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## FLORIDA INTERNATIONAL UNIVERSITY

Miami, Florida

## EVALUATION OF DURABILITY AND HOMOGENEITY OF REJUVENATED ASPHALT BINDERS

A dissertation submitted in partial fulfillment of the

requirements for the degree of

## DOCTOR OF PHILOSOPHY

in

## CIVIL ENGINEERING

by

Mojtaba Mohammadafzali

2017

## To: Interim Dean Ranu Jung College of Engineering and Computing

This dissertation, written by Mojtaba Mohammadafzali, and entitled Evaluation of Durability and Homogeneity of Rejuvenated Asphalt Binders, having been approved in respect to style and intellectual content, is referred to you for judgment.

We have read this dissertation and recommend that it be approved.

Atorod Azizinamini

Michael Bienvenu

Mohammed Hadi

Irtishad Ahmad

Khaled Sobhan

Hesham Ali, Major Professor

Date of Defense: March 29, 2017

The dissertation of Mojtaba Mohammadafzali is approved.

Interim Dean Ranu Jung College of Engineering and Computing

Andrés G. Gil Vice President for Research and Economic Development and Dean of the University Graduate School

Florida International University, 2017

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#### ACKNOWLEDGMENTS

I would like to express my deepest gratitude to my major professor, Dr. Hesham Ali, for his invaluable support throughout my Ph.D. studies. Without Dr. Ali's enthusiastic and critical guidance, and his fatherly support and encouragement, this dream would never come true. Apart from mentoring to sharpen my technical abilities, he was equally influential in development of my personality as an engineer and scholar. I also would also like to thank my dissertation committee members, Dr. Atorod Azizinamini, Dr. Michael Bienvenue, and Dr. Mohammed Hadi, Dr. Irtishad Ahmad, and Dr. Khaled Sobhan, for their sound advice, constructive comments and kind support. I am very thankful to Florida Department of Transportation State Material Office for providing the financial support for this project. I also appreciate the valuable technical input from Mr. Greg Sholar, Mr. Wayne A. Rilko, and Mr. Jim Mussalman. I would like to thank the administrative staff at Florida International University Department of Civil and Environmental Engineering for helping out with the departmental and university policies and procedures.

This dissertation in dedicated to my family. I would like to thank my beloved parents, Akbar and Monir, my wife, Elaheh, my brothers, Ali and Soroosh, and my sisters-inlaw, Mahsa and Ladan, for their unconditional love, endless support, and inspiration. Lastly, I would like to thank my intimate friends for their sincere camaraderie and heartwarming encouragement.

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## ABSTRACT OF THE DISSERTATION EVALUATION OF DURABILITY AND HOMOGENEITY OF REJUVENATED ASPHALT BINDERS

by

Mojtaba Mohammadafzali

Florida International University, 2017

Miami, Florida

Professor Hesham Ali, Major Professor

Despite the widespread recycling of Reclaimed Asphalt Pavement (RAP), a large portion of it is still wasted. One of the main reasons is the concern with the performance of high RAP mixtures. Asphalt binder aging and subsequent rejuvenation is one source of uncertainty. Rejuvenators are frequently added to high RAP mixes to enhance the properties of the binder. This enhancement is often perceived as simply lowering the viscosity. Two important parameters that are not adequately addressed by existing methods are durability and homogeneity of the recycled binder. This research investigated these two concerns and provided quantitative indicators to measure them.

The durability of rejuvenated binders was investigated through studying their longterm aging. Superpave PG tests and aging procedures were used for this purpose. Results indicated that the type and dosage of the rejuvenator has a significant impact on the aging of a rejuvenated binder. While using a proper rejuvenator can prolong the life of the binder, choosing a wrong product causes the binder to age significantly faster.

The asphalt film that coats aggregates is not necessarily homogeneous. Different layers of the film are affected differently by aging and rejuvenation processes.

A staged extraction method was implemented to provide representative samples from the different layers of asphalt. The stiffness of each sample was measured by Dynamic Shear Rheometer testing. Results indicated that most of the rejuvenator is absorbed by outer layers immediately after blending. As the mixture ages, the rejuvenator continues diffusing into the inner layers. Outer layers are also affected more intensely by the aging. The coupled effects of aging and rejuvenation make the recycled binder more homogeneous than virgin asphalt. This property can facilitate the use of higher target PG values for recycled binders without compromising the long-term performance.

This research introduces two quantitative measures, critical PAV time and Durability Index, for evaluating the durability of recycled binders and two other parameters, Stiffness Gradient Factor and Homogeneity Index for describing the binder film homogeneity. These indicators and the knowledge obtained from this research make it possible to design and evaluate the binder rejuvenation process in a more effective manner.

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#### **CHAPTER 1: INTRODUCTION**

### 1.1 Background

More than 2,000 lane miles of roads are milled and resurfaced every year in Florida's State Highway System. As a result, over 1.8 tons of Reclaimed Asphalt Pavement (RAP) are produced annually. The RAP is completely reusable and is recognized as America's most recycled material [1]. Recycling of asphalt pavement enhances the sustainable development both financially and environmentally. It has been estimated that using RAP results in saving 14% to 34% in material and construction costs for a RAP content of 20% to 50% [2]. Moreover, the use of RAP material helps preserve the environment by reducing waste material, and at the same time, decreases the consumption of natural resources like aggregate and petroleum. Although the benefits of recycling have been well recognized, there are still some considerable portions of RAP that are wasted or downgraded when used in landfills or non-asphalt applications, such as embankment, sub-base, base, and shoulders. In Florida, the average RAP content of mixes placed on the State Highway System is 29% and is only 19% when modified asphalt binder is used. In total, almost only one quarter of the RAP is recycled to its highest potential—when recycled into a new hot mix.

In order to gain the most value from the RAP material, it is necessary to increase the RAP content in asphalt mixtures used in surface layers. The material with over 25% RAP content is often considered a high RAP mixture [3]. Lack of confidence in high RAP content mixes continues to be among the main obstacles to increased mix recycling. Properties of recycled

pavement material are not necessarily similar to those of virgin material, and currently, there is a shortage of methods to adequately assess the differences. One of the most important sources of difference between recycled and new material is the aging of asphalt binder. When the asphalt ages, portions of its lighter components, known as Maltenes, are lost due to evaporation and oxidation. As a result, the binder becomes hard and brittle, and the mixture becomes more prone to cracking. To achieve a recycled mixture with good performance, it is necessary to rejuvenate the old asphalt by restoring its original properties. For this purpose, recycling agents or rejuvenators are frequently added to the aged asphalt.

#### **1.2 Problem Statement**

The process of rejuvenation is often perceived as softening of the hard asphalt. Therefore, in most cases, specifications rely on target ranges of penetration, viscosity, or Performance Grade (PG) to verify the success of rejuvenation. These criteria do not evaluate all properties of the rejuvenated binder. Two of the most significant properties that are adequately assessed are durability and homogeneity.

If the recycled asphalt binder ages faster than the virgin binder, the recycled pavement will deteriorate faster. The uncertainty of the durability of the recycled pavement would lead to an uncertainty of the benefits of recycling. Currently, there is no established method to ensure the durability of the recycled binder. The long-term aging of recycled asphalt binders might be quite different from that of virgin asphalt. A particular sample of recycled asphalt, which is capable of meeting the expectation at the start of its new life, might age faster than the virgin binder. If this happens, the recycled asphalt pavement will be less durable. Currently, there are no established methods to assess the aging of the rejuvenated

binder and ensure its proper durability. Furthermore, the type of rejuvenator might influence durability. There is no existing procedure to consider this factor for selecting the rejuvenator.

The successful recycling of asphalt pavement depends on the proper mixing of the recycling agent with the old asphalt. Even a very durable binder would not perform well if it is too heterogeneous. The asphalt film that coats aggregates is not necessarily homogeneous. Outer and inner layers might be affected differently by aging and rejuvenation processes. If the blending is not complete, the result might be an asphalt mix with a hard binder in the inner layers and over-softened binder in the outer layers.

An incomplete blending will cause unpredictable and potentially poor performance of the recycled mix. Little, if any, research has studied the effect of aging on different layers of the asphalt film. Although several studies have looked at the process of diffusion of the rejuvenator into the aged asphalt, there are no established criteria to provide a measure of binder homogeneity. Such a measure, if provided, will enable contractors and agencies to evaluate the quality of mixing and provide a basis for establishing quality control criteria.

#### 1.3 Objectives

The objective of this research was to develop and implement new methods for evaluating the effectiveness of asphalt binder rejuvenation. In this dissertation, a successful rejuvenation is defined as a rejuvenation process that enhances the properties of the aged asphalt in a way that its performance is similar to or better than a reference virgin asphalt. A reference virgin asphalt is a source virgin asphalt that is commonly used in the application where the use of the recycled binder is considered. Two critical aspects of the performance of the rejuvenated binder that are not adequately addressed by exiting methods are durability and homogeneity of rejuvenated binders. This study aims to investigate these parameters and propose methods to evaluate them. By shedding light on these two concerns, asphalt binder recycling procedures can be designed and evaluated more effectively. Evaluation methods proposed based on the outcome of this research are potentially a significant contribution to filling the gap in recycled asphalt pavement design and quality control specifications. Specifications that are more effective will result in more reliable recycled asphalt pavement and will enhance the reuse of RAP material.

### 1.4 Methodology

This research focused on the rheological properties of the asphalt binder. Superpave PG tests were used to measure these properties and the high temperature PG value was considered the main indicator of the stiffness of the binder.

A comprehensive literature review was conducted on various aspects of pavement recycling and asphalt rejuvenation. The experimental plan consisted of three major parts, as described in the next sections.

#### 1.4.1 Long-term aging and durability evaluation for 100% recycled binders

The aging of entirely recycled binders was compared with that of virgin binders. The aged binders were produced by artificial aging using rolling thin film oven (RTFO) and Pressure Aging Vessel (PAV). These aging procedures were also used to age test samples. Aging of recycled mixtures was also evaluated using an accelerated aging protocol.

#### 1.4.2 Long-term aging and durability evaluation for partially recycled binders

This experiment was generally similar to the one explained in the previous section, but certain differences existed: First, the recycled samples contained only 20 to 40 percent RAP, rather than being entirely recycled. Secondly, the aged binder was obtained by recovering of the asphalt from RAP mixes. Third, a comparative analysis was conducted after RTFO aging.

#### 1.4.3 Evaluation of rejuvenator blending and binder homogeneity

A staged extraction method was used to separate different layers within the asphalt film that covers aggregates. This method is explained in detail in Chapter 4. The relative properties of these layers were compared for samples with different compositions and aging statuses.

### **1.5** Organization of the Dissertation

This dissertation is comprised of five chapters. Chapter 1 is an introduction that provides the background of the research, explains the problem, and describes the research objective and methodology. Also, a comprehensive literature review is presented in this chapter. Chapter 2 covers the long-term aging experiments for 100% recycled binder and mixtures. The aging study for partially recycled binders is described in Chapter 3. Chapter 4 discusses the rejuvenator blending and binder homogeneity study. The observation and conclusions from this research are summarized in Chapter 5.

#### **1.6 Review of Literature**

#### 1.6.1 Mix Design for Incorporating RAP

The incorporation of RAP makes the mix design more complex. The properties of RAP, including binder content, aggregate gradation, and the extent of aging affect the design of the mix. A poor design that does not adequately address these considerations affects the performance of the mix adversely and decreases the longevity of the pavement. The U.S. Department of Transportation's Federal Highway Administration (FHWA) and the National Cooperative Highway Research Program (NCHRP) have been working for decades to enhance the mix design of RAP contained mixtures and have published several reports, guides, and manuals [3- 6].

Generally, the design of recycled mixtures includes adjusting the aggregate gradation, binder viscosity, and volumetric parameters. The FHWA Pavement Recycling Guidelines for State and Local Governments [4] suggests the procedure presented in Figure 1-1 for designing mixes with RAP. In this guide, the asphalt binder is characterized by the viscosity, and the rejuvenator content is determined by blending charts based on viscosity. With the increasing use of Superpave for the design of asphalt pavement, mixes containing RAP are also increasingly being designed with this method. The NCHRP Report 452 provided a technical manual for the use of RAP in a Superpave mix design [5]. This manual uses the PG to design the blending of the asphalt binder. Figure 1-2 shows the procedure used to determine the grade of the new binder based on the PG system. Equation 1-1 is used in this flowchart, as follows:

$$T_{virgin} = \frac{T_{blend} - (\% RAP \times T_{RAP})}{(1 - \% RAP)}$$
 Equation 1-1

Where:

 $T_{virgin}$  = Critical temperature of the new binder (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<sub>RAP</sub>* = Critical temperature of recovered RAP binder (high, intermediate, or low).

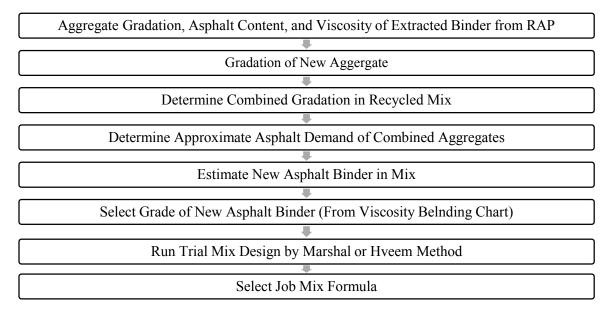


Figure 1-1 Flow Chart for Mix Design of Asphalt Mixtures Containing RAP [4]

In the scenario where the performance grade of the recycling agent is known, the percentage of the old asphalt binder is determined using Equation 1-2:

$$\% RAP = \frac{T_{Blend} - T_{virgin}}{T_{RAP} - T_{virgin}}$$
 Equation 1-2

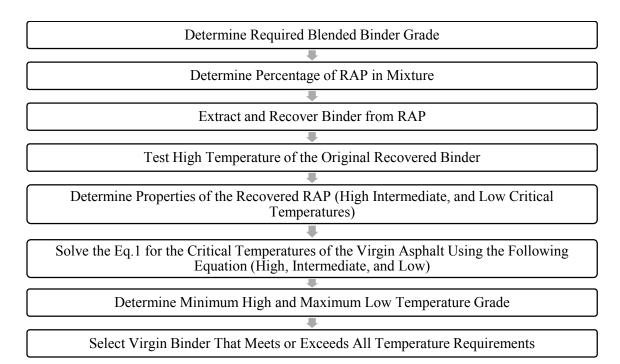


Figure 1-2 The Procedure to Determine the Properties of the New Binder [5]

In addition, ASTM D4887 provides blending charts based on viscosity and performance grade. These charts mainly provide a graphical presentation of the Equations 1-1 and 1-2. A procedure that determines the recycling agent dosage to satisfy PG requirements was proposed by Martins Zaumanis et al. [7]. According to this study, high, intermediate and low PG critical temperatures decrease linearly with an increased dose of the recycling agent. 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 recycling agents usually decreases the PG sum slightly, but this value still remains higher than that of the virgin binder. Based on these results, it was proposed that the following procedures be used to determine the minimum and maximum recycling agent dose:

• The maximum recycling agent dose is determined based on the target high PG temperature.

- The minimum recycling agent dose is determined based on the target low and intermediate PG requirement.
- According to these criteria, a sample of RAP can be rejuvenated by a recycling agent only if the dose required to meet the intermediate and low PG temperatures does not cause the binder fail at temperature requirements. Figure 1-3 shows the minimum recycling agent dose needed to meet the high and low PG temperatures.

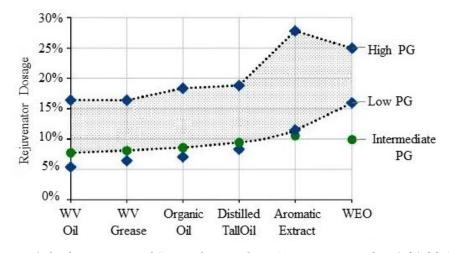


Figure 1-3 The Dosage of Several Recycling Agents to Reach PG64-22 [7]

#### 1.6.2 The Use of RAP in Florida

The Florida Department of Transportation (FDOT) has successfully used asphalt mixtures RAP since the early 1980s. The softer binder used in recycled mixtures has typically been an AC-30 grade material blended with some type of softening agent. The goal is to blend the softer binder (called a recycling agent in Florida) with the RAP binder so that the final blended product has a viscosity of approximately 5,000 poises at the mix design stage. In the past, each mix design was evaluated based on the viscosity of the RAP material and the percent of replacement binder in the mixture. A monograph was then used to determine the viscosity of the recycling agent. According to FDOT Specifications Section 324 [8], during

the production of the mixture, the agency used to monitor its viscosity in an effort to maintain the recovered viscosity in a range of 5,000 to 15,000 poises.

Recently, changes were made in the FDOT Specifications, specifically in Section 334 [9], where the recycling agent is standardized based on the percent of RAP in the mixture. The new RAP levels and permissible virgin binder grades are as follows:

Percent RAP	Asphalt Binder Grade
0 - 15	PG 67-22
16 - 30	PG 58-22
>30	PG 52-28

Table 1-1 Asphalt Binder Grade for Mixes Containing RAP [9]

The binder must meet the requirements of American Association of State Highway and Transportation Officials (AASHTO) M 320, with a few modifications, as outlined in Section 916 of FDOT Specifications [10]. Recycling agents need to meet the requirements listed in Table 1-2. Typically, suppliers start with a PG 67-22 binder and add some type of softening agent to produce a material that meets the specification requirements.

Test	Conditions	Recycling Agent Max/Min Value	
Absolute Viscosity AASHTO T-202	140 ° F	Target Viscosity $\mp 20\%$	
Viscosity Ratio After	Viscosity at 140°F after RTFOT	Maximum 3	
AASHTO T-240	Viscosity at 140°F before RTFOT	iniuminum 5	
Smoke Point FM 5-519	COC	Minimum 260°F	
Flash Point AASHTO T-48	COC	Minimum 400°F	
Solubility AASHTO T-44	In Trichloroethylene	Minimum 99%	

Table 1-2 Standard Specifications for Recycling Agents (FDOT Section 916)

#### **1.6.3 High RAP Content Mixtures**

According to a survey that the FHWA conducted in 2011 [1], 40 state agencies allowed the use of mixtures that contained more than 40% RAP. However, only 11 states reported that such mixtures were commonly used. Evaluation of pavements containing up to 50% RAP in different climate conditions showed good field performance [3]. This confirms that higher percentages of RAP can be used in pavements with good practice and proper mix design.

The NCHRP Report 752 [3] is a comprehensive study that promotes the use of high RAP mixtures by improving their mix design, material management, and evaluation. Some of the important findings from this work include:

- It is more appropriate to define high RAP mixture by the RAP Binder Ratio, which is the proportion of RAP binder to the total binder.
- The exisiting Superpave mix design can be applied to high RAP mixtures, with some minor changes, which are proposed as a modification to AASHTO R35 and AASHTO M323.
- The grade of the new binder shall be determined using a formula similar to Equation 1-1, except that the %RAP is defined by the RAP binder ratio, rather than by the total weight.
- Moisture damage tests should be conducted for mixtures containing RAP.

The National Asphalt Pavement Association published a practical guide for high RAP mixtures, which includes guidelines for material evaluation, mix design, plant verification and quality control [11].

The percentage of RAP can even increase to 100%. Zaumanis et al. conducted a comprehensive research program on 100% RAP mixtures. They considered low temperature properties, Penetration Index (PI) and Penetration Viscosity Number (PVN) as parameters influencing the effectiveness of rejuvenation [12]. It was found that a rejuvenator can effectively improve the low-temperature performance of the mixtures. Rejuvenators can maintain or increase the low-temperature creep compliance and at the same time, increase the indirect tensile strength and fracture energy. A recent study discussed the feasibility of using 100% RAP mixtures by analyzing the recorded performance of such mixtures, along with identification of typical high RAP distresses. The cost analysis showed at least a 50% savings, and an environmental analysis showed a significant reduction in CO<sub>2</sub> production [13].

#### **1.6.4** Asphalt Aging and Rejuvenation

Asphalt aging, hardening, and embrittlement are well documented in the literature. An excellent discussion of mechanisms contributing to aging is presented in [14]. The following is a summary of this discussion:

Asphalt hardening can take place both in a reversible or permanent manner. Reversible changes are referred to as molecular associations like steric effects or wax crystallization. Permanent changes, on the other hand, occur because 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 the 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 bending beam rheometer (BBR) stiffness directly correlates with a measured increase in asphalt density. Using a series of physico-chemical techniques, including Differential Scanning Calorimetry, phase contrast microscopy, and polarized light microscopy, Claudy and co-authors identified the cause of low-temperature physical hardening to be the reversible micro-crystallization of long-chain aliphatic molecules or waxes. As the waxes crystallize, both asphalt density and low-temperature stiffness increase [15].

*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 un-agitated, 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.

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#### 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.

#### 1.6.5 Aging and Durability of Recycled Binders

One important consideration regarding the use of recycled asphalt is the durability of the pavement. The recycled binder is expected to achieve as long a life as that of the virgin binder or longer. An early effort to compare the durability of RAP and virgin asphalt binder was a laboratory study performed by D. Fritchen in 1977 [16]. The moisture damage to asphalt concrete was simulated by a vacuum-submerged conditioning procedure, followed by several freeze-thaw thermal cycles. The performance of the pavement was monitored by non-destructive resilient modulus tests. Results showed that recycled asphalt mixture performed as good as new asphalt samples.

Superpave binder and mix tests were used to compare the performance of rejuvenated and virgin asphalt in another study [17]. The RTFO and PAV were used to simulate short-term and long-term aging, and the DSR and BBR were used to test the asphalt binder

performance. Results showed that the performance of rejuvenated samples was similar to or better than that of virgin asphalt.

In a study by Ohio State University and FHWA, the durability of mixes containing RAP was evaluated [18]. This work aimed to determine 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 contents between 0 and 30 percent. To quantify the durability of HMA, samples were aged through heating in an oven, and the absorbed energy at failure was determined before and after aging. No recycling agent was added to the mixes containing RAP. Results showed higher creep stiffness for samples that contained RAP. Samples containing 30% RAP had the best performance in terms of absorbed energy at failure.

Recycled asphalt was aged during an experiment that was performed in order to investigate the intermingling process between recycling agents and aged asphalt binders [19]. 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 could be concluded that long-term diffusion takes place.

Singh, Zaman and Commuri [20] studied the durability of recycled asphalt by using longterm oven aging of asphalt mixes containing RAP, and by conducting dynamic modulus tests. Two samples were tested in this study: Mix 1 contained PG 64-22, an unmodified binder with 25 percent RAP, and Mix 2 consisted of a Styrene-Butadiene-Styrene modified binder mix and 15 percent RAP. The asphalt content, type, and gradation of the aggregate were the same for both samples. Samples were compacted by a Superpave gyratory compactor and were subjected to long-term oven aging in accordance with AASHTO R 30. Dynamic modulus tests (AASHTO TP 62-07) 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 dynamic modulus due to long-term oven aging. An important finding from this study was that mixes with a 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 [21]. Several samples of asphalt mixtures containing different percentages (25 and 45 percent) 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-

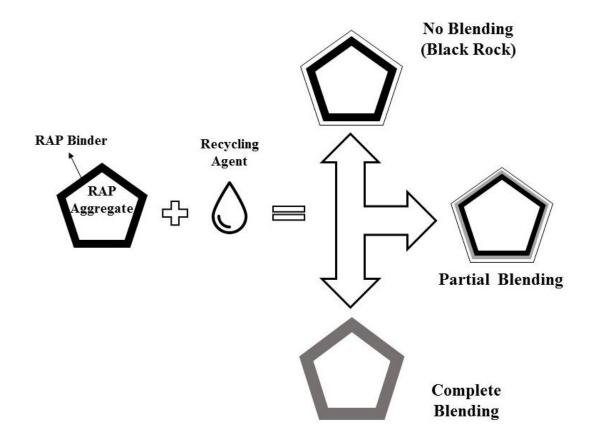
temperature performance grade of the asphalt was determined by DSR and BBR tests. These results showed the following:

- 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 for rejuvenated binders than those for the control binder. It can be concluded that using recycling agents in mixtures containing RAP improves fatigue cracking resistance without adversely affecting rutting resistance.

A study that used RTFO and PAV for simulating the aging and Fourier Transformed Infrared Spectroscopy (FTIR) for evaluating the level of aging showed that generally rejuvenated binder aged faster than virgin binder [22]. Therefore, it was concluded that rejuvenated asphalt has inferior performance, compared to the virgin binder. However, this conclusion is based on tests and samples that used only three rejuevantors. The use of other rejuvenators can lead to completely different results.

#### 1.6.6 Blending and Diffusion of the Rejuvenator

One of the most important concerns of asphalt recycling is the effectiveness of blending between the new binder or recycling agent with the old binder. Generally, when a recycling agent is added to the RAP, at least three scenarios are possible: no blending, partial blending and complete blending [23]. In the no blending scenario, also referred to as "black rock theory," no effective blending occurs between the old and new binder. Therefore, the old binder performs as a part of the aggregate. In the other extreme, in the complete blending scenario, a homogeneous binder is obtained by mixing the old and the new binder. In the partial blending scenario, although some mixing occurs, portions of the old asphalt do not effectively participate in the blending process. Figure 1-4 shows these scenarios schematically.



*Figure 1-4 Different Scenarios for Blending of the Recyclying Agent with the Old Binder* Among the earliest attempts to investigate diffusion of rejuvenators into old binders were the work of L. J. Zearley at the Iowa Department of Transportation [24] and the work of Carpenter and Wolosick [25]. They used a staged extraction method to obtain a

representative sample of different asphalt layers that surround aggregates. In a staged extraction, the mix is first soaked in a solvent for a short time so that only the outer layers of asphalt solved into the solvent. Then, soaking time is increased, and the inner layers are sampled. The solvent soaking times in this illustration are typical times for a three-stage extraction and can be changed based on material and conditions.

The staged extraction method is based on the following understanding of diffusion of the rejuvenator into the old asphalt:

1. The aggregate coated by hard, aged asphalt is surrounded by a very low viscosity layer of recycling agents.

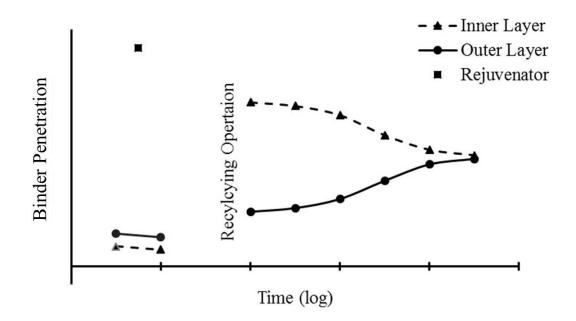
2. The recycling agents starts to penetrate the hard asphalt layers reducing their viscosity.

3. The recycling agents becomes mixed with the hard asphalt, penetrating toward the inner layers. Gradually the viscosity of the outer layers increases and that of the inner layers decreases.

4. In the case of a complete mixing, the equilibrium is approached and the majority of the recycled asphalt's body has almost consistent viscosity. Only the thin layer at the binder/aggregate interface might still have a higher viscosity.

Figure 1-5 shows variations in the stiffness of the inner and outer binder layers. The y-axis represents the binder penetration (a measure of softness). Prior to recycling, the outer layer is harder than the inner layer due to exposure to weathering. When the recycling agent is applied during the recycling operation, the outer layer becomes softer than the inner layer. This is the result of the recycling agent mixing with the outer layers. As time passes, the recycling agent penetrates to the inner layer and the stiffness differential between the layers

is reduced. The authors also concluded that an incomplete mixing could cause problems in predicting the performance of the pavement and affect its long-term field performance.



*Figure 1-5 Variation of stiffness of inner and outer binder layers with time [25]* 

Similar work was done later by Noureldin and Wood [26] and by Van der Kooij and Verburg [27]. The number of extraction stages increased to four in [26], and very hard RAP was tested in the latter work [27].

Fourier transform infrared spectroscopy by attenuated total reflectance (FTIR-ATR) is an effective tool that evaluates levels of diffusion. Karksson and Isacsson [28] used a staged extraction approach and implemented FTIR-ATR to make diffusion measurements. Their work showed that temperature has a significant effect on the rate of diffusion. It was also concluded that the diffusion process can be described using Fick's law.

Various studies on rejuvenator diffusion and the staged extraction methods were conducted at the University of Tennessee by Dr. Baoshan Huang and others (2005-2016). They performed a four-stage extraction and measured the stiffness of different layers at three different temperatures. Their results showed a significant difference between layers [29]. In the next step, they investigated a possible flaw with the stage extraction method: the solvent may dissolve lighter fractions of the asphalt (maltenes) first and the heavier fraction (asphaltene) later. If true, this would invalidate the ability of the staged extraction to separate the asphalt layers. To address this concern, a sample of RAP binder was washed in six stages using four different solvents: Trichloroethylene, Tetrahydrofuran, Toluene and Decalin. FTIR was implemented to characterize layers by determining their Carbonyl Index. Results did not show a notable difference between layers, and the concern of sequential dissolving of asphalt fractions was concluded to be insignificant. This study also showed that Trichloroethylene is an appropriate solvent used for staged extraction [30]. In another study, permeation chromatography, was used in addition to FTIR, to evaluate the effectiveness of rejuvenator blending. Both methods showed that mixing occurs, but not completely. FTIR was also found to be a better tool for this purpose [31]. A quantitative evaluation of blending with high RAP mixtures was performed recently using a modified staged extraction method [32]. In the modified method, an approximately equal portion of the binder was recovered in each stage of extraction. This study indicated that a partial blending (see Figure 1-5) occurs when the new binder is added to the RAP. However, the further diffusion during storage time results in almost complete mixing. The University of Tennessee's research showed that staged extraction with Trichloroethylene as the solvent, along with varying wash times, is an effective and feasible approach for separating asphalt layers and studying the diffusion of rejuvenators into hard asphalt.

# CHAPTER 2: LONG-TERM AGING OF 100% RECYCLED ASPHALT BINDERS AND MIXTURES

## 2.1 Introduction

This chapter describes the work performed to evaluate the long-term aging and durability of 100% recycled asphalt binders and asphalt mixtures. The rejuvenators used in this study were selected based on a preliminary evaluation of several products (Section 2.2). A long-term aging evaluation was performed on rejuvenated binders that contained the best rejuvenators selected through the screening process (Section 2.3). In addition, mixtures aging experiment was performed with a focus on long-term cracking resistance properties (Section 2.4).

## 2.2 Rejuvenator Screening Process

Eleven rejuvenators were tested for their softening power and some other properties for the selection of the proper products to be used in this study. The products were mainly supplied through a solicitation to the Asphalt Recycling and Reclaiming Association. Table 2.1 provides a brief description of these rejuvenators. The commercial name of these products cannot be disclosed. Therefore, they will be referred to by assigned tags.

#### 2.2.1 Specifications provided by manufacturers

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

Product Name <sup>1</sup>	Tag <sup>1</sup>	Product Description <sup>2</sup>
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 offer excellent
Cationic Water- based Emulsion	CWE	solvency, fluxing and mixing capabilities with the asphalt. NOL is asphalt-free, meaning that it contains 0% asphaltene and is composed of the 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 meet
Anionic Emulsion 2	AE2	Kansas HIR <sup>3</sup> specification, Division 1200.
Bio-Rejuvenator, Oil base Fluid	BOF	This is a mixture of long-chain and tricyclic organic acids, resin acids, fatty acids, esterified fatty acids and vegetable oils. These products are manufactured from renewable raw materials and can be used as a
Bio-Rejuvenator, Oil base Semi-fluid	BOS	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 types were selected based on previous use by
Residual Oil Solvent Extract	ROE	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 Rejuventors Nominated for the Screening Process

<sup>1</sup>Assigned by the author

<sup>2</sup> Claimed by manufacturers

<sup>3</sup> Hot In-Place Recycling

		Viscosi	ity (cSt <sup>1</sup> )		Flash Point	
	at 25°C	at 40 °C	at 60 °C	at 100°C	COC (°C)	
ASTM Standard	D-445	D-445	D-2170	D-445	D-92	
NOL	-	-	200-500		204 min	
CWE	217 - 434	-	200-500	-	-	
AE1	-	-	-	-	NA	
AE2	-	-	-	-	NA	
BOF	-	-	100 max	-	218 min	
BOS	228	-	50	-	260 (Closed Cup)	
HPE	-	-	104	-	210 min	
ROE	-	-	-	52.2	282	
PND	-	92.2	-	7.40	216	
APO		43		9	295 min	

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

<sup>1</sup>centiStokes

## 2.2.2 Laboratory Evaluation

The laboratory studies to evaluate rejuvenators included the following tests:

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

## 2.2.3 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). The samples contained a very hard RAP binder and various dosages of the rejuvenators.

Rejuvenators were mixed with asphalt in the Thermosel chamber. A sample of hard asphalt, heated to 135 °C, was poured into the chamber and precisely measures quantities of

rejuvenators 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 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, the viscosity reduction percentage was considered the criterion to evaluate the softening effectiveness.

Table 2-3 summarizes the results the viscosity tests, and Table 2-4 shows viscosity reductions. Figure 2-1 displays softening curves of rejuvenators.

Table 2-3 Viscosity of the Samples with Various Rejuvenator Contents (Poises)

Rejuvenator Content	PND	HPE	ROE	BOS	BOF	NOL	CWE	AE1	AE2	Motor Oil	APO
0%	1020	1110	1053	960	979	1028	1070	1147	1117	1110	1218
3%	576	652	632	585	354	615	680	890	840	861	707
6%	396	417	533	459	310	370	481	660	634	728	440
9%	226	305	440	440	202	263	348	577	551	607	265

Table 2-4 Viscosity Reduction vs Rejuvenator Content

Rejuvenator Content	PND	HPE	ROE	BOS	BOF	NOL	CWE	AE1	AE2	Motor Oil	APO
3%	44%	41%	40%	39%	64%	40%	36%	22%	25%	22%	42%
6%	61%	62%	49%	52%	68%	64%	55%	42%	43%	34%	64%
9%	78%	73%	58%	54%	79%	74%	67%	50%	51%	45%	78%

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

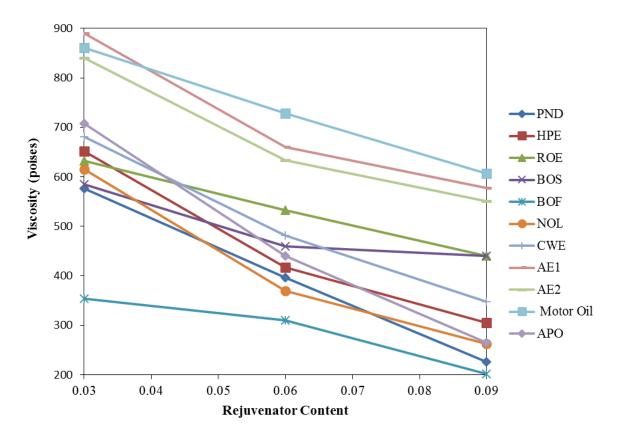


Figure 2-1 Softening Curves for the Rejuvenators

According to the results, BOF, APO, and PND had the best softening powers. Motor oil, on the other hand, was found to be the least effective. CWE performed significantly better than other emulsified rejuvenators did.

## 2.2.4 RTFO Mass Loss

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

Emulsified rejuvenators (CWE, AE1, and AE2) contained 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, an 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 [33]. ROE had just a 0.17% mass loss, and this value ranged between 2%-7% for other products. Table 2-5 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
NOL	166.256	201.221	200.184	34.965	-1.037	-2.966%	-3.050%	0.00119	0.00721
NOL	165.352	200.451	199.351	35.099	-1.100	-3.134%	-5.05070	0.00119	0.00721
PND	166.932	201.543	199.377	34.611	-2.166	-6.258%	-6.128%	0.00184	0.00832
FND	167.374	202.004	199.927	34.630	-2.077	-5.998%	-0.12070	0.00104	0.00852
DOE	168.719	203.649	202.028	34.930	-1.621	-4.641%	1 6570/	0.00023	0.00779
BOF	165.889	200.599	198.977	34.710	-1.622	-4.673%	-4.657%		0.00779
DOG	165.348	200.114	200.200	34.766	0.086	0.247%	0.2000/	0.00046	0.00600
BOS	169.156	203.985	204.094	34.829	0.109	0.313%	0.280%		
Mada a Oʻl	167.526	202.462	201.361	34.936	-1.101	-3.151%	2 1200/	0.00020	0.00704
Motor Oil	167.333	202.450	201.353	35.117	-1.097	-3.124%	-3.138%	0.00020	0.00724
DOF	165.357	200.221	200.166	34.864	-0.055	-0.158%	0.1600/	0.00017	0.00(1(
ROE	165.224	200.223	200.160	34.999	-0.063	-0.180%	-0.169%	0.00016	0.00616
LIDE	169.163	204.103	203.416	34.940	-0.687	-1.966%	1.0220/	0.000(1	0.00/00
HPE	167.526	202.418	201.762	34.892	-0.656	-1.880%	-1.923%	0.00061	0.00680
4.00	166.90	201.91	201.17	35.01	-0.74	-2.11 %	2 210/	0.001.41	0.00(0
APO	168.62	203.70	202.89	35.07	-0.81	-2.31%	-2.21%-	0.00141	0.0060

Table 2-5 RTFO Mass Loss Test Results

#### 2.2.5 Cleveland Open Cup Flash Point Tests

The high temperature during mixing and construction may cause the 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 nonemulsified rejuvenators.

Results from the COC flash point test are presented in Table 2-6 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° C (ASTM D-92). The difference between an observed flash point value and the declared minimum for PND is only 10° C, which falls in the acceptable range. The value declared by the BOS manufacturer is a close cup flash point and cannot be compared with COC values.

Product	COC 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 2-6 Open Cup Cleveland Flash Point Test Results

#### 2.2.6 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 2-7 presents results from the physical observation.

Rejuvenator	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
APO	Transparent, yellow oil	Low	Moderate	Liquid

Table 2-7 Physical Properties Observations

#### 2.2.7 Summary and Final Ranking

Ten commercially available rejuvenators and one type of conventional motor oil were studied and tested to determine the suitability of the 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-emulsified products to withstand construction heating was evaluated by RTFO mass loss and the COC Flash Point Test.

The results of the tests are summarized in Table 2-8. Softening effectiveness was considered as 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 procedure resulted in CWE being the selected emulsified rejuvenator among the three tested.

Rejuvenator	•	y reduction rejuvenator	RTFO Lo		Val	lue	Overall Rank	Remarks	
Kejuvenator	Value	Rank (1 is best)	Value	Rank	Value	Rank	(Lower is better)	Kemar K5	
NOL	74.45%	4	-3.05%	5	$224^{\circ}C$	4	3.15		
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		
APO	78.24%	2	- 2.21 %	4	304°C	2	2.3	Selected	
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		

Table 2-8 Summary of Rejuvenator screening Evaluation Process

\*Overall Rank is based on70% weight for Softening, 15% mass loss, and 15% Flashpoint; For emulsions, based on Softening only.

\*\* 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 with the next ranked product.

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. Therefore, the five rejuvenators selected for further study were BOF, APO, HPE, PND and CWE.

# 2.3 Asphalt Binder Long-term Aging Experiment

## 2.3.1 Experimental Approach

The experimental approach for binder tests was designed based on Superpave PG tests and aging procedures. The aging was simulated by the PAV, which exposes the asphalt to heat and pressure. The standard PAV aging time is 20 hours. There is no definite correlation between PAV aging and actual field aging time, but a study performed in Florida for this purpose estimated that the aging caused by a 20-hour PAV cycle is equivalent to eight years of service [34]. This estimate was used to provide an approximate correlation between PAV time and field aging. Furthermore, the goal was to examine the aging beyond the first eight years. Samples were subjected to three PAV cycles to increase the PAV aging time to 60 hours and simulate roughly 24 years of in-service aging.

One of the objectives of this research was to introduce a quantitative description for binder durability. Hence, the PAV time that increased the high PG temperature of each sample to 95 °C was considered a measure of aging that makes the binder too hard to perform well. This value is referred to as the *Failure PAV Time* in this chapter. Based on the Florida Department of Transportation's testing of over 21 RAP stockpiles, a high temperature PG of 95 °C is the typical grade of a RAP binder in Florida. Each sample was subjected to four levels of aging, which included RTFO and three cycles of PAV. After each level, the

samples underwent DSR testing. The primary parameter used for characterizing the stiffness of the binder, and thereafter the level of aging, was the critical high temperature performance grade of the binder. This parameter is briefly referred to as *High PG* in this dissertation.

Low-temperature properties of samples were also tested. The BBR was used on samples with standard (20 hours) and ultimate (60 hours) PAV aging. Table 2-9 shows tests, aging procedures used, and the corresponding standards.

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 2-9 Tests and Aging Procedures

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).

## 2.3.2 Material

Two sources of virgin asphalt 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 [35]. Tables 2-10 and 2-11 summarize the results from the DSR and BBR tests for asphalt samples.

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

Table 2-10 DSR Test Results for Virgin Asphalt Samples

Table 2-11 BBR Test Results for Virgin Asphalt Samples (at -12 °C and 60 Seconds)

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

Five rejuvenators were selected through the screening process, namely: CWE, HPE, PND, BOF, and APO. The properties of these rejuvenators and the screening process are described in Section 2.2.

## 2.3.3 Results and Discussions

## Step 1: Aging

The continuous (or true) PG values of the samples were determined. Then they were subjected to further PAV aging. The aging time increased at 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 2-1 [36].

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

In which  $T_C$  is continuous grade,  $T_1$  and  $T_2$  are two testing temperatures and  $G^{*}_1$ ,  $\delta_1$ ,  $G^{*}_2$ ,  $\delta_2$  are DSR complex modulus and phase angles at temperature  $T_1$  and  $T_2$  respectively. Table 2-12 presents test results for virgin asphalt binders aged in various PAV times.

Variations of high temperature grade with PAV time are shown in the form of high temperature PG vs. PAV time curves (Figure 2-2). The specific PAV time required to age asphalt samples to the failure point was interpolated and determined as 55 hours for Binder 1 and 44 hours for Binder 2.

Asphalt Sample	PAV Time (Hours)	Test Temperature (°C)	δ (°)	G*/sin δ (kPa)	High PG (°C)	
	0	67	83	1.15	68.36	
	0	76	86	0.45	08.30	
	10	76	86	1.31	78.21	
	10	82	88	0.63	/0.21	
	20	82	86	1.02	82.18	
	20	88	88	0.50	02.10	
Binder 1	30	82	84	1.65	87.06	
Dilider 1	30	88	85	0.91	87.00	
	40	82	80	2.71	89.99	
		88	82	1.28	09.99	
	50	82	79	4.00	92.90	
		88	82	1.87	92.90	
	60	82	79	5.94	96.61	
		88	80	2.86	90.01	
	0	67	85	1.70	71.63	
	0	76	88	0.60		
	10	67	79	6.85	92 (7	
	10	82	85	1.07	82.67	
	20	82	84	1.52	85.46	
	20	88	86	0.74	03.40	
Binder 2	30	82	81	2.54	89.72	
Billdel 2	30	88	84	1.23	09.12	
	40	82	79	3.48	92.66	
	40	88	82	1.72	92.00	
	50	82	75	5.59	97.92	
	50	88	77	2.92	71.72	
	60	82	70	11.77	101.34	
	00	88	74	5.48	101.34	

Table 2-12 DSR Test Results for Asphalt Samples Aged after Various PAV Times

Binder 2 was generally harder than Binder 1 and reached the failure point in a shorter aging time. For both binders, the aging occurred at a faster pace in first ten hours. From there, the aging slowed down and the rate of increasing high temperature grade with PAV time remained almost constant at 0.36 and 0.38 <sup>o</sup>C/hour for Binder 1 and Binder 2, respectively.

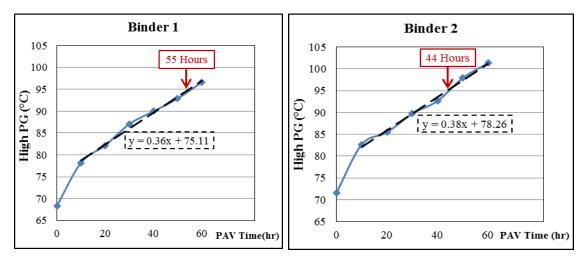


Figure 2-2 High PG vs. PAV Time for Virgin Asphalt Samples

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 PG. Results showed that the RTFO+ 20 hours PAV ages asphalt similarly to the 32 to 36 hours of PAV aging (Table 2-13).

Sample	Aging Level	Test Temperature	δ(°)	G*/sin δ (kPa)	High PG (°C)	Equivalent PAV Time (Hours)
Binder 1	RTFO	82	83	1.91	87.72	32
Dinati	+20hr PAV	85	84	1.36	0111	5-
Binder 2	RTFO	82	80	3.32	91.37	36
Binder 2	+20hr PAV	88	82	1.54	91.57	50

Table 2-13 DSR Test Results for RTFO+PAV Aged Samples

At least 1 kg of each sample was aged at the determined times to supply the PG 95-XX hard asphalt for the next steps: rejuvenation and re-aging. To assure the accuracy of the

grade of aged samples, a DSR test was performed. As shown in Table 2-14, aged samples had a high temperature grade of  $95 \pm 1$  °C.

Sample	Aging Condition	Test Temperature	δ (°)	G*/sin δ (kPa)	High PG(℃)			
Binder 1	55 hr PAV	82	77	5.10	94.61			
Bilidel 1	55 III PAV	88	81	2.35	94.01			
Binder 2	44 hr PAV	82	75	6.16	95.55			
Blider 2	44 III PAV	88	79	2.75	95.55			

Table 2-14 DSR Test Results for Aged Samples

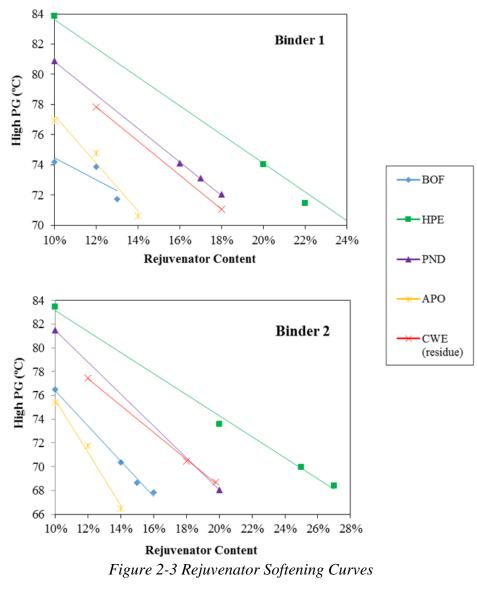
#### Step 2: Rejuvenation

Rejuvenators were mixed with aged asphalts in various proportions and softening curves were established. The mix was hand-stirred with a spatula when the asphalt was at temperatures of 140 °C and 160 °C. Table 2-15 displays required mass contents of each rejuvenator to decrease the grade of the hard asphalt binder samples to their original grade before aging. Softening curves were created in the form of high temperature performance grade vs. rejuvenator content (Figure 2-3).

 Table 2-15 Required Rejuvenator Contents to Soften Aged Asphalt Samples

Asphalt Sample		Binde	er 1			Bind	er 2	
Rejuvenator	CWE	HPE	PND	BOF	CWE	HPE	PND	BOF
Required Content	33% Emulsion	27%	20%	15%	30% Emulsion	22%	18%	13%

The target grade for rejuvenation was set equal to the original continuous grade of virgin asphalt samples: 68.36<sup>+</sup> 1<sup>o</sup>C for Binder 1 and 71.63<sup>+</sup> 1<sup>o</sup>C for Binder 2. Since the rejuvenator CWE is an emulsion, its softening curves were established with consideration of both total mass content and residue content. Eight samples of recycled asphalt were prepared by adding proper amounts of rejuvenators to the hard asphalt obtained in Step 1.



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 2-16 and 2-17.

Binder	Rejuvenator	PAV Time	Test Temperature	δ(°)	G*/sin δ (kPa)	High PG (°C)					
			67	83	1.15	(0.2)					
		0	76	86	0.45	68.36					
		20	82	86	1.02	00 10					
	O · · · 1	20	88	88	0.50	82.18					
	Original	40	82	80	2.71	00.00					
		40	88	82	1.28	89.99					
		(0)	82	79	5.94	0.6.61					
		60	88	80	2.86	96.61					
		0	67	84	1.21	(0.(0					
		0	76	87	0.42	68.63					
		•	76	83	1.20						
	<b>a</b> 1115	20	82	85	0.59	77.56					
	CWE		82	80	1.39						
		40	88	83	0.68	84.75					
			82	74	4.09						
		60	88	78	1.89	92.97					
			67	83	1.28						
		0	76	87	0.44	69.11					
		-	76	82	1.38						
		20	82	84	0.70	78.83					
	HPE		82	79	1.72						
		40	82	82	0.83	86.48					
		-	88	74	3.72						
		60				93.42					
Binder 1			88	78 82	1.87						
		0	67		1.31	69.29					
							-	76	86	0.45	
		20	76	77	1.90	81.38					
	PND		82	80	0.93						
		40	82	72	3.29	92.06					
			88	76	1.62						
		60	82	63	12.09	99.72					
			88	68	5.20						
		0	67	81	1.30	69.21					
			76	87	0.45						
		20	76	81	1.63	80.40					
	BOF	-	82	84	0.84						
	~ -	40	82	77	3.28	91.71					
			88	79	1.57	/ 1.// 1					
		60	82	71	10.76	101.76					
			88	79	5.23	101.70					
		0	67	80	1.25	68.92					
	APO	0	76	84	0.44	00.72					
		20	76	80	1.55	79.56					
		20	82	82	0.74	19.50					
		40	82	77	1.95	88.09					
		+0	88	79	1.01	00.07					
		60	82	62	8.82	98.84					
		00	88	73	4.06	70.04					

 Table 2-16 DSR Test Results for Re-aged Samples - Binder 1

Binder	Rejuvenator	PAV Time	Test Temperature	δ (°)	G*/sin δ (kPa)	High PG (°C)											
		0	67	85	1.70	71.62											
		0	76	88	0.60	71.63											
		20	82	84	1.52	95.46											
	Original	20	88	86	0.74	85.46											
	Original	40	82	79	3.48	02.00											
		40	88	82	1.72	92.66											
		(0)	82	70	11.77	101.24											
		60	88	74	5.48	101.34											
		0	67	82	1.90	72.20											
	CWE	0	76	85	0.64	72.29											
		20	76	81	1.75	80.72											
		20	82	83	0.86	80.72											
		40	82	78	2.17	00.01											
		40	88	81	1.03	88.24											
		(0)	82	73	5.22	06.20											
		60	88	76	2.61	96.30											
		0	67	80	1.86	70 (0											
		0	76	84	0.69	72.60											
		• •	82	80	1.17												
		20	88	83	0.53	83.18											
	HPE		82	77	2.50	89.40											
		40	88	80	1.19												
			82	74	4.23												
Binder 2		60	88	77	2.13	94.61											
			67	79	1.77												
		0	76	84	0.60	71.77											
				-	-									82	77	1.58	
		20	88	80	0.85	86.43											
	PND		82	68	5.73												
		40	88	73	2.73	96.11											
			82	62	11.69												
		60	88	66	5.91	103.63											
			67	80	1.85												
		0	76	84	0.69	72.61											
			82	80	1.60	0.1.1-											
		20	88	82	0.80	86.07											
	BOF		82	73	5.59												
		40	88	76	2.90	97.71											
			88	76	9.99												
		60	91	69	7.37	110.73											
	APO -		67	80	1.65												
		0	76	77	0.60	71.46											
			76	77	2.09												
		20	82	80	1.05	82.43											
			82	73	3.13												
		40	88	73	1.49	91.22											
			88	65	12.02												
		60	88	70	5.77	102.33											

Table 2-17 DSR Test Results for Re-aged Samples - Binder 2

Table 2-18 summarizes the high temperature grading for 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 the re-aging curves of rejuvenated binders are more linear than those of original asphalt; while the 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.

PAV		High Temperature Performance Grade (°C)										
Time		Binder 1 Binder 2										
(Hours)	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 2-18 Summary of Aging Behavior of Original Rejuvenated Samples

Table 2-19 displays the 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 PG vs. aging time curves in Figure 2-4.

	Tuble 2-19 Hurdening rules of Original Refuvenaled Samples											
	]	Hardening Rate based on High Temperature Performance Grade ( $^{\circ}C/hr$ )										
Aging Phase			Binde	r 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	36         0.39         0.36         0.46         0.53         0.48         0.40         0.39         0.29         0.43         0.62         0							0.50			

Table 2-19 Hardening rates of Original Rejuvenated Samples

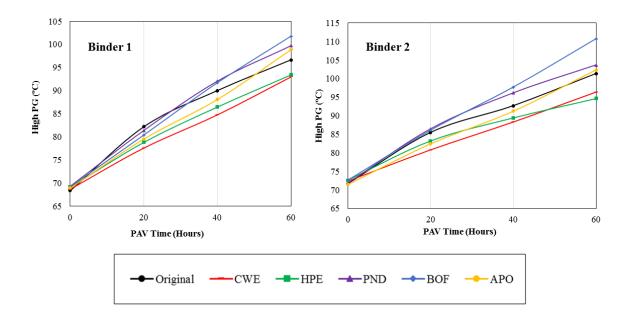


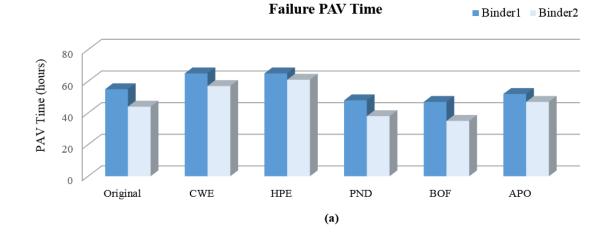
Figure 2-4 Re-aging of Rejuvenated Binder Samples

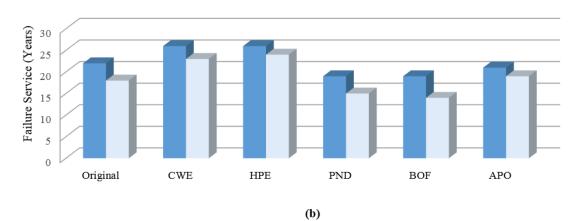
The longevity of rejuvenated and original asphalt samples was evaluated. Reaching a high temperature grade of 95°C was considered a typical failure point, and the PAV time it took each sample to reach this grade was called PAV failure time. The service life of samples was calculated from PAV times, assuming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

In addition, a durability index was defined as the Failure PAV Time of the rejuvenated samples to that of the corresponding virgin asphalt. This index indicates the effect of a rejuvenator on the durability of the rejuvenated binder. As reflected in Table 2-20 and Figure 2-5, service life analyses showed that selecting the proper rejuvenator has a significant effect on the durability of recycled asphalt binder.

		1		1									
Longevity		Binder 1						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	
Durability Index	1.0	1.18	1.18	0.87	0.85	0.95	1.0	1.30	1.39	0.86	0.80	1.07	

Table 2-20 Longevity of Rejuvenated and Virgin Asphalt Samples





**Field Service Life** 

Figure 2-5 Longevity of Rejuvenated Binders Based on (a) Failure PAV Time and (b) Failure Service Years

# Low Temperature Properties

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 RTFO and 20 hours of exposure to PAV, and at the ultimate aging condition (60 hours PAV). Tables 3-21 and 3-22 display results from the BBR tests.

	2-21 LOW 16mp			I i j i i	Critical	Critical
Rejuvenator	Aging	Temperature	Stiffness	m-value	Temperature	Temperature
Rejuvenator	Aging	(°C)	(MPa)	III-value	(Stiffness)	(m-value)
	RTFO + 20hr	-12	190	0.309		
	PAV	-12	430	0.228	-25.36	-22.67
Original	1111	-6	143	0.299		
Oliginai	60 hr PAV	-12	263	0.258	-23.34	-15.85
	00 111 1 1 1 1	-18	475	0.202	20.01	10.00
	RTFO + 20hr	-12	81.1	0.346		
	PAV	-18	187	0.288	-31.39	-26.76
CWE		-6	55.3	0.311		
	60 hr PAV	-12	112	0.289	-31.26	-19.00
		-18	212	0.241		
	RTFO + 20hr	-12	72.8	0.353	21.50	25.02
	PAV	-18	178	0.27	-31.50	-25.83
HPE		-6	46.1	0.319		
	60 hr PAV	-12	89.8	0.29	-33.45	-19.93
		-18	169	0.258		
	RTFO + 20hr	-12	52.1	0.319	26.60	24.00
	PAV	-18	107	0.262	-36.60	-24.00
PND		-6	37.3	0.295		
	60 hr PAV	-12	69	0.205	-37.93	-15.67
		-18	120	0.206		
	RTFO + 20hr	-12	66.4	0.36	-30.44	-24.38
	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		
	RTFO + 20hr	-12	29.3	0.385	-41.61	-34.14
	PAV	-18	59.7	0.343	-11.01	-37.17
APO		-6	33.4	0.333		
	60 hr PAV	-12	73.1	0.258	-37.56	-18.64
		-18	126	0.243		

Table 2-21 Low Temperature Grading of Samples from BBR Tests- Binder 1

Rejuvenator	Aging	Temperature (℃)	Stiffness (MPa)	m-value	Critical Temperature (Stiffness)	Critical Temperature (m-value)	
		-12	159	0.313	27.47	24.17	
	RTFO + 20hr 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 PAV	-12	80.3	0.332	-33.47	-27.82	
	$K1FO \pm 2000 PAV$	-18	160	0.299	-33.47	-27.82	
CWE		-6	58.4	0.309			
	60 hr PAV	-12	112	0.282	-32.38	-18.00	
		-18	198	0.265			
		-12	62.3	0.342	20.90	24.22	
	RTFO + 20hr 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	
		-18	233	0.205			
		-12	44.5	0.322	25 (4	24.60	
	RTFO + 20hr 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 PAV	-12	88.5	0.314	-35.38	-26.00	
	$K1FO \pm 2000 PAV$	-18	153	0.293	-33.38	-20.00	
BOF		-6	47.7	0.308			
	60 hr PAV	-12	113	0.286	-32.17	-18.18	
		-18	201	0.258			
	RTFO + 20hr PAV	-12	40.8	0.33	-40.15	-34.86	
	RTFO + 20hr PAV	-18	78.9	0.316	-40.13	-34.00	
AOP		-6	33.5	0.325			
	60 hr PAV	-12	67.3	0.29	-34.62	-20.29	
		-18	137	0.217			

Table 2-22 Low Temperature Grading of Samples from BBR Tests - Binder 2

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 2-2 and 2-3 to arrive at their results. Low- temperature grades of samples at two aging stages were based on the BBR m-value at 60 seconds graphically.

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

$$T_c = T_1 + \left[\frac{0.3 - m_1}{m_1 - m_2} \times (T_1 - T_2)\right] - 10$$
 Equation 2-3

In which  $T_C$  is continuous grade,  $T_1$  and  $T_2$  are two testing temperatures and  $S_1$ ,  $m_1$ ,  $S_2$ , and  $m_2$  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. In all cases, the m-values were more critical and resulted in a higher low temperature grade. Figure 2-6 shows the low temperature grades of samples at two aging stages based on the BBR m-value at 60 seconds.

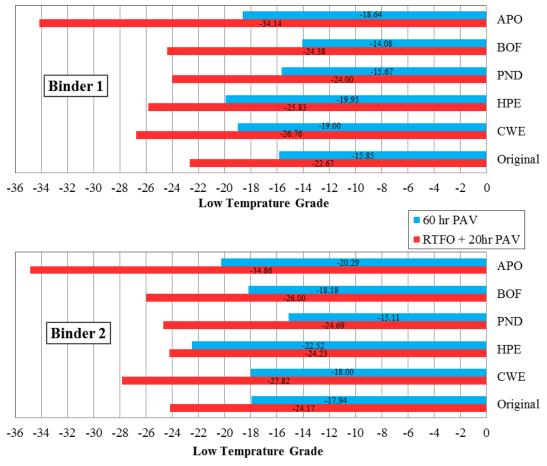


Figure 2-6Low Temperature Grade Based on BBR m-value

All rejuvenated binders passed the M320 low temperature requirements (S $\leq$  300 MPa and m  $\geq$  0.300 at 60 seconds and -12°C) for PG 67-22. Rejuvenated samples were significantly softer (with a lower creep stiffness) at low temperatures, when compared with 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.

A study on the effects of BBR parameters on the thermal stress properties of asphalt binders had showed that stiffness (s) is the factor that primarily controls low temperature thermal stress development [37]. Results from the current work indicated 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.

## 2.4 Asphalt Mixture Aging Experiment

## 2.4.1 Experimental Approach

The Texas Overlay Test (TOT) was used to measure the cracking resistance of rejuvenated mixtures and compare it with the control mixtures. To assess the durability, the rejuvenated mixtures underwent artificial aging by the Accelerated Pavement Weathering System (APWS). Two samples of rejuvenated mixture and two control samples were used in this study.

#### 2.4.2 Sample Preparation

The RAP was sampled from a HIR project on Florida State Road 15. The asphalt mat was heated to an average temperature of 250°F, and then milled to a 1-inch depth. The material was collected from the windrow prior to the introduction of the rejuvenator. Thus, this mixture represented the non-rejuvenated RAP. The virgin binder used for the control mixtures was a PG 67-22 non-modified asphalt. The two rejuvenators that performed the best in the binder testing experiment, namely HPE and CWE, were selected to this part of the study.

#### **Control Samples**

Two control mixtures were used in this study. Control I consisted of the aggregate extracted from the RAP and virgin asphalt binder. The aggregate was extracted from the RAP using an ignition oven. The asphalt content was determined in accordance with AASHTO T 308 using the provided calibration factor of 0.1. Then, the extracted aggregate was mixed with a PG 67-22 binder at the same binder content as determined by extraction (6.3%).

Control II samples were SP-9.5 and FC-9.5 mixtures prepared according to FDOT requirements. These samples represented common asphalt mixtures used in Florida with gradations similar to the obtained RAP.

## **Rejuvenated Samples**

The two rejuvenated samples were the RAP mixtures, softened by CWE and HPE rejuvenators. To characterize the binder and establish softening curves, 180 grams of the binder was recovered in accordance with ASTM D5404. The PG was determined in accordance with AASHTO M320, as presented in Table 2-23. The mixtures experienced

heating when being milled and sampled. This heating was estimated to have almost the same aging effect as construction heating. Therefore, the criterion for the RTFO residue  $(G^*/\sin\delta < 2.2 \text{ kPa})$  was used to determine its high temperature grade.

Table 2-23 Performance Grade of the Recovered Binders

Р	Property		Specifications	Temperature	Results		
<b>Recovered Bine</b>	ler						
Dumannia Shaar				70 °C	6.11		
Dynamic Shear G*/sinδ, 10 rad/	a l/Do	T 315	2.2 min.	76 °C	2.86		
G <sup>*/sino</sup> , 10 lau/	s, кга			82 °C	1.38		
PAV Residue (1	100°C, 300 psi, 20 hr.)	)					
Dynamic Shear		Т 315	5000	22 °C	5800		
G*•sino, 10 rad/	's, kPa	1 315	5000 max.	25 °C	4110		
	Stiffness, MPa (60 s)		300 max.	-12 °C	143		
Bending Beam	m-value	Т 313	0.300 min.	-12 C	0.334		
Denuing Deam	Stiffness, MPa (60 s)	1 515	300 max.	-18 °C	279		
	m-value		0.300 min.	-18 °C	0.288		
AASHTO M 320 Superpave Binder Grade, PG:							

A softening curve was established for each rejuvenating agent when blended with the recovered RAP binder. The purpose of establishing the curves was to determine the dosage needed to reduce the high PG to 67 °C. Figure 2-7 shows the softening curves, and Table 2-24 displays the rejuvenator percentages of the mixtures and their high PGs. All percentages are reported by the Total Weight of Mixture (TWM).

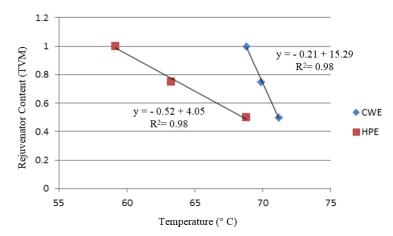


Figure 2-7 Softening Curves of Rejuvenators Mixed with Recovered RAP

Mixture	Binder	Rejuvenator % (TWM)	High Temperature True Grade
RAP	RAP Binder	0	78.2 °C
CWE Recycled	Rejuvenated Binder	1	68.8 °C
HPE Recycled	Rejuvenated Binder	0.5	68.8 °C
Control I	Virgin PG 67-22	0	67.3
Control II	Virgin PG 67-22	0	67.0 (Design value)

Table 2-24 Rejuvenator Percentage and High Temperature Grades of Samples

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

The rejuvenated mixtures and the Control I were evaluated for their design at 50 gyrations, and their maximum specific gravity was determined in accordance with ASTM D 2041. Table 2-25 displays some properties of the specimens.

	Test Method	Sample					
Property		Control II		Control I	CWE	HPE	
		SP-9.5	FC-9.5	Control I	CWE	пге	
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 2-25 Volumetric Properties of Mixtures

## 2.4.3 Testing Procedures

The cracking resistance of samples was tested by the TOT in accordance with the Tex 248-F specifications [38]. First, three replicates of all samples were tested. The Control II was tested only in the initial stage. Three replicate specimens from CWE and HPE mixtures, along with two replicates from the Control I, were aged in the APWS for 1,000 hours. Two other replicates from rejuvenated samples were exposed to the APWS for 3,000 hours. A 3,000-hour APWS exposure simulates the aging that occurs in the field in 7 to 10 years [39]. Following is a brief description of TOT and APWS:

## **Texas Overlay Test**

The TOT was developed by the Texas Department of Transportation to evaluate the susceptibility of asphalt mixtures to fatigue and reflective cracking. This apparatus applies repeated tension loads to the specimen to simulate the repeated opening and closing of pavement joints and cracks due to temperature variations and traffic loading. The TOT was performed for all samples in accordance with the Tex-248-F standard specification [38].

#### Accelerated Pavement Weathering System (APWS)

The long-term aging of pavements is affected by many environmental factors such as temperature, ultraviolet radiation, and water exposure. The Superpave aging protocols, RTFO and PAV, only age the asphalt binder. In addition, these protocols are not capable of simulating the effects of all of the affecting factors. The aging of asphalt pavement material varies by the pavement's depth. While surface layers experience more intense aging, less aging occurs in deeper layers [40]. The APWS is designed by PRI Asphalt Technologies, Inc. to apply accelerated aging on asphalt pavement specimens. It ages specimens by simulating rain, sunshine and temperature variations, which are major factors that cause aging of the surface layers of pavement. Grzybowski et al. explained development of this system and showed that the aging profile resulting from the APWS aging is similar to that observed in real pavement [41]. Figure 2-8 shows the APWS at PRI Asphalt Technologies, Inc.



Figure 2-8 Accelerated Pavement Weathering System (APWS)

## 2.4.4 Results and Discussions

Table 2-26 displays the results from the Texas Overlay Test before and after APWS aging. At the initial stage, a 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 the two control samples, which were made with virgin asphalt. These observations show that RAP binder can even enhance the cracking performance of pavement if is rejuvenated appropriately. Figure 2-9 shows the variations of the Texas Overlay Test results with APWS aging time.

Mixture	Replicate	Starting Load, kN	Final Load, kN	Decline in Load, %	Cycles to Failure	Average Cycles to Failure	
0 Hours							
Control I	1	2.185	0.153	93	55	71	
	2	1.724	0.117	93.2	72		
	3	2.325	0.159	93.2	86		
	1 (SP)	4.230	0.282	93.3	104		
Control II	2 (SP)	0.155	0.008	94.7	62	63	
	3 (FC)	2.582	0.175	93.2	24		
	1	1.653	0.112	93.2	384		
HPE	2	1.759	0.12	93.2	145	239	
	3	1.797	0.119	93.4	189		
	1	1.576	1.109	93.1	347	267	
CWE	2	1.742	0.118	93.2	144		
	3	1.707	0.118	93.1	310		
1000 Hours							
Control I	1	2.435	0.167	93.1	36	58	
Control I	2	2.438	0.168	93.1	79	50	
	1	2.213	0.151	93.2	186		
HPE	2	2.135	0.147	93.1	98	186	
	3	2.386	0.167	93	275	1	
	1	2.53	0.174	93.1	153	253	
CWE	2	2.721	0.19	93	256		
	3	2.526	0.174	93.1	349		
3000 Hours							
HPE	1	2.987	0.23	93.2	75	71	
	2	2.55	0.17	93.4	66		
CWE	1	2.927	0.199	93.2	58	98	
	2	2.663	0.18	93.3	137	70	

Table 2-26 Results from TOT tests, Before and After APWS Aging

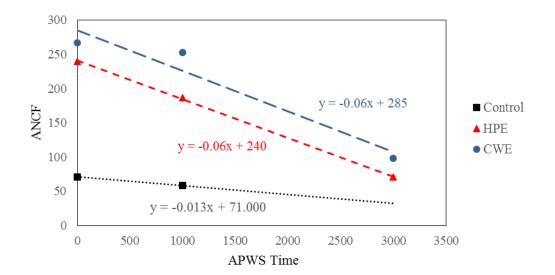


Figure 2-9 Variations of the ANCF with APWS Aging Time

The results showed that the Average Number of Cycles to Failure (ANCF) decreases with an increased APWS aging time. This trend confirms that weathering of the pavement makes it more susceptible to fatigue and reflective cracking. The rate of decrease in the ANCF with APWS time was considerably faster for rejuvenated mixtures than for Control I. This means that cracking susceptibility, which is an indication of aging, increased significantly faster in mixtures containing rejuvenated asphalt than in those made with a 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 unaged control samples. It can be concluded that although rejuvenated samples have an overall better cracking performance, they might lose their resistance faster than virgin asphalt mixtures. This trend needs further investigation, with more samples and longer aging times.

## **Considerations**

It should be noted that there are several factors that limit the generalization of the observed trends. These include:

- 1. The variability of TOT results
- 2. The variability of the air voids between the control and recycled samples
- 3. The relatively small size of this experiment.

# 2.5 Summary

The durability of recycled asphalt 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.

#### 2.5.1 Binder Aging Experiment

Two asphalt binder samples were aged by PAV until their high PG reached 95±1°C. Each of these aged asphalt samples were softened by adding one of the five rejuvenators (CWE, HPE, PND, APO and BOF) until their high PG 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 accelerated it.
- While the slope of aging curves dropped significantly after 20 hours for virgin asphalt samples, the aging curve of recycled binders was 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 to rejuvenating with a less effective product.
- Almost the same trend experienced for high PG was true for low temperature PG.

## 2.5.2 Mixture Aging Experiment

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

- Recycled asphalt mixes can be more resistant to fatigue and reflective cracking than virgin asphalt mixes, when rejuvenated properly.
- The resistance of recycled pavement to cracking decreased faster due to aging when compared with new asphalt.

# CHAPTER 3: AGING AND DURABILITY OF PARTIALLY RECYCLED ASPHALT BINDERS

# 3.1 Introduction

This chapter covers the research performed on the long-term aging and durability of partially recycled asphalt binders, containing 20 to 40 percent RAP binder. This study was a continuation to the research presented in Chapter 2. The experience gained in that study was used to improve the experimental approach and some additional aspects of durability were investigated.

# 3.2 Experimental Approach

The experimental approach was generally similar to the one used in Chapter 2 and described in Section 2.2. The Superpave PG tests were used to measure the properties of the binder and RTFO and PAV were used to simulate short-term and long-term aging, respectively. The PAV aging time was extended from the standard 20 hours to 60 hours to study the aging for a longer time. Although the overall experimental approach was similar to the one used in Chapter 2, there were several differences in this part of the study:

- 1. The aged binder was obtained by recovering the asphalt from the RAP sample. Artificial aging was used for this purpose in Chapter 2.
- 2. The samples contained both virgin and RAP binder. RAP content was 20 to 40 percent.
- 3. Samples underwent RTFO aging prior to the extended PAV aging. The short-term aging of the rejuvenated binders was also studied comparatively.

4. The critical PAV time was defined as the PAV time that increases the high PG of each sample from 70 °C to 95 °C. This parameter was considered a measure of aging that makes the binder too hard to perform well. In the method described in Chapter 2, failure PAV time was used as the longevity indicator and was defined as the PAV time that increases the high PG from the existing condition to 95 °C.

## 3.3 Material

Two types of RAP, two virgin binders, and two rejuvenators were used in the samples.

## 3.3.1 RAP Binders

A medium-aged RAP and a hard RAP, recently milled in Florida, were used in this study. These are referred to as *RAP 1* and *RAP 2*, respectively. The RAP binder was recovered using a centrifuge extractor and a rotary evaporator (Figure 3-1), in accordance with ASTM D2172 and ASTM D5404.

## 3.3.2 Virgin Binders

Two types of virgin binders were used in this study. These are referred to as VB1 and VB2. Although both binders had an incremental grade of PG 67-22, the continuous grade of VB1 was slightly higher than VB2. Table 3-1 shows the results from the high temperature DSR tests on RAP and virgin binders resulting in high PG values. The RTFO mass loss was 0.61% for VB1, and 0.43% for VB2.



*Figure 3-1 Binder Recovery Apparatus: Centrifuge Extractor (a) and Rotary Evaporator (b)* 

Binder	Test Temperature (°C)	δ (°)	G*/sin δ (kPa)	High PG (°C)	
RAP 1	82	82	3.25	91.95	
(Medium)	88	84	1.60	91.95	
RAP 2	82	76	14.60	104.24	
(Hard)	88	79	7.08	104.24	
VB1	67	86	1.48	70.10	
(Not Aged)	76	86	0.49	70.19	
VB2	67	88	1.29	(0.19	
(Not Aged)	76	88	0.45	69.18	
VB1	67	83	3.68	70.07	
(RTFO Aged)	73	85	1.69	70.97	
VB2	67	85	2.77	(0.9(	
(RTFO Aged)	73	87	1.32	68.86	

Table 3-1 DSR Test Results for RAP and Virgin Binders

The high PG was determined through logarithmic interpolation to obtain the highesttemperature so that PG criterion corresponding to each stage is satisfied. These criteria are:Original (non-aged) sample:  $G^*/\sin \delta \ge 1.0$  kPaRTFO-aged sample:  $G^*/\sin \delta \ge 2.2$  kPaEquation 3-2

Where  $G^*$  is the complex modulus and  $\delta$  the phase angle measured by DSR tests.

#### 3.3.3 Recycling Agents

Two recycling agents (rejuvenators) were used. These are commercial products used in Florida, referred to as RA1 and RA2.

RA1 was similar to the rejuvenator named HPE in Chapter 2. This rejuvenator is a dark yellow heavy paraffinic oil with a high aromatic content that provides good softening power. The rejuvenator contains no Asphaltene. This helps restore the Maltene to Asphaltene ratios reduced by aging. The flash point of this oil was 420 °F, as determined by the COC Test (ASTM D92). The material has a good high temperature stability and does not emit much smoke at mixing temperatures. However, its high aromatic content allows it to evaporate quickly during the mixing procedures. The RTFO mass loss was determined to be as high as 1.92% for RA1.

RA2 is a semi-solid black substance with an asphalt odor. This product is manufactured by re-refining used oils through vacuum distillation. Using a re-refined product a step toward enhancing the use of recycled material. This rejuvenator has a high flash point of 522 °F and does not release much smoke in high temperatures. It also evaporates much less than RA1 at mixing temperatures, and its RTFO mass loss is only 0.21%.

## **3.4** Sample Preparation

Sixteen samples were prepared by varying the RAP content and the type of RAP, virgin binder and recycling agent. The two virgin binders were used as controls. The samples were prepared by mixing a soft binder with the RAP binder. The soft binder is a mixture of a virgin binder with a recycling agent. This sequence correlates with the practice often followed by the industry. To facilitate the comparison between samples, similar initial high PG values are required. Thus, the target grade for the samples was set as the high PG of virgin binders  $\pm 1$  °C. To determine the proportion of the components that would make up samples with those target grades, three steps were required, as discussed in the next sections.

#### **3.4.1** Step 1: Determination of Soft Binder Grade

The first step was to determine the grade of the soft binder so that after blending with the RAP binder, a sample with the target grade is achieved. Hence, a linear interpolation was used to estimate the grade of the soft binder by using the RAP content, the high PG of the RAP and the target high PG. This is in accordance with the method recommended in ASTM D4887. Figure 3-2 shows this interpolation for each combination of virgin and RAP binder.

#### **3.4.2** Step 2: Establishing Softening Curves

Softening curves were established for each combination of virgin binder and recycling agent, as shown in Figure 3-3. The dotted lines represent the linear trend. The softening power of the RA2 was considerably lower than the RA1. Therefore, a very high content was needed to soften the binder to the desired grade. This fact makes the RA2 an inappropriate choice when high RAP content is considered, and especially when the RAP is highly aged.

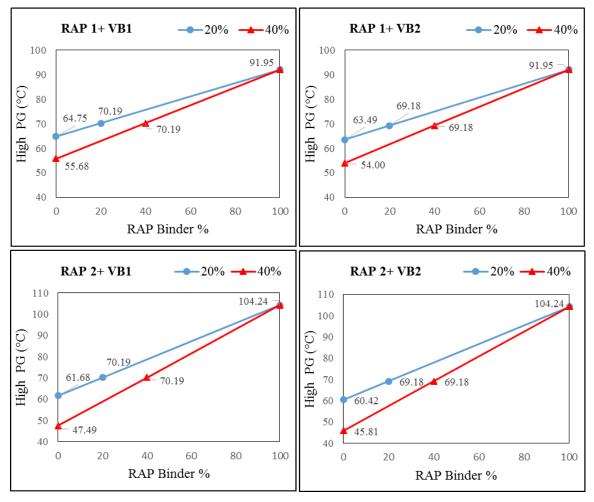


Figure 3-2 Soft Binder Grade Determination

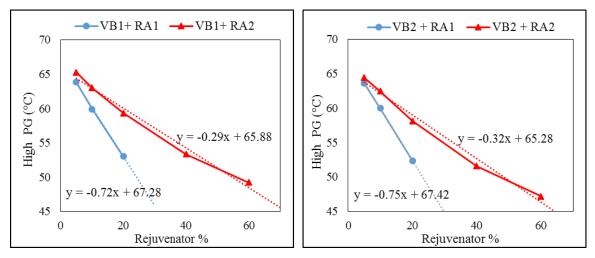


Figure 3-3 Softening Curves

#### **3.4.3** Step 3: Calculating the Proportion of Material

The results from the two preceding steps make it possible to calculate the proportion of RAP binder, virgin binder and recycling agent for each sample. Table 3-2 displays the factorial design of samples and the composition of each. All of the percentages in this table are by weight.

Sample				mple Comp	0		Target High	Soft Binder	RA /VB	
Number	VB	RA	RAP	VB%	RA%	%RAP	PG (°C)	High PG (°C)	(%)	
S01			P1	76.6	3.4	20	$70.19 \pm 1$	64.75	4.3	
S02		11	RAP1	50.3	9.7	40	$70.19 \pm 1$	55.68	16.1	
S03		RA1	P2	73.8	6.2	20	$70.19 \pm 1$	61.68	7.7	
S04	31			RAP2]	43.2	16.8	40	$70.19 \pm 1$	47.49	28.0
S05	VB1		P1	75.1	4.9	20	$70.19 \pm 1$	64.75	6.2	
S06		RA2	RAPI	40.7	19.3	40	$70.19 \pm 1$	55.68	32.2	
S07		$\mathbb{R}^{J}$	P2	69.1	10.9	20	$70.19 \pm 1$	61.68	13.6	
S08			RAP2	19.8	40.2	40	$70.19 \pm 1$	47.49	67.0	
S09				75.9	4.1	20	$69.18 \pm 1$	63.49	5.2	
S10		11	RAP1	49.3	10.7	40	$69.18 \pm 1$	54.00	17.9	
S11		RAI	P2	72.5	7.5	20	$69.18 \pm 1$	60.42	9.4	
S12	VB2		RAP2	42.8	17.2	40	$69.18 \pm 1$	45.81	28.6	
S13	Ŋ			74.1	5.9	20	$69.18 \pm 1$	63.49	7.4	
S14		72	RAP1	40.5	19.5	40	$69.18 \pm 1$	54.00	32.6	
S15		RA2	RAP2	68.2	11.8	20	$69.18 \pm 1$	61.42	14.7	
S16			RA	20.2	39.8	40	$69.18 \pm 1$	45.81	66.3	

Table 3-2 Factorial Design and Composition of Samples

## 3.5 **RTFO Aging**

#### 3.5.1 Results

The RTFO simulates the aging that the binder undergoes during construction. This aging is primarily due to the evaporation of lighter components of the asphalt binder when it is heated. Table 3-3 shows the results of the DSR tests and the high PG of the samples before and after RTFO aging. The high PG values for non-aged and RTFO-aged samples were determined differently based on Equations 2-1 and 2-2, respectively.

				v	Aging			0	TFO			
Sample			Test Temp. (°C)	δ (°)	G*/sin δ (kPa)	High PG (°C)	Test Temp. (°C)	δ (°)	G*/sin δ (kPa)	High PG (°C)	Difference (RTFO-No Aging) (°C)	
VB1	-	0	67	86	1.48	70.19	67	83	3.68	70.97	0.77	
			76 67	86 86	0.49		73 67	85 82	1.69 3.19			
S01		3.42	73	87	0.65	69.47	73	84	1.58	70.17	0.70	
G 0 2		0.60	67	85	1.46	<0.0 <b>7</b>	67	80	4.08	<b>51</b> 00		
S02		9.68	73	87	0.68	69.97	73	83	1.91	71.88	1.91	
502	RA1	( 10	67	85	1.62	70.06	67	82	4.25	72 10	1 1 4	
S03		6.19	73	87	0.78	70.96	73	84	1.96	72.10	1.14	
S04		16.82	67	84	1.53	70.72	67	79	4.88	72 59	2.86	
504		10.82	73	86	0.77	70.72	73	82	2.36	73.58	2.80	
S05		4.93	67	85	1.46	70.55	67	80	3.79	71.32	0.77	
505		ч.75	73	86	0.77	70.55	73	82	1.78	/1.52	0.77	
S06		19.33	67	83	1.35	69.74	67	67	3.31	70.65	0.90	
500	RA2	17.55	73	85	0.70	07.74	73	73	1.69	70.05	0.90	
S07	Я	10.48	67	84	1.61	70.87	67	79	3.80	71.92	1.04	
507		10.10	73	86	0.77	/0.07	73	82	1.95	,1.)2	1.01	
S08		40.18	67	74	1.42	70.59	67	69	3.34	70.65	0.06	
			73	75	0.79		73	72	1.68			
VB2	-	0	67	88	1.29	69.18	67	85	2.62	68.39	-0.79	
			76	88	0.45		73	87	1.23			
S09		4.14	67	86	1.42	69.96	67	84	2.98	69.37	-0.59	
			73	87	0.70		73	85	1.38			
S10	_	10.73	67 73	86 87	1.26 0.63	69.04	67 73	83 84	2.97 1.41	69.42	0.38	
	RA1		67	86	1.34		67	84	2.92			
S11		7.53	73	88	0.64	69.36	73	86	1.41	69.33	-0.02	
			67	85	1.43		67	81	3.42			
S12		17.18	73	87	0.71	70.07	73	84	1.65	70.63	0.56	
G12		5.00	67	84	1.15	(0.00	67	82	2.52	(0.00	0.20	
S13		5.88	73	85	0.60	68.29	73	84	1.19	68.09	-0.20	
014		10.54	67	82	1.36	(0.55	67	79	2.70	(0, (1	0.04	
S14	RA2	19.54	73	84	0.66	69.55	73	81	1.26	68.61	-0.94	
S15	R∕	11.75	67	85	1.38	69.85	67	82	2.85	69.16	6 0.00	
515		11.73	73	86	0.70	07.83	73	84	1.39	09.10	6 -0.69	
S16		39.79	67	77	1.44		67	74	2.74	68.90	0 -1.10	
510		57.19	73	80	0.69	/0.00	73	77	1.37	00.70	-1.10	

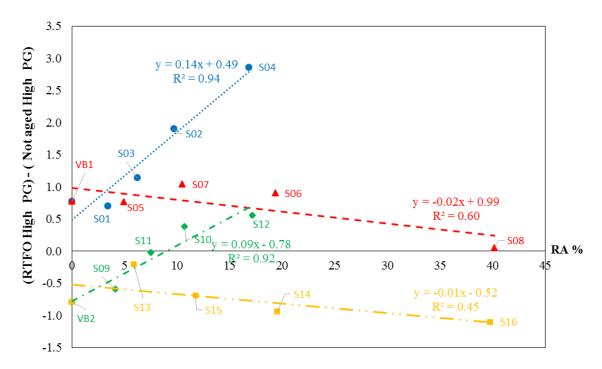
Table 3-3 High PG of Samples Based on Non-aged and RTFO-aged criteria

#### 3.5.2 Discussions

The following trends were observed:

- The degree of aging caused by the RTFO depended on the type of asphalt and recycling agent. VB1 lost more weight in the RTFO (0.61% compared to 0.43% for VB2) and experienced more aging. Its RTFO grade (based on Equation 3-2) was 0.77 °C higher than its non-aged grade (based on Equation 3-1). On the other hand, the RTFO grade was 0.79 °C less than the non-aged grade for VB2.
- 2. RA1 increased RTFO aging. There is a meaningful correlation between the percentage of RA1 and the extent of RTFO aging (see the correlation in Figure 3-4). This is in agreement with the fact that RA1 has high aromatic content and RTFO mass loss. RA2 was impacted less intensely by RTFO aging. Faster RTFO aging is not necessarily a negative quality. In fact, PG specifications call for a minimum stiffness for the pavement to have the adequate strength after construction. However, if a rejuvenator causes faster aging, this should be known and considered during the mix design phase.
- 3. The phase angle is relatively small for samples with a high content of RA2. For instance, samples 12 and 16 have almost similar magnitudes of G\*/sin  $\delta$ , but the phase angle is 8° smaller for sample 16. Therefore, RA2 decreases the viscous portion of the complex modulus.

• VB1 + RA1 • VB1 + RA2 • VB2 + RA1 • VB2 + RA2



*Figure 3-4 Change in the High PG Due to RTFO Aging (RTFO PG – Non-aged PG)* 

## 3.6 PAV Aging

#### 3.6.1 Results

Three 20-hour cycles of the PAV aging with a temperature of 100 °C and a pressure of 2.1 MPa were applied. Table 3-4 presents the results of the DSR tests on samples after each PAV cycle. These samples were already RTFO-aged. Therefore, the criterion for the RTFO samples (Equation 3-2) was used to determine the high PG.

		20	Hour PA	V			our PAV		60 Hour PAV				
Sample	Test Temp. (°C)	δ (°)	G*/sin δ (kPa)	High PG (°C)	Test Temp. (°C)	δ (°)	G*/sin δ (kPa)	High PG (°C)	Test Temp. (°C)	δ (°)	G*/sin δ (kPa)	High PG (°C)	
VB1	82	78	2.51	83.05	82	74	8.47	92.36	82	66	22.79	100.53	
VDI	88	81	1.18	83.03	88	78	3.88	92.30	88	70	10.69	100.55	
S01	76	78	4.31	81.39	82	76	5.35	89.51	82	69	13.79	95.78	
501	82	81	2.04	01.57	88	79	2.63	07.51	88	73	6.2	75.76	
S02	82	81	2.22	82.07	82	75	5.54	89.36	82	69	11.28	96.63	
302	88	83	1.03	82.07	88	78	2.61	89.30	88	73	5.77	90.03	
S03	82	80	2.60	83.43	82	73	7.00	91.78	82	69	14.82	97.46	
305	88	83	1.29	85.45	88	77	3.44	91.70	88	73	7.07	97.40	
S04	82	79	2.80	83.50	82	73	6.64	91.36	82	69	13.31	96.08	
504	88	82	1.36	85.50	88	77	3.27	91.50	88	73	6.18	90.08	
505	82	79	2.84	04 20	82	71	8.64	02.50	82	64	23.20	100.97	
S05	88	81	1.45	84.28	88	75	3.98	92.59	88	68	10.97	100.87	
506	82	75	3.05	04 77	82	66	10.66	04.77	82	59	30.65	102 72	
S06	88	78	1.50	84.77	88	70	5.08	94.77	88	62	14.81	103.73	
0.07	82	77	3.40	85.58 -	82	68	11.25	05.07	82	62	26.36	102.00	
S07	88	80	1.64		88	72	5.32	95.07	88	66	12.96	102.99	
<b>G</b> 00	82	74	4.27	00.57	82	68	9.50	106.00					
S08	88	74	2.33	88.57	88	68	6.67	106.82		Inva	alid Data		
VD2	76	82	3.65	70.00	82	81	3.82	0676	82	74	9.67	02.12	
VB2	82	85	1.66	79.86	88	83	1.91	86.76	88	78	4.35	93.12	
G00	76	81	3.72	00.00	82	80	3.83	06.47	82	76	7.36	01.00	
S09	82	84	1.78	80.28	88	82	1.82	86.47	88	80	3.51	91.80	
010	76	81	3.20	70.00	82	80	3.19	05.00	82	75	6.68	00.57	
S10	82	83	1.47	78.89	88	83	1.59	85.20	88	78	3.07	90.57	
011	76	80	3.79	70.02	82	80	3.59	05.02	82	75	7.03	00.07	
S11	82	83	1.19	78.82	88	81	1.70	85.93	88	79	3.23	90.96	
G1 <b>0</b>	76	79	3.56	70.07	82	78	3.55	06.02	82	75	6.98	00 71	
S12	82	82	1.72	79.97	88	81	1.74	86.03	88	78	3.15	90.71	
G12	76	76	4.15	01.00	82	75	4.35	07.50	82	71	9.00		
S13	82	80	1.96	81.08	88	79	2.09	87.58	88	75	4.38	93.74	
C1.4	82	74	2.71	02.70	82	67	7.57	01.07	82	61	17.34	00.07	
S14	88	77	1.30	83.70	88	72	3.57	91.86	88	66	8.35	98.95	
617	82	80	2.27	02.20	82	71	5.83	00.07	82	69	12.91	06.41	
S15	88	82	1.15	82.28	88	76	2.80	89.97	88	72	6.18	96.41	
616	82	66	4.20	07.20	82	62	8.58	101.40		- [mr-s1: 1	Data		
	88	69	2.02	87.30	88	63	5.64	101.49		Invalid	Data		

Table 3-4 DSR Tests Results for PAV Aged Samples

DSR testing on samples 8 and 16 after 60 hours of aging did not result in valid data. Large complex modulus values were measured during the first few iterations, but the measurements dropped rapidly and finally converged to very low values. In some cases, the target strain of 10% was not achieved with the maximum stress that the DSR could apply. These samples also exhibited unusual physical behavior. Although they were expected to be extremely hard after 60 hours of aging, they were easily cut off by a spatula due to their brittle condition at room temperature. This is an indication of weak cohesion, and of the poor shear and tensile strengths of the binder. These samples had very low values of  $\delta$  even after the first PAV cycle. This infers that they exhibit less viscous behavior when compared with conventional asphalt binders.

Table 3-6 summarizes the results from the PAV aging experiment and shows the increase in the high PG that takes place in each stage. Critical PAV values are also presented. Figure 3-5 shows the variations of high PG with aging time.

The critical PAV time was calculated for samples as a measure of durability. This parameter is defined as the PAV aging time it takes to increase the high PG from 70 °C to 95 °C. PAV times corresponding to high PGs of 70 °C and 95 °C were obtained by interpolation or extrapolation.

The durability index  $(I_d)$  was defined as the critical PAV time of the recycled binder to that of the virgin binder used in the mixture. This index can express the effect of a certain combination of RAP and rejuvenator on the durability of the binder. An  $I_d$  greater than 1.0 indicates an improved durability, while a smaller value shows relatively poor durability.

		H	ligh PG (		1 imes u		~	PG (°C)	Critical	Durability
Sample	Not Aged	RTFO Aged	20 Hour PAV	40 Hour PAV	60 Hour PAV	0 - 20 Hours	20 - 40 Hours	40 - 60 Hours	PAV Time (Hours)	Index (Id)
VB1	70.19	70.97	83.05	92.36	100.53	12.08	9.31	8.17	48.07	1.00
S01	69.47	70.17	81.39	89.51	95.78	11.22	8.11	6.27	57.83	1.20
S02	69.97	71.88	82.07	89.36	96.63	10.19	7.29	7.27	59.21	1.23
S03	70.96	72.10	83.43	91.78	97.46	11.33	8.35	5.69	55.04	1.15
S04	70.72	73.58	83.50	91.36	96.08	9.92	7.86	4.72	62.65	1.30
S05	70.55	71.32	84.28	92.59	100.87	12.96	8.31	8.28	47.86	1.00
S06	69.74	70.65	84.77	94.77	103.73	14.12	10.00	8.96	41.42	0.86
S07	70.87	71.92	85.58	95.07	102.99	13.67	9.49	7.91	42.62	0.89
S08	70.59	70.65	88.57	106.82	-	17.92	18.25	-	27.77	0.58
VB2	69.18	68.39	79.86	86.76	93.12	11.47	6.91	6.36	62.97	1.00
S09	69.96	69.37	80.28	86.47	91.80	10.91	6.19	5.33	70.84	1.12
S10	69.04	69.42	78.89	85.20	90.57	9.47	6.32	5.37	75.29	1.20
S11	69.36	69.33	78.82	85.93	90.96	9.48	7.11	5.03	74.66	1.19
S12	70.07	70.63	79.97	86.03	90.71	9.34	6.06	5.72	79.68	1.27
S13	68.29	68.09	81.08	87.58	93.74	12.99	6.50	6.16	61.16	0.97
S14	69.55	68.61	83.70	91.86	98.95	15.09	8.16	7.09	47.01	0.75
S15	69.85	69.16	82.28	89.97	96.41	13.11	7.69	6.44	54.32	0.86
S16	70.00	68.90	87.30	101.49	-	18.40	14.19	-	29.66	0.47

 Table 3-5 The Increase in High PG of Samples after Each Level of Aging and Resulting

 Critical PAV Times and Durability Indices

#### 3.6.2 Discussions

The following trends were observed:

- 1. RA1 caused slower aging of the binders and increased the critical PAV time. A meaningful correlation exists between increasing the dosage of RA1 and the durability index for both binders (see Figure 3-6). The slower aging of samples containing RA1 can be identified in the aging curves presented in Figure 3-5.
- RA2 caused faster aging of the binders and increased the critical PAV time. A meaningful correlation exists between increasing the dosage of RA2 and decreasing the durability index.

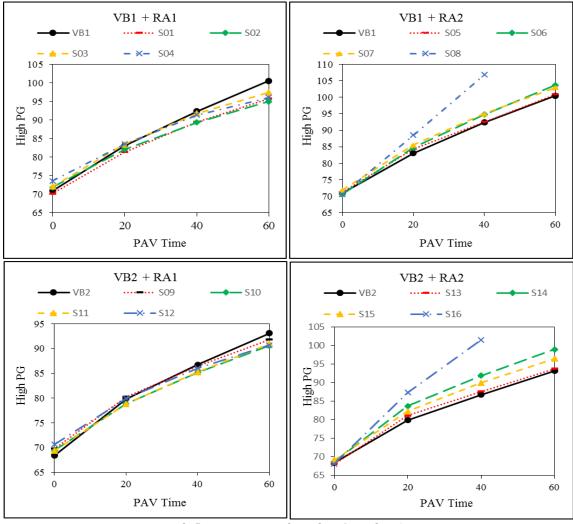


Figure 3-5 Variations of High PG with PAV Time

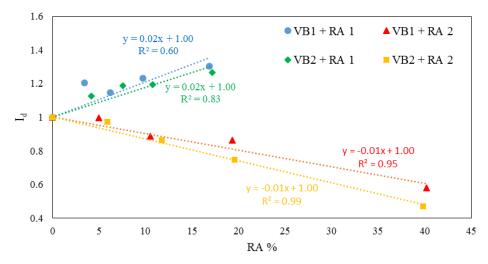
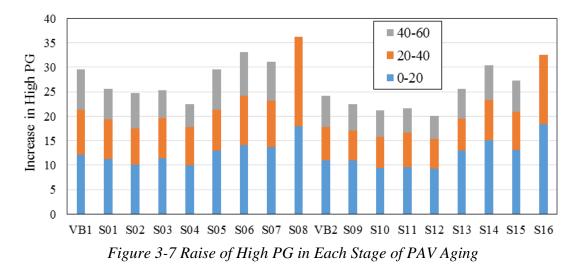


Figure 3-6 Variations of the Durability Index with Recycling Agent Content

3. The type of virgin binder had a significant influence on the rate of aging and the critical PAV time. VB1 aged considerably faster than VB2. This difference can be identified by comparing samples S01 to S08 with samples S09 to S16 in Figure 3-7. In addition, the aging of samples that contained VB1 was more influenced by rejuvenators.



- 4. The rate of aging decreased with an increase in PAV time. The first cycles increased the high PG by an average of 12 °C. This increase was respectively 9 °C and 6 °C for the second and third cycles. Figure 3-7 shows the increase in high PG for each sample in each stage of aging.
- 5. A 20-hour cycle of PAV simulates almost eight years of field aging [34]. Therefore, to estimate pavement service life (the service time before excessive binder aging), every hour of PAV aging time was assumed to correspond to 0.4 years of field aging. Based on this assumption, the field longevity of the binders was estimated, as illustrated in Figure 3-8. The right vertical axis in this figure indicates service life.

6. Samples 8 and 16, which contain large quantities of RA2, aged extremely fast. Their aging after 40 hours was more than that of any other sample after 60 hours. Also, relatively small phase angles were obtained.

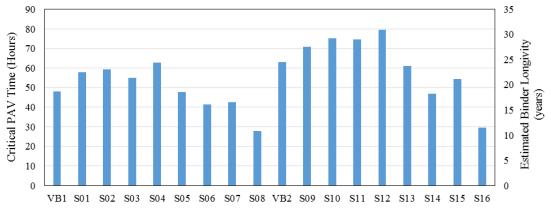


Figure 3-8 Critical PAV Time (Left Axis) and Estimated Longevity (Right Axis) of Samples

## **3.7 BBR Tests**

The BBR test evaluates an asphalt binder's low temperature cracking resistance. The stiffness is obtained by applying a point load on a small asphalt beam and measuring the deflection at 8, 15, 30, 60, 120, and 240 seconds. The output of the BBR consists of two parameters:

- *Creep stiffness (S)*, which is a measure of thermal stresses in the asphalt due to contraction.
- The *m-value*, which is the slope of the creep stiffness master curve and indicates the ability of the asphalt to relieve stresses through plastic deformation.

The BBR test was performed on all samples after the standard (20 hours) and ultimate (60 hours) of PAV aging. The results are displayed in Tables 3-6 and 3-7. The PG system specifies the following requirement at 60 seconds and at a temperature 10 °C higher than the low temperature specification. This is based on the time-temperature superposition

principle that allows shortening the loading time by increasing the temperature. For a PG 67-22 binder, these requirements should be met at -12 °C for a 20-hour PAV-aged residue.

- $S \le 300 \text{ MPa}$  Equation 3-3
- M-value  $\geq 0.300$  Equation 3-4

In Table 3-7, color codes are used to show whether these criteria are met. Green shows passing, red shows failure and yellow shows that values are very close to the criteria.

#### 3.7.1 Discussions

The following trends were observed:

- 1. For all samples, the m-value criterion was more critical and dominated the determination of the low temperate PG.
- 2. Virgin binders did not meet the m-value criterion for PG 67-22, but they were very close (0.299 for VB1 and 0.291 for VB2). VB1 had a better low-temperature performance compared to VB2. It had a smaller creep stiffness and a higher m-value, despite VB1's higher stiffness at high temperature and greater high temperature PG.
- 3. The samples with RA1 passed the criteria for PG 67-22. Samples with RA2, on the other hand, did not meet these criteria and yielded lower m-values.
- 4. Generally, the addition of RA1 did not significantly change the creep stiffness. The RA2, however, caused a fast drop in the stiffness. The higher the dosage of RA2, the smaller the values of creep stiffness (Figure 2-9). A smaller amount of low-temperature creep stiffness showed that less thermal stresses are expected. However, the very small stiffness found in the samples that contain a large dosage of RA2 is an indication of the detrimental behavior of RA2 when applied at a large dosage.

Table 3-6 BBR Test for 20-hour PAV-aged Sam											1	
Sample	VB	RA	RAP	RAP	RA%	Test Temp. (°C)	S	S Grade	m-	m-Value	Low Temp.	
Sumple	, ,	Type	1111	Content	1.1.70	Temp. (°C)		(°C)	value	Grade (°C)	<b>PG</b> (°C)	
						-6	161		0.362			
		VB1			0	-12	205	-25.54	0.299	-21.90	-21.90	
				•		-18	391		0.256			
						-6	109		0.370			
1			_	20%	3.42	-12	191	-26.15	0.313	-23.37	-23.37	
			RAP1			-18	367		0.267			
_			$\mathbb{R}^{J}$			-6	78.7		0.377			
2				40%	9.68	-12	173	-27.07	0.319	-24.00	-24.00	
		RA1				-18	332		0.262			
		R		• • • • •	<i></i>	-6	116		0.358	•• • • •		
3			2	20%	6.19	-12	203	-25.62	0.309	-23.10	-23.10	
			RAP2			-18	388		0.253			
			$\mathbb{R}^{\prime}$			-6	90.4		0.364			
4	_			40%	16.82		194	-25.57	0.314	-23.68	-23.68	
	B ]					-18	404		0.251			
_	VB			• • • • •		-6	74.4	• • • • •	0.345	• • • •	• • • • •	
5			_	20%	4.93	-12	153	-28.61	0.285	-20.50	-20.50	
			٩P			-18	282		0.255			
			RAP1			-6	39.6		0.340			
6			2		40%	19.33		91	-39.49	0.267	-19.29	-19.29
		RA2				-18	137		0.248			
		R				-6	54.2		0.321			
7				20%	10.48		114	-33.03	0.272	-18.57	-18.57	
			RAP2			-18	193		0.246			
			RA			-6	Invalid		Invalid			
8				40%	40.18		34.1	Invalid	0.241	Invalid	Invalid	
						-18	55.1		0.231			
						-6	149		0.352			
		VB2	2		0	-12	301	-21.95	0.291	-21.11	-21.11	
						-18	454		0.262		ļ	
						-6	112		0.360			
9				20%	4.14	-12	215	-25.31	0.303	3 -22.62	-22.62	
			RAP1			-18	393		0.274			
			RA			-6	83.6		0.365			
10				40%	10.73		180	-26.34	0.327	-24.84	-24.84	
		A1				-18	365		0.270			
		RA1				-6	98.4		0.351			
11				20%	7.53	-12	181	-25.99	0.317	-23.96	-23.96	
			P2			-18	387		0.265			
			RAP			-6	85.4		0.373			
12				40%	17.18		175	-26.32	0.329	-25.63	-25.63	
	3 2					-18	370		0.281			
	VB				_	-6	76.3		0.346		7	
13				20%	5.88	-12	169	-27.65	0.287	-20.68	-20.68	
			$\mathbf{P1}$			-18	311		0.268			
			RAP1			-6	36.6		0.345			
14			_	40%	19.54	-12	74.8	-36.11	0.293	-21.19	-21.19	
		RA2				-18	135		0.260			
						-6	51.2		0.329			
15				20%	11.75		115	-33.58	0.274	-19.16	-19.16	
			P2			-18	189		0.235		-19.10	
			RAP2			-6	Invalid		NA			
16			Я	40%	39.79	-12	37.5	Invalid	0.271	Invalid	Invalid	
						-18	47.5		0.266			
	. I				l							

Table 3-6 BBR Test for 20-hour PAV-aged Samples

PA DA					-211 1	Test			1804 54	4	Low Temp. PG	
Somplo	VD		RAP	RAP	RA%		S	S Grade	m-	PG (°C)	Increase (°C)	
Sample	۷D		KAP	Content	KA 70	Temp.	ъ	(°C)	value			
		e				(°C)	1.47	· ,		(m-value)	20 to 60 hours	
					0	-6	147	24.27	0.282	14.40	7.42	
		VB1			0	-12	235	-24.27	0.211	-14.48	7.43	
						-18	448		0.178			
1				200/	2 12	-6 -12	159 281	22 70	0.292 0.256	1467	<u> </u>	
1			Ξ	20%	3.42	-12	465	-22.78	0.230	-14.67	8.70	
			RAPI									
2			Я	40%	9.68	-6 -12	135 248	-23.73	0.298	-15.74	8.26	
2		1		4070	9.00	-12	480	-23.13	0.182	-13.74	0.20	
		RA1				-18 -6	152		0.182			
3		К		20%	6.19	-12	273	-23.05	0.257	-15.54	7.56	
5			2	2070	0.19	-12	468	-23.05	0.238	-15.54	7.50	
			RAP2			-18	149		0.301			
4			R	40%	16.82	-12	269	-23.21	0.266	-16.17	7.51	
4	1			4070	10.62	-12	461	-23.21	0.232	-10.17	7.51	
	VB					-6	126		0.273			
5				20%	4.93	-12	193	-26.60	0.228	-12.40	8.10	
5			5	2070	ч.95	-12	343	-20.00	0.190	-12.40	0.10	
			RAP1			-6	94.9		0.270			
6			К	40%	19.33	-12	144	-33.39	0.234	-11.00	8.29	
Ū		5		1070	17.55	-18	212	55.57	0.202	11.00	0.27	
		RA2				-6	108		0.268			
7		Π	RAP2	20%	10.48	-12	161	-28.66	0.227	-11.32	7.25	
,				2070	10.10	-18	282	20.00	0.194	11.52	1.20	
			[Y]			-6	Invalid		Invalid			
8			R	40%	40.18	-12	39.4	Invalid	0.245	Invalid	-	
Ŭ				1070	10.10	-18	61.3	mvunu	0.230	mvunu		
						-6	184		0.276			
		VB2			0	-12	341	-19.84	0.215	-13.64	7.48	
					-	-18	487	-17.04	0.183			
						-6	182		0.294			
9				20%	4.14	-12	319	-20.96	0.264	-14.80	7.82	
			P1			-18	455		0.232			
			RAP1			-6	144		0.305			
10			4	40%	10.73	-12	267	-23.37	0.270	-16.86	7.98	
		11				-18	445		0.239			
		RA1				-6	173		0.298			
11				20%	7.53	-12	307	-21.68	0.275	-15.48	8.48	
			RAP2			-18	472		0.248			
			<b>RA</b>			-6	137		0.312			
12			_	40%	17.18	-12	261	-23.42	0.274	-17.89	7.73	
	3 2					-18	470		0.246			
	VB					-6	129		0.259			
13			_	20%	5.88	-12	264	-24.52	0.210	-10.98	9.70	
			RAP1			-18	358		0.191			
			$\mathbb{R}^{A}$			-6	77.9		0.256			
14		RA2		40%	19.54	-12	126	-35.55	0.234	-4.00	17.19	
						-18	185		0.214			
						-6	81.2		0.261		0.5-	
15			2		11.75	-12	193	<del>93</del> -29.32 77	0.226	-9.31 97	9.85	
			\P.			-18	277		0.197			
			RAP2			-6	45.8	10	0.238			
16					40% 3	40% 39.79	-12	71.9	-43.56	0.219	Invalid	Invalid
						-18	107		0.200			

Table 3-7 BBR Test for 60-hour PAV-aged Samples

5. The RA1 increased the m-value, and the RA2 decreased it. Therefore, samples with RA1 had a lower low temperature PG. This effect was more significant when the RA content was higher (Figure 3-9). A higher m-value shows a binder with a more viscous behavior and a greater ability to relieve stresses. The less viscous behavior of samples containing RA2 was also observed in DSR tests where these samples had lower phase angles.

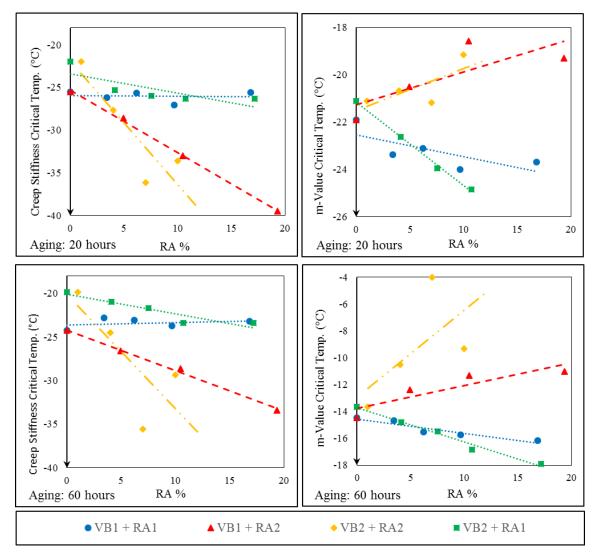


Figure 3-9 Variations of BBR Critical Temperatures with the RA Content

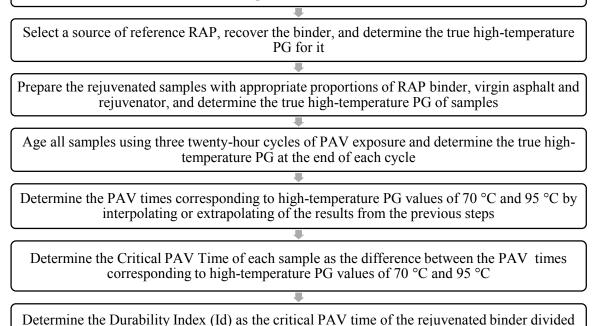
- 6. Alike in the DSR experiment, samples 8 and 16 did not output valid data. The samples were very soft, and they broke under the BBR load at -6 °C. Also, their results at -12 °C and -18 °C yielded critical temperature values that were out of acceptable ranges.
- 7. Applying the extended PAV aging (60 hours) increased the creep stiffness and decreased the m-value. The change in the m-value was more significant. While the creep stiffness critical temperature increased by 3 °C on average, the average rise in the m-value critical temperature was 9 °C.
- Since the 60-hour aged samples were excessively hard, it was difficult to pour the BBR mold with these samples. Therefore, they were heated to 175 °C for ten minutes to achieve the required fluidness.

## **3.8 Recommended Method for Durability Evaluation of Rejuvenators**

As mentioned in the research objective statement of this dissertation, "A successful rejuvenation is defined as a rejuvenation process that enhances the properties of the aged asphalt in a way that its performance is similar to or better than a reference virgin asphalt. Therefore, a successfully rejuvenated binder cannot be excessively aged in a shorter time span than a reference virgin asphalt. This time span is affected by the initial conditions of the asphalt binder and the rate of aging. It was shown in this research that the type of the rejuvenator has a significant influence on the aging rate and longevity.

The flowchart in Figure 3-10 shows the procedure to determine the critical PAV time and durability index. It is recommended that this process is implemented by considering one source of virgin asphalt and RAP, and several rejuvenators that can potentially be used.

Select a source of virgin asphalt as the reference virgin asphalt and determine the true hightemperature PG for it



to that of the reference virgin asphalt

Figure 3-10 The process to Determine Critical PAV Time and Durability Index

By assuming a linear relation between the PAV aging time and the changes in the PG, it can be concluded that:

$$\frac{PAV Time}{\text{Increase in High PG}} = \frac{Critical PAV Time}{95^{\circ}\text{C} - 70^{\circ}\text{C}} = \frac{Critical PAV Time}{25^{\circ}\text{C}}$$
Equation 3-5

On the other hand, the required PG for the rejuvenated binder in grade that prevents excessive aging during the pavement design life. Therefore, the maximum initial grade for the rejuvenated binder is equal to:

Rejuvenated Binder Max PG = Failure PG – Increase in PG during Pavement's Design Life Where the Failure PG is the PG that is considered as a failure (For instance 95 °C, based on FDOT's experience). The Increase in PG during pavement's design life can be estimated as the increase that happens in a PAV exposure time equal to  $\frac{Pavement Design Life}{0.4}$ , based on [34]. Therefore, rejuvenated binder maximum PG is equal to:

Rejuvenated Binder Max PG = Failure PG - 
$$\frac{\frac{Pavement Design Life}{0.4}}{\frac{Critical PAV Time}{25}}$$
Equation 3-6

Rejuvenated Binder Max PG = Failure PG -  $62.5 \frac{Pavement's Design Life}{Critical PAV Time}$  Equation 3-7

Therefore, the required extent of softening is dependent on the critical PAV time, which is dominated by the type of the rejuvenator. The value gained from using a particular rejuvenator can be assessed by the durability index, which indicates the PAV time in comparison to a reference virgin asphalt. If there is a roughly linear relation between the rejuvenator dosage and the reduction in the PG, the required rejuvenator content, can be adjusted based on the durability index (Equation 3-8). This adjustment, does not consider the durability effect entirely, but can be used for a rough estimation. The cost comparison between rejuvenators shall be conducted using the adjusted rejuvenator content.

Adjusted Rejuvenator Dosage = 
$$\frac{\text{Conventional Rejuventor Dosage}}{\text{Durability Index}}$$
 Equation 3-8

In which the conventional rejuvenator dosage is the dosage that is determined without considering the durability effect of the rejuvenator.

Alternatively, the rejuvenator dosage can be determined using the process explained in the flowchart in Figure 3-1. This method determines the required PG of the binder and the rejuvenator dosage based on the design life of the pavement and the critical PAV time of the binder.

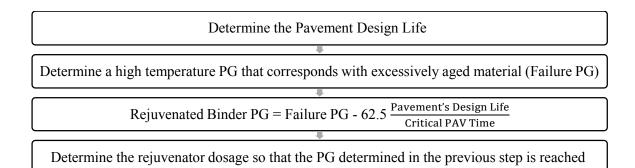


Figure 3-11 The Process to Determine the Rejuvenator Dosage Based on Critical PAV Time

#### **3.8.1** Example Calculations for Implementing the Proposed Method

In this section, the calculations for determining the critical PAV time, durability index, and adjusted rejuvenator dosages is reported for samples #2 and #6.

Critical PAV Time and Durability Index

#2:

PAV time corresponding to high PG of 70 °C =  $20 \times \frac{70-72.10}{83.43-72.8} = -3.71$  hours

PAV time corresponding to high PG of 95°C =  $40 + 20 \times \frac{95-91.78}{97.46-91.78} = 51.34$  hours

Critical PAV Time = 51.34 - (-3.71) = 55.04 hours

Durability Index  $=\frac{55.04}{48.07} = 1.15$ 

#6:

PAV time corresponding to high PG of 70 °C =  $20 \times \frac{70-70.65}{83.43-70.65} = \%$  7.9 = -0.91 hours

PAV time corresponding to high PG of 95 °C =  $40 + 20 \times \frac{95-94.77}{103.73-94.77} = 40.50$  hours

Critical PAV Time = 40.50 - (-0.91) = 41.41 hours

Durability Index  $=\frac{41.41}{48.07} = 0.86$ 

#### Adjusted Rejuvenator Dosage

Based on durability index (Equation 3-8)

- #2: Adjusted dosage  $=\frac{\%9.7}{1.23} = \%$  7.9 #6: Adjusted dosage  $=\frac{\%19.3}{0.86} = \%$  22.4 Based on critical PAV Time (Figure 3-11) Failure PG: 95 °C Design Life: 20 years The required initial PG for the samples is determined based on Equation 3-6
- #2: Rejuvenated Binder PG = 95- 62.5  $\frac{20}{55.04}$  = 72.29 °C
- #6: Rejuvenated Binder PG = 95- 62.5  $\frac{20}{41.41}$  = 64.81 °C

The grade of the softer binder is calculated using charts similar to those in Figure 3-2, and rejuvenators contents are determined based on the softening curves in Figure 3-3.

- #2: Softer PG Grade = 59.18°C  $\rightarrow \frac{RA}{VB} = \%11.06 \rightarrow RA \text{ dosage} = \% 6.64$
- #6: Softer PG Grade =  $46.72^{\circ}C \rightarrow \frac{RA}{VB} = \%72 \rightarrow RA \text{ dosage} = \% 43.2$

It can be observed that the rejuvenator dosages determined by the procedure explained in Figure 3-11, are more affected by the durability properties of the rejuvenator. In some cases, such as for the case of sample #6, the use of this method yields large dosages that are impractical. It can be concluded from such circumstances that the tested rejuvenator ,RA2 in this example, is not applicable for that particular application.

# CHAPTER 4: BLENDING EFFECTIVENESS AND HOMOGENEITY

## 4.1 Introduction

This chapter explains the work performed to investigate the effectiveness of blending of the recycling agent or rejuvenator with the aged asphalt and proposes methods to ensure acceptable binder homogeneity for recycled mixtures. When the recycling agent is applied to the RAP, the outer layer of the asphalt film is directly exposed to it. Therefore, this layer is affected almost immediately. Afterward, the recycling agent starts to penetrate the inner layers through the diffusion process. The extent and rate of diffusion depend on various parameters, including the temperature and material type. This part of the research investigates the diffusion of rejuvenators into the old asphalt and the stiffness gradient of the rejuvenated asphalt binder film that surrounds RAP aggregates.

## 4.2 Methodology and Sample Preparation

The staged extraction method was used to separate the layers of asphalt. The extraction was done in three stages. In each stage, the samples were soaked in Trichloroethylene for a designated time, as shown in Table 4-1. Then, the solvent that had dissolved a portion of the asphalt binder was extracted using a centrifuge extractor (Figure 3-1-a). The last extraction consisted of two subsequent washes to make sure that all of the remaining binder was extracted. The extracted liquid was placed in a centrifuge with an 800 relative centrifugal force (RCF) for 30 minutes to make suspended fine aggregates sediment. Thereafter, the solvent was distilled using a rotary evaporator (Figure 3-1-b) and the binder

was recovered. The binder recovered from each stage of extraction represents the corresponding layer of asphalt. The last extraction was done in two washes to make sure that all of the remaining binders were recovered.

Extraction Number	Solvent Soaking Time	Sampled Asphalt Layered		
X1	1 Minute	Outermost		
X2	3 Minutes	Intermediate		
X3	45 Minutes 15 Minutes	Innermost		

Table 4-1 Extraction Stages and Corresponding Times

Figure 4-1 shows the appearance of the sample in each stage of extraction. Before the first extraction, aggregates are completely coated by a relatively thick layer of asphalt. The first extraction washes a large portion of the asphalt film away, leaving a thinner layer. After the second extraction, only a very thin layer of asphalt remains on the aggregates. The last extraction, which includes two washes, extracts almost all of the remaining asphalt binder.

Table 4-2 shows the composition of the samples used in this study, as well as the time and temperature of their mixing, and the aging they underwent before extraction. The initial plan for this experiment involved testing 16 samples (#1 to #16). Ten supplementary samples were added later to provide necessary data to explain the trends observed for the first sixteen samples.



Before Extraction

1<sup>st</sup> Extraction

2<sup>nd</sup> Extraction

3<sup>rd</sup> Extraction

Figure 4-1 The Appearance of Samples at Each Stage of the Extraction Process

Table 4-2 Composition,	Mixing	Conditions.	and Aging	of Samples
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Sample Number		Material	Mixing Time (Minutes)	Mixing Temp. (°C)	Aging
#1				149	5 Days at 85°C
#2			2		_
#3	-	RA1 (Oil)		165	1 Hour at 165 °C
#4				-	2 Hours at 165 °C
#5	RAP 1		5	140	5 Decement 050C
#6 #7	-			149	5 Days at 85°C
#7		RA3 (Emulsion)	2		1 Hour at 165 °C
#9				165	2 Hours at 165 °C
#10	-		5		2 110 115 11 105 0
#11			2	1.10	
#12		RA1	5	149	
#13			2	165	5 Days at 85°C
#14	RAP 2		2	149	5 Duys at 05 C
#15		RA3	5	147	
#16			2	165	
#17	RAP1	RA1	2	165	
#18	RAP1	RA3	2	165	No Aging
N1	V	B 1 + Aggregate	5	165	No Aging
N2	V	B 2 + Aggregate	5	165	
N3	V	B 1 + Aggregate	5	165	5 Dava at 95°C
N4	V	B 2 + Aggregate	5	165	5 Days at 85°C
N5	VB 1 + Aggregate		5	165	1 Hour at 165 °C
N6	VB 2 + Aggregate		5	165	1 110ul at 105 C
R1		RAP1	None	NA	No Aging
R2		RAP2	None	NA	No Aging

The material used in this experiment included two types of RAP (RAP1 and RAP2), two recycling agents (rejuvenators), two types of PG 67-22 non-modified asphalt binder (VB1 and VB2) and limestone aggregates recovered from RAP1.

All of the materials, except for the RA3, were similar to those used in the long-term aging study (Chapter 3). The RA3 was similar to the rejuvenator CWE that was used in Chapter 2. This rejuvenator is a water-based emulsion manufactured from Naphthenic crude, which is a wax-free, low pour point crude with high solvency ability. The residue content of this emulsion was measured at 60%, and its residue has a dynamic viscosity of 200-500 cSt at 60 °C. Therefore, the two rejuvenators that exhibited the best performance in the study explained in Chapter 2 (HPE and CWE) were used in this experiment.

In order to prepare samples, the RAP or aggregate was heated to the designated mixing temperature for 45±5 minutes in a laboratory mixer bowl. Then, a predetermined amount of the recycling agent or virgin binder was added, and the mixing continued for the designated time shown in Table 4-2. The final weight of each sample was 1100±20 gram.

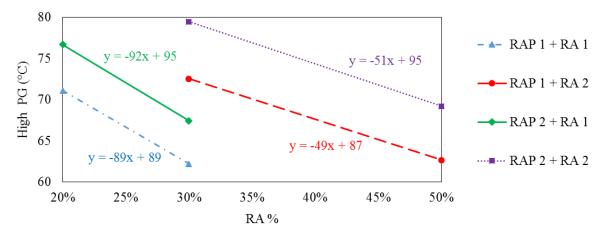


Figure 4-2 Softening Curves of Rejuvenators

The rejuvenator dosage of samples was determined by recovering the RAP binder and establishing softening curves (Figure 4-2). The target high temperature PG of samples was  $69 \pm 1$  °C. The proportion of the rejuvenator to the total weight of the mixtures was determined by multiplying the dosage obtained from the softening curves by the binder content of RAP mixtures. Table 4-3 displays the composition of recycled mixtures.

Don Type	DA Tuno	<b>RAP Binder</b>	RA Weight	RA Weight
<b>Rap Type</b>	RA Type	Content	<b>RAP Binder Weight</b>	<b>RAP Mixture Weight</b>
RAP1	RA1	5 700/	0.21	0.012
KAPI	RA3	5.70%	0.35	0.02
RAP2	RA1	( (0)/	0.27	0.018
	RA3	6.69%	0.48	0.032

Table 4-3 Rejuvenator Dosage of Recycled Samples

## 4.3 **Results**

All of the 26 samples were extracted in three stages. The binder that was recovered in each stage was tested with the DSR. Table 4-4 presents the results from these tests. For each sample, the amount of binder recovered in each extraction was determined. In addition, the high PG values of each layer, which were obtained from two DSR tests, are presented. The weighted average PG of the sample was calculated using Equation 4-1. This average represents the whole binder and is similar to the PG value that would be obtained if the whole binder were recovered in a single stage.

$$PG_{ave} = a_1 PG_{x1} + a_2 PG_{x2} + a_3 PG_{x3}$$
 Equation 4-1

In which:

$$a_i = \frac{\text{The mass of the binder recovered in } i^{\text{th stage of extraction}}}{\text{The mass of total recoverd binder}}$$
Equation 4-2

 $PG_{xi}$  = The high temperature PG of the  $i^{th}$  layer

In order to compare the stiffness of asphalt layers, the following parameters are presented in Table 4-4:

- PG<sub>i</sub> PG<sub>ave</sub>: The difference between the PG of each layer and the average PG of all layers
- $\frac{PG_i}{PG_{ave}}$ : The normalized PG of each layer
- PG<sub>max</sub>- PG<sub>min</sub>: The gap between the minimum and the maximum PG

Two parameters were defined to provide a quantitative description of the stiffness gradient and homogeneity of samples. These parameters are Stiffness Gradient Factor (SGF) and homogeneity index (I<sub>h</sub>) and are defined by Equations 3 and 4, respectively.

Stiffness Gradient Factor (SGF) = 
$$\frac{PG_1 - PG_3}{PG_{ave}} \times 100\%$$
 Equation 4-3

$$I_{h} = 1 - \frac{Maximum PG_{i} - Minimum PG_{i}}{PG_{ave}}$$
 Equation 4-4

The SGF is a measure of the stiffness gradient of the asphalt film coating the aggregates and shows how stiff the outer layer is compared to the inner layer. A positive value of SGF means that the outer layer is harder, while a negative SGF indicates that the outer layer is relatively softer. The homogeneity index is an indication of the level of homogeneity within the asphalt binder. An I<sub>h</sub> close to one shows a very homogeneous binder, while low values, departing farther from one, indicate an increasing lack of homogeneity.

	Extraction Number	(grams)	ai (Eq 4.2)	PGi	PG <sub>ave</sub> (Eq 4.1)	PGi – PGave	$\frac{PG_i}{PG_{ave}}$	PG <sub>max</sub> - PG <sub>min</sub>	SGF(%) (Eq. 4.3)	I <sub>h</sub> (Eq. 4.4)
	X1	35.17	0.56	80.97		0.23	1.00			
#1	X2	16.04	0.25	82.58	80.75	1.83	1.02	4.91	2%	0.94
	X3	12.11	0.19	77.67		-3.08	0.96			
	X1	34.79	0.56	79.89		0.78	1.01			
#2	X2	14.57	0.24	80.83	79.11	1.72	1.02	5.90	6%	0.93
	X3	12.50	0.20	74.93		-4.18	0.95			
	X1	47.16	0.65	78.27		-0.37	1.00			
#3	X2	13.56	0.19	82.00	78.64	3.36	1.04	5.81	3%	0.93
	X3	11.41	0.16	76.19		-2.45	0.97			
	X1	35.29	0.58	81.36		0.08	1.00			
#4	X2	16.28	0.27	84.31	81.28	3.03	1.04	8.56	7%	0.89
	X3	9.43	0.15	75.75		-5.53	0.93			
	X1	23.26	0.40	78.78		-1.29	0.98			
#5	X2	20.68	0.36	83.30	80.06	3.23	1.04	5.89	2%	0.93
	X3	13.93	0.24	77.41		-2.65	0.97			
	X1	46.17	0.66	77.81		-0.15	1.00			
#6	X2	15.31	0.22	79.29	77.97	1.32	1.02	2.85	2%	0.96
	X3	8.61	0.12	76.44		-1.53	0.98			
	X1	43.41	0.62	75.84		-1.52	0.98			
#7	X2	17.69	0.25	82.08	77.36	4.73	1.06	6.64	1%	0.91
	X3	9.23	0.13	75.44		-1.92	0.98			
	X1	40.16	0.56	74.28		-1.64	0.98			
#8	X2	18.86	0.26	81.02	75.93	5.10	1.07	7.51	1%	0.90
	X3	12.43	0.17	73.51		-2.42	0.97			
	X1	40.24	0.59	83.13		1.57	1.02			
#9	X2	15.45	0.23	83.72	81.56	2.16	1.03	10.12	12%	0.88
	X3	12.15	0.18	73.60		-7.96	0.90			
	X1	50.36	0.66	77.74		0.20	1.00			
#10	X2	15.45	0.20	79.43	77.54	1.89	1.02	5.49	5%	0.93
	X3	10.96	0.14	73.94		-3.60	0.95			
	X1	34.63	0.55	74.12		-0.89	0.99			
#11	X2	15.10	0.24	77.87	75.01	2.86	1.04	3.77	0%	0.95
	X3	13.22	0.21	74.10		-0.92	0.99			
	X1	40.10	0.51	74.90		-1.59	0.98			
#12	X2	21.70	0.27	79.53	76.50	3.04	1.04	4.63	-2%	0.94
	X3	17.44	0.22	76.38		-0.12	1.00			
	X1	32.77	0.53	71.57		-2.90	0.96			
#13	X2	23.06	0.38	78.70	74.47	4.22	1.06	7.13	-3%	0.90
	X3	5.52	0.09	74.06	,,	-0.41	0.99	,		
	X1	46.12	0.62	81.94		-0.15	1.00			
#14	X2	16.41	0.22	84.82	82.09	2.73	1.03	5.94	4%	0.93
	X3	11.77	0.16	78.88		-3.21	0.96		.,.	
	X1	27.20	0.40	79.94		1.46	1.02			
#15	X1 X2	21.43	0.32	79.98	78.48	1.49	1.02	5.31	7%	0.93
	X2 X3	18.78	0.32	74.67	, 0.10	-3.81	0.95	0.01	, , 0	0.75
	XI	46.37	0.26	80.68		0.81	1.01			
111.6	X1 X2	13.15	0.00	79.53	-	-0.35	1.00	2.01	<b>5</b> 0 (	0.05
#16					79.88			3.91	5%	0.95
	X3	10.56	0.15	76.77		-3.11	0.96			

Table 4-4 Results from Staged Extraction and DSR Tests

Sample Number	Extraction Number	Recovered Binder (grams)	ai (Eq 4.2)	PGi	PG <sub>ave</sub> (Eq 4.1)	PGi-	$\frac{PG_i}{PG_{ave}}$	PG <sub>max</sub> - PG <sub>min</sub>	SGF(%) (Eq. 4.3)	I <sub>h</sub> (Eq. 4.4)
	X1	36.86	0.65	65.66		-4.09	0.94			
#17	X2	12.52	0.22	76.42	69.75	6.66	1.10	13.13	-19%	0.81
	X3	7.46	0.13	78.79		9.04	1.13			
	X1	36.25	0.59	61.79		-6.39	0.91			
#18	X2	13.07	0.21	76.37	68.18	8.18	1.12	16.92	-25%	0.75
	X3	11.85	0.19	78.71		10.53	1.15			
	X1	48.80	0.74	69.17		0.18	1.00			
N1	X2	11.49	0.17	68.47	68.99	-0.51	0.99	0.72	1%	0.99
	X3	5.73	0.09	68.45		-0.54	0.99			
	X1	50.82	0.75	68.16		0.22	1.00			
N2	X2	10.43	0.15	67.25	67.94	-0.69	0.99	0.90	1%	0.99
	X3	6.80	0.10	67.37		-0.57	0.99			
	X1	45.19	0.71	84.36		2.65	1.03			
N3	X2	10.65	0.17	78.67	81.71	-3.04	0.96	13.81	17%	0.83
	X3	7.83	0.12	70.55		-11.16	0.86			
	X1	42.09	0.69	79.07		2.27	1.03			
N4	X2	9.61	0.16	73.90	76.80	-2.90	0.96	9.50	12%	0.88
	X3	9.33	0.15	69.56		-7.24	0.91			
	X1	41.89	0.68	80.88		2.13	1.03			
N5	X2	11.24	0.18	78.17	78.75	-0.58	0.99	11.58	15%	0.85
	X3	8.77	0.14	69.30		-9.45	0.88			
	X1	42.27	0.69	77.02		2.05	1.03			
N6	X2	10.17	0.17	72.10	74.97	-2.86	0.96	8.44	11%	0.89
	X3	9.03	0.15	68.58		-6.38	0.91			
	X1	33.46	0.66	92.60		1.32	1.01			
R1	X2	9.53	0.19	91.07	91.27	-0.20	1.00	6.76	7%	0.93
	X3	7.80	0.15	85.84		-5.44	0.94			
	X1	34.39	0.61	97.26		1.93	1.02			
R2	X2	14.90	0.27	94.79	95.33	-0.54	0.99	10.32	11%	0.89
	X3	6.93	0.12	86.94		-8.39	0.91			

Table 4-4 Continued

## 4.4 Analyses

## 4.4.1 First Sixteen Samples

The first 16 samples are 100% recycled mixtures that had undergone oven aging. Results from these samples show that there is not a large difference in stiffness between the layers.

The average  $I_h$  of 0.93 shows that these samples have a relatively homogenous asphalt binder layer. The average SGF of 3% indicates that the outer layers are slightly stiffer than the inner layers. Figure 4-3 shows the difference between the PG of each layer and the average PG for the first sixteen samples.

These values are averaged for each layer as shown by dashed horizontal lines. The outermost layers had PG values close to the average PG. The PG of inner layers were 3.1°C lower than the average and intermediate layers were 2.5°C higher than the average PG value.

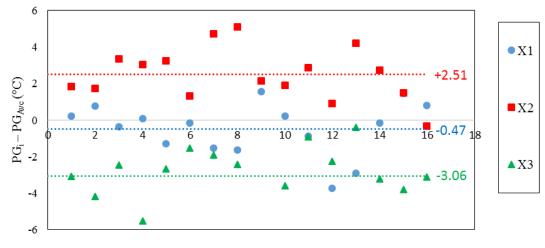


Figure 4-3 (PG<sub>i</sub> - PG<sub>ave</sub>) for First Sixteen Samples

These observations are not in accordance with the investigators' initial expectation that the outer layer is softer than the inner layers. The reason behind this hypothesis was the fact that the rejuvenator initially is only in direct contact with the outer layers inducing more rejuvenation effects on those layers. However, the initial mixing condition is not the only parameter that affects the stiffness gradient of the binder. Other factors that have a significant influence on the asphalt binder include diffusion and aging. The first sixteen

samples were exposed to oven aging, which means they experienced high temperatures for hours or days after mixing and before being extracted. Therefore, diffusion of the rejuvenator from the outer layers into the inner layers was accelerated. In addition, different layers of asphalt are not necessarily aged similarly by the heating process; rather, they might experience different levels of aging.

#### 4.4.2 Supplementary Samples

In order to study the factors mentioned previously and provide proper explanations for the observation from the first sixteen samples, the following additional samples were prepared and tested:

**#17 and #18** are similar to samples #2 and #7, respectively, except that they did not go through any aging process. These samples represent the condition of recycled samples before aging.

**N1 and N2** contain the aggregate recovered from RAP 1 and virgin binder VB1 and VB2. These samples represent the condition of a new mixture before exposure to any aging. Since these samples have a homogeneous binder, no significant difference was expected between the layers. Results fulfilled this expectation, and their homogeneity indices were over 99%.

**N3 and N4** are similar to N1 and N2, respectively, but they underwent long-term oven aging for five days at 85 °C. The purpose of testing these samples was to study the effects of long-term oven aging on samples in the absence of rejuvenation.

**N5 and N6** are similar to N1 and N2, except that they experienced short-term oven aging for one hour at 165 °C. The purpose of testing these samples was to study the effects of short-term oven aging on samples in the absence of rejuvenation.

**R1 and R2** are non-processed RAP. These samples have experienced natural field aging. The purpose of testing RAP samples was to compare oven aging with natural aging.

#### 4.4.3 Non-aged Recycled Mixtures

Samples #17 and #18 are recycled mixtures with no aging. The rejuvenator had a limited time (90±10 minutes) to diffuse into the asphalt layers of these samples. Therefore, the outer layers contained much more rejuvenator and were significantly softer. The PG of the outermost layers were 12 °C to 17 °C lower than the innermost layers, and the SGFs of these samples were large negative values ranging from -19% to -25%.

In many in-place and plant recycling circumstances, the mixing time and the storage time between mixing and placing of the asphalt concrete are short. Therefore, little aging and diffusion have occurred when the road is open to traffic and a stiffness gradient pattern similar to those for samples #17 and #18 may exist. Such a condition may lead to an unpredictable performance, and in particular, rutting is of concern due to the softer binder on the outer layers.

Table 4-5 shows how adding rejuvenators changes the properties of different layers of the asphalt. It should be noted that the RAP itself does not have a homogeneous binder film and is stiffer in the outer layer. The rejuvenator content of each layer was also estimated based on the softening curves. It can be seen that the outer layers have almost four times more rejuvenator when compared with the innermost layer.

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Extraction	RAP1		#17		#18				
Number	PGi	PGi	Drop of PG <sub>i</sub>	RA %	PGi	Drop of PG <sub>i</sub>	RA%		
X1	92.60	65.66	26.94	26	61.79	30.81	47		
X2	91.07	76.42	14.65	14	76.37	14.70	22		
X3	85.84	78.79	7.05	7	78.71	7.13	11		
Ave	91.27	69.75	21.52	21	68.18	23.09	35		

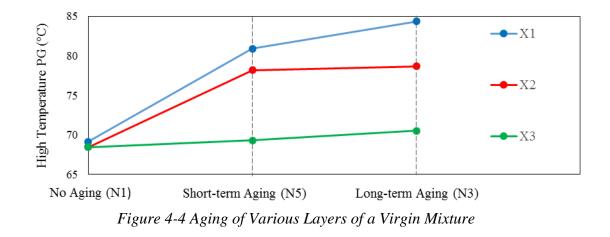
Table 4-5 Rejuvenation of Layers of Asphalt Binder

#### 4.4.4 Aging of Virgin Mixtures

Samples N1 through N6, and also R1 and R2, were used to study the effects of aging on the new mixtures. Table 4-6 summarizes the results for these samples. Results show that there is a very significant difference in the aging of the different layers. For instance, while the PG of the outer layer of sample N1 increased more than 14 °C due to long-term aging, the PG on the innermost layer increased only 2 °C (Figure 4-4). The reason is that the outer layers are more exposed to air; therefore, both of the major mechanisms that cause aging, oxidation and evaporation of volatiles occur at a faster pace. A similar trend was observed for short-term aging. As a result, new mixtures had a non-homogeneous binder film stiffness gradient after aging.

Control 1									
Sample	Aging	PG <sub>1</sub>	PG <sub>2</sub>	PG <sub>2</sub> PG <sub>3</sub>		SGF	Ih		
N1	No Aging	69.17	68.47	68.45	68.99	1%	0.99		
N5	1 hour at 165°C	80.88	78.17	69.30	78.75	15%	0.85		
N3	5 days at 85°C	84.36	78.67	70.55	81.71	17%	0.83		
Control 2									
N2	No Aging	68.16	67.25	67.37	67.94	1%	0.99		
N6	1 hour at 165°C	77.02	72.10	68.58	74.97	11%	0.89		
N4	5 days at 85°C	79.07	73.90	69.56	76.80	12%	0.88		
RAP									
R1	Natural Field	92.60	91.07	85.84	91.27	7%	0.93		
R2	Aging	97.26	94.79	86.94	95.33	11%	0.89		

Table 4-6 Aging of Virgin Mixtures



RAP samples had a stiffness gradient pattern almost similar to artificially-aged samples. However, there was a smaller gap between the outer and inner layer. The reason is that their aging mechanism is different from oven heating, and takes place over a longer time span.

### 4.4.5 Aging of Recycled Mixtures

Samples #2, #3, #7, #8, #17, and #18 were considered to study the effect of heating on recycled mixtures. Table 4-7 shows the increase in PG values due to short-term and long-term aging. For this purpose, samples #2 and #3 were compared with sample #17. These three samples had the same compositions and mixing conditions and were different only in the level of aging. Similarly, samples #7 and #8 were compared with sample #18.

Sample	Aging	X1			X2			X3		
		Total	Aging	Diffusion	Total	Aging	Diffusion	Total	Aging	Diffusion
The change in PG (°C) comparing to non-aged condition (Sample #17)										
#3	1 hr. at 165°C	12.61	10.80	1.81	5.58	8.15	-2.57	-2.60	0.97	-3.57
#2	5 days at 85°C	14.23	11.46	2.77	4.42	7.10	-2.69	-3.87	2.18	-6.04
The change in PG (°C) comparing to non-aged condition (Sample #18)										
#8	1 hr. at 165°C	12.49	9.61	2.88	4.66	6.13	-1.47	-5.20	1.12	-6.31
#7	5 days at 85°C	14.05	11.26	2.79	5.72	6.94	-1.22	-3.27	2.18	-5.45

Table 4-7 Changes in PG of Recycled Mixtures Due to Aging and Diffusion

Generally, the aging affects outer layers more intensely, and those layers experienced more increase in their PG value due to oven heating. On the other hand, the innermost layers became softer after aging. The reason is that in the case of recycled mixtures, the heating accelerates two phenomena simultaneously: aging and diffusion. Similar to virgin mixtures, the outer layers age more severely than the inner layers. At the same time, the rejuvenator migrates from the outer layer toward the inner layers through the diffusion process. Therefore, these two mechanisms work with each other to stiffen the outer layer at a fast pace. On the other side, while aging barely affects the innermost layers, diffusion of the rejuvenator continuously softens them. The aging behavior of new mixtures was used to separate the effect of aging and diffusion on the recycled mixtures. For instance, the PG of the outermost layer of sample #3 with one hour of oven aging at 165°C was 12.61°C higher than PG of sample #17 with no aging. The portion of aging in this increase was estimated by interpolating the PG rises from samples N1 and N2 to samples N3 and N4, and was calculated to be 10.80 °C. The remaining increase (1.81°C) was considered to be caused by diffusion of the rejuvenator from the outer layer toward the middle and inner layers. Figure 4-5 shows the total changes in PG values due to short-term and long-term aging by comparing samples #17, #2, and #3. In addition, the portions of this increase that were induced by aging and diffusion were illustrated separately. It can be seen in this graph that while aging and diffusion work in the same direction for the outer layer and both increase the PG, they act in an opposite direction for layers 2 and 3. Figure 4-6 shows the changes in the PG of different layers, caused by rejuvenation and aging schematically.

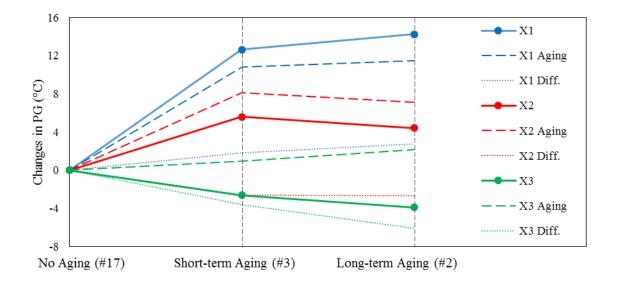


Figure 4-5 Variations PG of Recycled Mixtures Asphalt Layers Due to Aging and Diffusion

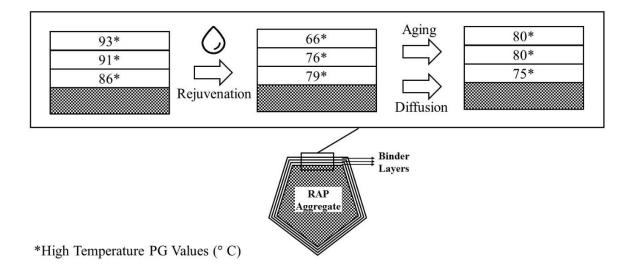


Figure 4-6 Rejuvenation and Aging of Recycled Mixtures

## 4.5 Discussions

In general, recycled mixtures that were aged were more homogeneous than virgin mixtures with the same aging. The average SGF of samples #1 to #16 was 3%. This value was 14% for samples N3 to N6, which were virgin samples that underwent aging. The reason is that rejuvenation and the aging of recycled mixtures both occur from the outside to the inside, and to some extent, balance each other. In other words, the outer layer that is most affected by the aging process has the highest rejuvenator content, and the inner layer that experiences less aging has less rejuvenator.

This lower SGF of recycled binders indicates that even if the average binder PG of a recycled and a new mixture is similar, the outer layer of the recycled mixture is softer than the virgin mix. In addition, it was concluded in Chapter 3 that properly rejuvenated asphalt binders age slower than virgin asphalts. As a result, if a virgin and a recycled mixture have binders with the same initial PGs, after a while, the outer asphalt layer of the recycled mixture would be considerably softer. When the asphalt pavement is subjected to repeated loading, the asphalt undergoes repeated small deformations that can trigger fatigue cracking [42]. While the inner layer of the asphalt is attached to the aggregate, the outer layers that interact with each other experience the most deformation. Therefore, the outer layer has more influence on the fatigue cracking resistance of the mixture. Hence, the smaller stiffness gradient of recycled mixtures can be considered as an advantage for their long-term cracking resistance. This correlates with various studies such as [43] and [12], which confirmed the better performance of recycled mixtures. Moreover, it is probable that recycled mixtures with higher initial PG values perform satisfactorily. If this hypothesis is

confirmed by performance tests, the target PG of recycled mixture can increase without compromising the durability of the mixture.

It should be noted that these observations are based on test with only two rejuvenators. These rejuvenator are those with the best performance according to the study described in Chapter 2. Despite the fact that using these rejuvenators creates a relatively homogeneous recycled binder, it is possible that other rejuvenators are incompatible with asphalt or diffuse slower and form a non-homogeneous recycled binder. It is recommended to perform the staged extraction for other rejuvenators and use the homogeneity index as a measure of rejuvenator compatibility. A low I<sub>h</sub> can be an indication of the use of the wrong type of rejuvenator.

#### **CHAPTER 5: SUMMARY AND CONCLUSIONS**

The objective of the research presented in this dissertation was to improve the effectiveness of asphalt binder rejuvenation. Two critical aspects of rejuvenation were investigated: durability and homogeneity. Some procedures and quantitative measures were introduced to facilitate the assessment of these parameters.

The experimental plan consisted of three major parts. A summary of the conclusions from each of these parts follows in Sections 5.1 to 5.3. The overall conclusions and practical recommendations based on these studies are discussed in Section 5.4, and suggestions for related future works are described in Section 5.5.

## 5.1 Aging of 100% Recycled Asphalt Binders and Mixtures

The first part of the research, which is described in Chapter 2, was a long-term aging study for 100% recycled asphalt binders and mixtures. Virgin asphalt was aged by an extended exposure of PAV. Then the aged binder was softened using five different rejuvenators. These samples were aged for three PAV cycles and their aging was compared together and to that of virgin asphalt. Also, the cracking susceptibility of rejuvenated asphalt mixes and the effects of aging on this parameter was evaluated using the TOT and the APWS aging procedure. The major observations from this study are as follows:

1. There is a significant difference between long-term aging rates of samples rejuvenated by different rejuvenators. Compared to the aging rate of the reference virgin binder, two rejuvenators out of five (CWE and HPE) caused slower aging, while three others (PND, BOF, and APO) accelerated aging.

- The aging behavior of the rejuvenated binder is different from that of virgin asphalt. The aging rate of virgin asphalt samples decreased after the first PAV cycle, but it remained almost constant for rejuvenated binders.
- 3. Even if the rejuvenated binder does not age faster in the first PAV cycle, it may age at a faster pace when the aging continues beyond this point. Such situations, which were true for samples rejuvenated by PND and BOF, can lead to a shorter life for the binder before excessive aging. This observation confirms the importance of studying the long-term aging beyond performance grade standard requirements.
- 4. The service life of recycled asphalt is highly dependent on the properties of the rejuvenator. Selecting the proper rejuvenator was observed to increase the service life up to nine years, compared to rejuvenating with a less effective product.
- 5. The rejuvenator BOF, which is a bio product, caused the fastest aging. Also, samples that contained the other bio-based rejuvenator, APO, experienced fast aging during the last PAV cycle. These observations show that bio-based rejuvenators may cause fast aging, especially in the latter stages of a pavement's life cycle.
- 6. The 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.
- Recycled asphalt mixes can be more resistant to fatigue and reflective cracking than virgin asphalt mixes if rejuvenated properly.

- 8. 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 an aging procedure equivalent to seven to ten years of inservice aging, recycled mixtures can have a better cracking resistance than new asphalt.
- The cracking resistance of recycled mixtures was affected by the type of rejuvenator.
   The mixtures rejuvenated with CWE performed better than those that contained HPE.

# 5.2 Aging and Durability of Partially Recycled Asphalt Binders

The second part of the experimental plan (Chapter 3) was also an aging study, but on partially recycled binders and using an improved procedure. Two virgin binders and 16 samples containing RAP binder and rejuvenator were aged in four stages: One RTFO, and three PAV cycles. The samples were different in the type of RAP binder, virgin binder and recycling agent. The samples were prepared so that their initial high PG was similar to that of the virgin binder they contained. After each stage of aging, DSR tests were conducted, and the high PG was determined. BBR tests were performed at two stages, after the 20 and 60 hours of PAV aging, and the low temperature PG were obtained for all samples. The conclusions are as follows:

 The properties of the rejuvenator have a significant effect on the aging rate of the binder. A recycled binder can age either faster or slower than a virgin binder, depending on the selected rejuvenator. In this experiment, RA1 (similar to HPE in Chapter 2) caused slower aging, and RA2 caused faster aging. The higher the percentage of recycling agent, the greater its effect on the aging of the binder.

- 2. The type and amount of rejuvenator can considerably affect the longevity of the binder. A binder that is recycled by a fast-aging rejuvenator can reduce the life of the binder to less than on 50 percent. Conversely, a slow-aging rejuvenator can increase the life of the binder by up to 30 percent.
- 3. The source of the virgin binder has an effect on both short-term and long-term aging.
- 4. The extent of construction aging, which was simulated by the RTFO, is affected by the properties of the rejuvenator. A recycled binder containing a rejuvenator with higher aromatic content is expected to undergo more aging due to construction heating. However, construction aging is not necessarily undesirable since it gives extra stiffness to the binder early after the construction stage. But if a recycling agent causes fast construction aging, this should be considered during the design of the mixture. For instance, if it is established that certain types of rejuvenators cause excessive short-term (construction) aging, then a slightly softer target PG may be selected.
- 5. Generally, RA2 decreased the phase angle in DSR test. This means that the complex modulus has a smaller viscous portion. Hence, a binder containing RA2 is less viscous than a virgin binder with similar stiffness.
- 6. The effectiveness of RA2 for rejuvenating high RAP mixtures is questionable. This rejuvenator has relatively low softening power. Therefore, a large quantity of it is required to soften a binder with a high RAP content. In addition, binders containing a high volume of this recycling agent age very quickly and have a short life before they become extensively aged again.

- 7. RA1 has desirable properties for recycling high RAP mixtures. It has a high softening power, and a relatively small quantity is enough to rejuvenate a highly aged binder. Also, it has an advantageous aging behavior. It makes the binder age faster during construction and gives extra strength to the pavement immediately after construction when the strength is most needed. Afterward, it decelerates aging and gives the binder a longer life span.
- 8. The low-temperature behavior of recycled binders is significantly affected by the type and dosage of the rejuvenator. In this experiment, samples with RA1 had higher m-values, indicating their greater ability to relieve stresses. As a result, although the virgin binders did not pass the low temperature criteria for PG 67-22, all RA1 samples did pass. Samples with the RA2, on the other hand, had smaller m-values. This correlates with the smaller viscous portion of the complex modulus, which was observed for RA2 samples in high-temperature DSR tests. It is concluded from both DSR and BBR tests that RA2 causes a reduction in the viscous behavior of the binder.
- 9. Unlike DSR tests, BBR tests on samples with extended PAV aging did not add any important information about the effects of using RAP and rejuvenation. Therefore, performing BBR tests on samples with standard aging is adequate for durability evaluation.
- 10. It is necessary to differentiate between rejuvenators that reduce the longevity of the binder and those that increase it. To achieve this, a quantitative description of durability is needed. Critical PAV time can serve as a measure for the longevity of the binder. In addition, a Durability Index (I<sub>d</sub>) was introduced to assess the effects of a rejuvenator on the longevity of the recycled binder.

# 5.3 Blending Effectiveness and Binder Homogeneity

In the last part of this research (Chapter 4), the blending of the rejuvenator with the old asphalt was investigated and the homogeneity of the asphalt film that coats aggregates was studied. The staged extraction method was implemented to investigate the homogeneity and stiffness gradient of the asphalt film. Twenty-six samples were tested to study virgin and recycled mixture before aging and after short-term and long-term aging. The conclusions from this study are as follows:

- 1. The asphalt binder in rejuvenated mixtures is non-homogeneous immediately or shortly after mixing. The outer layer absorbs most of the rejuvenator, while the inner layer is barely rejuvenated and remains hard. Therefore, in cases where the mixture is placed without any major aging or allowing sufficient time for rejuvenator diffusion, such as in in-place recycling methods, the non-homogeneous asphalt binder can lead to an unpredictable performance and may cause rutting issues.
- 2. When the sample is heated, the diffusion of the rejuvenator accelerates causing the asphalt layer to homogenize. In addition, the heating process ages the asphalt binder; however, this aging process is not similar in all layers. The outer layers are most influenced by aging, while the inner layers are only slightly aged.
- 3. In recycled mixtures, both rejuvenation and aging occur from the outside to the inside. Therefore, to some extent, these two processes balance each other and deliver a relatively homogeneous asphalt binder. This can be considered an advantage for recycled mixtures over virgin mixtures that are less homogeneous due to inconsistent aging.

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4. In order to quantify the stiffness gradient and homogeneity, two parameters were introduced: Stiffness Gradient Factor (SGF) and Homogeneity Index (I<sub>h</sub>). These parameters are defined by Equations 4-3 and 4-4.

# 5.4 Overall Conclusions and Discussions

The knowledge obtained from this research provides a better understanding of asphalt binder rejuvenation. While rejuvenated asphalt is often treated similarly to virgin asphalt for design and performance prediction purposes, there are certain factors that make it different. It is necessary to consider these factors to improve the asphalt pavement recycling practice. The observations from different parts of this research indicated that:

- Recycled pavement material can potentially perform even better than new material. The results from this research showed that if a proper rejuvenator is used, the asphalt binder could be more durable, more homogeneous, and more resistant to cracking.
- 2. The properties of the rejuvenator have a significant influence on the effectiveness of rejuvenation. Traditional criteria such as penetration, viscosity, or PG requirements are not capable of indicating all aspects of the rejuvenator quality.
- 3. This research introduced four parameters that provide quantitative measures for durability and homogeneity of the asphalt binder:

a) *Critical PAV Time*: The PAV aging time (in hours) it takes to increase the high PG of a sample of asphalt binder from 70 °C to 95 °C.
This parameter can serve as an indicator of the longevity of a certain asphalt binder. The higher the critical PAV time, the more durable the binder. As a rough estimate, every hour of critical PAV time correlates with 0.4 years of service life.

- b) *Durability Index (I<sub>d</sub>)*: The critical PAV time of the recycled asphalt divided by that of the virgin asphalt with similar initial PG.
  - This parameter provides a measure of the influence of an RAP-rejuvenator combination on a binder's longevity. An I<sub>d</sub> greater than 1.0 implies that the rejuvenator improves a binder's longevity, while a smaller value indicates its undesirable effect on the longevity.

c) Homogeneity Index (I<sub>h</sub>) =  $1 - \frac{Maximum PG_i - Minimum PG_i}{PG_{ave}}$ 

This parameter shows how homogeneous the asphalt binder film is. Values closer to 1.0 indicate more homogeneous binder and small values show poor homogeneity.

d) Stiffness Gradient Factor (SGF) = 
$$\frac{PG_{Outermost Layer} - PG_{Innermost Layer}}{PG_{ave}} \times 100\%$$

This parameter shows how the binder film is structured. A positive SGF shows that outer layers are harder than the inner layers and a negative SGF shows that the outer layers are softer. A large negative SGF can be an indication of improper blending.

- 4. Two methods are suggested for adjusting the rejuvenator dosage, based on the durability properties of the rejuvenator. The use of the proposed adjusted dosages, will affect the cost of the recycling, and the rejuvenator selection process.
- 5. Using a proper rejuvenator can lead to slower aging of the rejuvenated binder, compared to virgin asphalt. On the other hand, it is expected that recycled mixtures have a more homogeneous binder film in the long-term. These properties can facilitate the use of recycled binder with a higher initial PG without compromising the long-term performance. The use of a higher PG decreases the required amount of

rejuvenator, leading to a lower cost, and provides a better initial stiffness and rutting resistance.

# 5.5 **Recommendations for Future Work**

 In this research, only a limited number of rejuvenators were tested. It is necessary to standardize and repeat the procedures for several rejuvenators. After collection of a database with a proper number of rejuvenators, the parameters introduced in this research, especially the durability index, can serve as a quality measure for rejuvenators.

The staged extraction experiment was performed on samples with only two well performing rejuvenators and resulted in desirable homogeneity of the rejuvenated binders. Using different rejuvenators, especially those that are incompatible with the RAP binder, can lead to different scenarios. It is recommended to conduct the staged extraction procedure for mixtures recycled by various rejuvenators.

- 2. This research was based on rheological properties of the binders. A study on similar problems, with a focus on the chemical properties, can provide a better understating of these concerns.
- 3. Based on the results of this study, the author expects that if a proper rejuvenator is used, a recycled binder with higher initial high PG can be used without adversely affecting the long-term performance properties. This hypothesis needs to be verified through mixture performance tests and field performance evaluations. Approval of this hypothesis can serve as a major step to enhance the design and quality control procedures for high RAP mixtures.

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#### VITA

## MOJTABA MOHAMMADAFZALI

2005	B.S., Civil Engineering, Tehran Azad University (Iran)
2009	M.S., Geotechnical Engineering, University of Tehran (Iran)
2013-2014	Dwight David Eisenhower Transportation Fellowship Recipient
2015	Best Younger Member Paper Award, ASCE Highway and Airfield Conference
Present	Ph.D. Candidate, Civil Engineering, Florida International University

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