increase the air voids to 3.5%.

Our past experience is that the mix design may require modification after the first day of production to account for the variability in the in-place material and the fact that the lab design does not perfectly simulate the heating, milling and mixing in the field. In the first day of production, RSR will vary the level of sand, rejuvenating oil, and temperature to balance binder rejuvenation and mix air voids. Furthermore, aggregate degradation (in the 2% range) in the milling process may change amount of screenings needed.

VMA was determined for all trial batches and was in the 13-16% range, which exceeded the minimum requirement.

	Existing Mix	Sample #1 4% Rejuv + 3% Screenings	Sample #2 6% Rejuv + 6% Screenings	SP 9.5 Gradation Requirements
% Rejuvenator	0	4	6	
% Screenings	0	3	6	
1"	100	100	100	100
3/4''	100	100	100	100
1/2''	99.8	100	99.7	100
3/8''	95.5	97.9	95.2	90-100
#4	70.3	72.6	72.3	<90
#8	54.7	56.5	55.9	32-67
#16	45.9	46.6	46.2	
#30	40.6	40.1	40.0	
#50	30.6	29.4	29.7	
#100	15.4	14.3	14.9	
#200	7.4	6.3	7.0	210
Air Voids	6.1	3.52	2.51	
Pen (dmm)	23	45	54	
AC	6.48	6.43	6.65	
Gmm	2.368	2.387	2.391	
Gmb	2.225	2.303	2.331	

Table 4B-2 Test Results

AC Softening Curve

The extracted asphalt was tested for penetration in accordance with AASHTO T-49. The softening curve shown in Table 4B-3 was established for this rejuvenator from testing in recent similar projects, where several quantities of rejuvenator were added and the resulting penetration was determined. Table 4B-3 shows that the in-place asphalt has a penetration of 22 dmm. In addition, it shows that adding 4% rejuvenator increased the penetration values from 22 to 46. This is consistent with the test results in Table 4B-2. This also meets the requirements of 40-80, as specified in FDOT Specification, Section 324. The rejuvenating agent that was used is KENDEX-MNE, as shown in Table 4B-4.


 Table 4B-3 Binder Softening Curve

	B-2 Mix	Design	for	Control	Π
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					S	tate Of	Florida	Departm	nent C	Of Tra	nspo	rtation	- Asph	alt Pla	ant	Worksh	eet				6 M	75-030-2		
	Fin, Pro	i ID:			C	ontracto	c.		Lot Begin Date: Mix De										sion No : 05-39384					
	Mix T	ype: F(2-9.5			Plant No						Traff	ic Leve	I: C			Ch	eck E	v:	BB				
			Note	26	Erasutes	are not all	owed. Mesta	kes shall ha	shall have a single line through the original data with the correct entry written close to it . In initialed and dated. Line undeted forms when they become a writeble															
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ea							Date		-		-		-	Jub	-	101	00	~	t	101	00			
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						T	ons	-	5				-						+					
					В	asket w	t. gl(A	3	084	5	T	<u> </u>						1	t			1		
			Bgn.	. Baske	t + Sa	mple v	rt., g (B	4	721.	6	1								ŀ					
			B	gn. San	nple v	vt., g, (B-A)	1	637.	1	1								ľ					
E			End	Basket	t + Sa	mple w	rt., g (C	4	596.	7	1								Ī					
nte			Fi	inal San	nple v	rt. g. (C-A) (D	1	512.	2		-				_			ſ					
S				7.65]			-					[
alt	(Calibra] .	-0.13	3									ſ										
spł	Des.	AC, %		7.52		1 Š				÷.				ž[<u>ج</u>							
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			N	/t. of Wa	ashec	Samp	le, g (H	1.	478.9	9	15				윈			lo				2		
	Wt	of - 75	um Mať	I lost due	e to W	ashing,	g. (G-H)		33.1	-	A				P			4	ž I		-	Pe		
	Sieve T						arget	WtR	Ret	% Pa	SS	Wt F	let 9	% Pa	SS	WtR	et %	Pas	s	Wt Re	1 %	Pass		
	50	Was 100	1"	(25.0mm)), g	-	100		100	-		-	100			10	0	ŀ		10	0			
	99	200	3/4	(19.0mm), g			100	<u> </u>	-	100			-	100			10	0	ŀ		10	10		
	100	np 66	2/9	(12.5mm)	(2.5mm), g	1.5mm), g	2.5mm), g		00	10.	-	00.70				-				-	ŀ		+-	_
S	0.41	los:	No.4	(9.5mm), g		-	60	19.	6	72.09	-	-					_	-	ŀ		+	-		
lati	d Ag	P.M	No.8	(2.36mm)	2.36mm), g		48	600	0	53 71	4					-	-	-	ŀ		+	-		
irac	Ext'o	N) (ue	No.16	(1.18mm)	, g a	-	39	883	1	41 59	B	-	-	-				-	ŀ		-	-		
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	s Pa	Wr	No.100) (150µm)), g		6	1370	.3	9.37	- Be			_	me		1		e i		+	- e		
	6 8		No.20	0 (75µm)	75µm), g		4	1467	.2	2.93	12				휟				ēľ		1	- 년		
	Wt of Matl. in Pan, g						11.2	2		Pe				Pe	100001000			2			Pe			
	Performed By:								BE	3			BB						T					
																Τ								
	Weigh	nt of Fla	ask + S	Sample	2101	.3	2057	.9																
	Weight of Flask								.8	1022	2.6			_				_						
ε	Weigh	nt of Sa	mple				(A)	1023	3.5	1035	5.3				_						_			
B	Weight of Flask + Water (D)							3363	.1	3421	.0		-		_	_	_		+		-			
	Weight of Flask + Water + Sample (E)								.1	4017	.9		_		_				+		_			
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	Con	r Facto				Averan	e Gmm	-	2.34	50	-				-				+			-		
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