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RHEOLOGICAL AND CHEMICAL CHARACTERISTICS OF ASPHALT BINDERS RECYCLED USING RECYCLING AGENTS AND NANOPARTICLES

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DEDICATION

I dedicate this dissertation to my beloved parents and my brothers.

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ABSTRACT OF THE DISSERTATION

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by

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Florida International University, 2020

Miami, Florida

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The reuse and recycling of materials, especially materials derived from nature, is necessary for a sustainable environment and world. Asphalt mixture is a combination of asphalt binder and aggregates, both of which originated from nature. The reuse and recycling of aged asphalt materials (RAP) is widespread. However, a large portion of these materials is wasted. One primary reason for the reluctance to use a high percentage of RAP in recycled asphalt is uncertainty in the long-term performance of recycled asphalt pavement. To improve the long-term performance of recycled asphalt, new additives and Recycling Agents (RA) are used to modify the binder's properties. However, different RAs will change the recycled binder's properties differently, resulting in different long-term recycled binder properties. Therefore, this study was conducted to correlate the chemical indices and rheological properties of the recycled binder using different recycling agents. Furthermore, the performance of recycled binders was evaluated to predict the long-term performance of recycled binders according to their unaged and initial properties. The results showed that the chemical indexes (carbonyl and sulfoxide) of recycled binders can be used to predict the long-term performance of recycled binders. These results could also be used to select the proper RA in the recycling of RAP material. Also, an asphalt design procedure based on the chemical indices of binders was developed for use by practitioners and researchers. The previous design method was based on Performance Grade tests, while the developed method is mostly based on the chemical properties of binders, and its tests are easier and more economical.

Furthermore, the incorporation of RAP percentage in the unmodified PG 67-22 binder was limited to 20% by the Florida Department of Transportation due to uncertainty in the long-term performance of recycled asphalt. To increase the percentage of RAP in recycled asphalt, new additives are used to modify the asphalt binder. One of the newly introduced additives is nanoclay particles, which were applied in the recycling of RAP and were investigated in this study. The results showed an increase in the incorporation of RAP percentage in the nanomodified binder by 30% compared to using unmodified (neat) asphalt binder. The improved properties of the nanomodified binder compared to the unmodified binder proved the feasibility of using a nanomodified binder in severe weather conditions and roads that experience higher loads and traffic.

TABLE OF CONTENTS

CHAPTER PAGE

LIST OF TABLES

TABLE PAGE

LIST OF FIGURES

FIGURE PAGE

1 CHAPTER 1: INTRODUCTION

1.1 Background

Hot Mix Asphalt (HMA) is the most utilized material in the construction of roads. However, HMA performance can degrade during its service life. The aged asphalt can be removed and reconstructed or rehabilitated to improve pavement serviceability. Reclaimed Asphalt Pavement (RAP) is the removed and milled aged asphalt material from the surface of roads. The RAP is used in the subsurface of roads, or is recycled and implemented into the production of HMA. Recently, there have been efforts to recycle RAP due to its environmental and economic benefits. According to the Federal Highway Administration (FHWA) and National Asphalt Pavement Association, asphalt is the most recyclable material, of which more than 99% is reusable (Al-Qadi et al. 2007). However, most states have limited the incorporation of RAP in the production of HMA to 20% because of uncertainty in the performance and reduced service life of the recycled asphalt pavement. Recycling RAP not only saves the landfill from disposal of the RAP, but can also reduce fuel emissions.

The primary focus of recent studies in asphalt technology is to recycle 100% of RAP. To achieve this milestone, different modifiers have been introduced that can be added to RAP during the recycling process to recover RAP properties. Recycling Agents (RAs) or Rejuvenators are the most common and effective recycling agents that have been used to recover RAP binder properties (Elwardany et al. 2017, Sharma et al. 2017). Another new modifier that has been implemented in recent studies as a recycling additive is nanomodified asphalt binder. It was demonstrated that using nanomodified asphalt binder with 3% of nanomaterial in the recycling of RAP can enhance the recycled pavement performance significantly (Sarsam and Shujairy 2015). These nanomaterials consist of up to a 100 nm (nanometer) dimension (Abdelrahman et al. 2014, Fang et al. 2016).

1.2 Asphalt Pavement Structural Performance

Asphalt pavement is the combination of asphalt binder and aggregates that are mixed and paved in the surface of roads. To produce asphalt pavement, several manufacturing methods like hot-mix asphalt (HMA), warm-mix asphalt (WMA), and cold-mix asphalt (CMA) are available. In the HMA method, which is the most commonly used method, asphalt binder and aggregates are mixed at an approximate temperature of 300 °F.

The structural performance of asphalt pavement is related to its material, traffic loading, and environmental conditions. Asphalt pavement materials and environmental conditions of where pavement is placed are playing essential roles in asphalt pavement performance because asphalt binder has a viscous behavior and its behavior is highly related to the temperature. Also, exposing asphalt binder to the air and water causes oxidation of asphalt pavement and degradation of its performance over time. Therefore, short-term and long-term performance of asphalt pavement should be considered in evaluating the asphalt pavement performance.

Superpave Performance Grading (PG) is a method to characterize asphalt binder properties used in HMA. This grading method is designed to address HMA performance including rutting, fatigue cracking, and thermal cracking. The PG of asphalt also considers aging and climate conditions where the pavement is placed. The PG of asphalt determines the tests and criteria that binder at a specific temperature, which is related to the climate conditions, should pass.

At high temperatures, asphalt pavement is softening, and densifying due to traffic load. Therefore, ruts will develop on the surface of the asphalt pavement. With oxidation of asphalt, asphalt pavement get stiffer. Therefore, rutting performance is not in concern in asphalt pavement long-term performance and it is only should be considered in evaluating the short-term performance of asphalt pavement. Therefore, to have a high-quality asphalt pavement in its short-term age and high temperatures, asphalt binder should be stiff enough or have a high PG.

With oxidation of asphalt due to sunlight and heat, the asphalt binder gets stiffer and cracks form in asphalt pavement due to repetitive loads of traffic. These cracks are classified as fatigue cracking. To prevent these cracking, asphalt pavement should have an elastic behavior in medium temperatures to dissipate repetitive loads from vehicles. In this case, the binder with lower PG at medium temperature can provide better long-term performance and more elastic behavior.

With decreasing asphalt pavement temperature, asphalt binder will contract, and internal stresses develop in the binder. When the reduction of asphalt pavement temperature is fast, asphalt binder has no time to relax the developed stress; therefore, cracks form in the pavement. These cracks are called thermal or transverse cracks. The critical temperature that develops the critical stresses in the binder is used to define low-temperature PG of asphalt binders. Therefore, the asphalt binder that has lower low-temperature PG at low temperatures can withstand lower critical temperatures. The binder with desirable low-temperature performance is not too stiff at low temperatures and is able to relax the contract stresses.

Asphalt binder is a byproduct of crude oil. Therefore, asphalt binders produced from different sources of crude oil have different chemical composition and properties. Since asphalt binder includes various types of chemical compositions, the asphalt binder chemical molecules are classified into some groups based on their chemical functionality. It has been shown that with aging and oxidation of asphalt binder, asphaltene portion of asphalt binder will increase, leading to producing of carbonyl and sulfoxide chemical functional groups. The rate of carbonyl and sulfoxide production can show the aging rate and degradation rate in the performance of asphalt pavement. Therefore, the chemical composition of asphalt binder and the rate of its changes with oxidation can play an essential role in pavement performance that should be considered in evaluating pavement performance.

Based on the aforementioned description, asphalt pavement performance and properties are highly dependent on the source of asphalt binder crude oil and its refinery process, environmental and climate condition of where pavement is placed, material and aggregate type of asphalt, kind of asphalt production and mixing of aggregate with the binder, quality of pavement construction, and asphalt binder oxidation rate. Considering these highly variable characteristics of asphalt pavement, evaluating the pavement performance and asphalt pavement mix design is highly based on performing experimental testing on asphalt pavement. Influenced by chemistry, temperature and time, the performance of asphalt pavement is predictable with a large amount of experimental testing. This is not desirable.

This effort is aimed at relating chemical indicators, which are easier, faster to test and temperature independent with rheological parameters, which are costly, temperature dependent and time consuming. Building on these correlations will facilitate a more efficient pavement recycling.

1.3 Motivation

Asphalt pavement is the most recyclable material. However, most of the states limited the incorporation of RAP material in recycled asphalt to 20% with virgin materials. This limitation is due to uncertainty in the short- and long-term performance of recycled asphalt. As the asphalt pavement cost is highly dependent on its material cost, increasing the content of RAP in recycled asphalt can save money and natural resources. To increase RAP content, the modified binders or RAs should be used. In this case, there is a need to evaluate the use of a nanomodified binder and different RAs to recycle asphalt pavement and define the increase in the content of RAP in the recycled asphalt pavement. Due to improvement in the nanomodified binder properties and antiaging property of nanoparticles, it is expected that using a nanomodified binder instead of virgin binder in the recycling process will increase the incorporation of RAP content.

Furthermore, it is possible to recycle 100% RAP material using RAs. However, because RAP materials and RAs have different properties, it is necessary to select proper RA in the recycling of a specific RAP material to have a recycled asphalt pavement with high quality. If proper RA is not selected, the recycled asphalt would not have acceptable performance. But, if a proper RA is selected in the recycling of asphalt pavement, the recycled asphalt can have better short- and long-term performance compared to virgin asphalt pavement. In this case, the recycled asphalt binders using different RAs will be evaluated based on their chemical, short- and long-term properties to develop a procedure in selecting the proper RA in the recycling of asphalt pavement.

As mentioned in the previous section, the focus in the mix design of asphalt pavement is to provide asphalt pavement with better short- and long-term performance. The asphalt pavement mix design is based on rheological experimental testing of the asphalt binder which is time-consuming and costly. However, conducting chemical testing on the asphalt binder is not time-consuming and economically efficient by 50% approximately in comparison with rheological tests. In this case, if a correlation between chemical properties of asphalt and rheological properties of asphalt is determined, it would be possible to rely on chemical testing in asphalt pavement mix design and selecting the proper RAs in the recycling of asphalt pavement. In this case, it also would be possible to predict and define the short- and long-term performance of asphalt pavement based on the chemical composition of asphalt pavement.

1.4 Problem Statement

Durable asphalt is the asphalt binder that represents good performance during the service life of asphalt pavement. A durable asphalt is defined as asphalt with desirable initial performance properties and resistance to the rheological properties that change due to environmental impacts (Petersen 2000). To have a durable asphalt binder, the recycled asphalt binder should have an aging rate equivalent to or less than that of the virgin binder. Therefore, the aging rate of recycled asphalt binder or the change of chemical and rheological properties of the recycled asphalt binder during its aging is indicative of asphalt pavement durability.

Oxidation of the asphalt binder during the asphalt serviceability is the main factor influencing the decline of asphalt durability (Petersen 2000). During the oxidation of asphalt, the chemical composition and flow properties of asphalt will change, causing brittleness and hardness of the asphalt binder. The recycling agents or rejuvenators and other additives are added to the RAP to restore the chemical and physical properties of the aged binder. However, little information can be found in the literature about the changes in the chemical properties of recycled asphalt using a rejuvenator and nanomodified binder during the service life of pavement.

Recycling agents impact RAP properties differently, resulting in various aging rates of recycled asphalt. Moreover, the relationship between the chemical composition and rheological properties of recycled asphalt can be used to determine the age of recycled asphalt. Therefore, determining this relationship, if applicable, can help predict the performance of asphalt binder using chemical testing of the recycled asphalt binder. This relationship also helps identify the maintenance and treatment time for pavement. Because there are different asphalt binder sources and recycling agent types, this study can help categorize the recycling agents with respect to their effects on the asphalt binder durability and aging rates.

Limited studies have been conducted to compare recycled asphalt binder performance with virgin asphalt binder performance, and especially on the recycled asphalt binder with the nanomodified asphalt binder. According to previous studies, implementation of nanomaterial in the asphalt binder has anti-aging properties and can improve fatigue cracking and rutting resistance of asphalt pavement. Therefore, in this study, the feasibility of using a nanomodified asphalt binder as a recycling additive will be evaluated by considering the rutting, fatigue, and low-temperature cracking resistance of recycled asphalt binder.

1.5 Objectives

In summary, the following two major objectives are the primary focus of this study:

1- Study and correlate the chemical indices and rheological properties of the recycled binder using different recycling agents and a nanomodified asphalt binder.

2- Study and evaluate the performance of a recycled asphalt binder using different recycling agents and a nanomodified asphalt binder.

By finding the correlation between chemical indexes and rheological properties of a recycled asphalt binder, it is possible to develop a method to determine the RA percentage in the recycling process based on chemical indices. Also, it is possible to predict the long-term performance of recycled binders and select the proper recycling agent in the recycling process of asphalt pavement. Moreover, the study on the application of a nanomodified binder in the recycling of asphalt pavement can help increase the amount of RAP percentage in the recycled mix by 30% using a nanomodified binder compared to an unmodified binder.

1.6 Methodology

To achieve these research objectives, the chemical indices of a binder was measured using the Fourier Transform Infrared Spectroscopy (FTIR) test (Figure 1-1). The chemical functional groups that contain oxygen and change during the oxidation of asphalt binder are carbonyls and sulfoxides. The intensity of carbonyls and sulfoxides in a binder can be measured using the FTIR test. Carbonyls and sulfoxides are the chemical functional groups of asphalt that represents asphalt binder age.

Figure 1-1: FTIR device

To measure the rheological properties of asphalt binder, the Dynamic Shear Rheometer (DSR) test was conducted. The DSR (Figure 1-2 a) is a device that measures the viscous and elastic behavior of asphalt binder. Complex shear modulus (G*) and phase angle (δ) are the two viscus properties of asphalt binder that can be measured by the DSR test. Phase angle shows the lag between applied sheer stress and produced sheer strain in binder. The lower phase angle, the more elastic behavior of asphalt. DSR test can be conducted at high and medium temperatures to determine the asphalt binder Superpave PG grade. To assess the low-temperature performance of asphalt binder, the Bending Beam Rheometer (BBR) test was conducted. The BBR (Figure 1-2 b) test measures the creep stiffness (S) and m-value. Creep stiffness is an indication of thermal stresses in the binder due to the contraction, and the m-value is the slope of the master stiffness curve, which determines the rate of stress relief.

a) b)

Figure 1-2: a) DSR and b) BBR devices

In order to measure the asphalt binder chemical indices and rheological property changes during its aging process, as well as to evaluate the recycled asphalt binder performance, the following parameters were assessed by conducting the FTIR, DSR, and BBR tests.

1- Find the relationship between the chemical indices and rheological properties of the recycled binder. The rheological properties of asphalt binder (G^* and δ) were measured using DSR at 74˚C and 30˚C. The chemical indices of the asphalt binder were evaluated using the measured intensity of carbonyls and sulfoxides.

2- Study the fatigue behavior of virgin and recycled binders using the DSR test at an intermediate temperature.

3- Study the rutting behavior of virgin and recycled binders using the DSR test at a high temperature.

4- Study the low-temperature cracking resistance of virgin and recycled binders using the BBR test.

In order to perform this study, the following hypotheses were considered.

Hypothesis 1: The intensity of carbonyls and sulfoxides have a logarithmic linear relationship with the shear modulus of recycled asphalt binder.

Hypothesis 2: The intensity of carbonyls and sulfoxides have a linear relationship with the shear phase angle of recycled asphalt binder.

1.7 Organization of Dissertation

This dissertation includes five chapters. Chapter 1 is an introduction that defines the objectives of the study and the knowledge gap. A comprehensive literature review is also presented in this chapter. Chapter 2 describes the methodology and the tests that were conducted to perform this study. Chapter 3 presents the results of the recycling of RAP binder using different recycling agents and the correlation between the chemical and rheological properties of recycled binders. Chapter 4 compares the properties of nanomodified and unmodified binders and evaluates the feasibility of using nanomodified binder in the recycling of asphalt binders. Chapter 4 also investigates the recycled binder with nanomodified binder, and comparisons are made between recycled binders with unmodified binder. Finally, Chapter 5 is the conclusion of this study and presents recommended future studies.

1.8 Review of Literature

1.8.1 Recycling of Asphalt Using Recycling Agents

The main issues in the incorporation of RAP in the HMA are susceptibility in pavement long-term performance and premature cracking (Al-Qadi et al. 2009, Al-Qadi et al. 2012, Al-Qadi et al. 2015). Accordingly, in order to increase the incorporation of RAP in HMA production, the longevity, durability, and long-term performance of asphalt pavement should be addressed. Asphalt pavement during its serviceability and design life will age and become stiffer, leading to the brittleness of asphalt (Baqersad et al. 2019, Baqersad &

Ali 2019). The essential factor in the degradation and brittleness of asphalt during its service life is the oxidation of bitumen. The recycling agent (RA) or virgin asphalt (new asphalt binder) should be added to the RAP in the recycling process to rejuvenate and recover the RAP properties. The ideal bitumen must have high stiffness at a high temperature to decrease the rutting susceptibility, as well as high adhesion between the binder and aggregate in the presence of moisture to minimize the stripping susceptibility (Robertson et al. 1991). The bitumen should also not be too stiff to crack and have an elastic behavior to dissipate energy and resist fatigue cracking. These features indicate that the chemical and physical properties of bitumen and their changes during the service life of pavement should be designated to produce a mixture with desired and durable features (Robertson et al. 1991). In fact, a durable asphalt has desirable initial performance properties and resists the change of physical properties during its design life due to environmental impacts (Petersen 2000). To have a durable asphalt binder, the recycled asphalt should be homogeneous, and the aging rate of the recycled binder should be the same or less than that of the virgin binder (Mohammadafzali et al. 2018). Oxidation of the asphalt binder during the asphalt serviceability is the main factor influencing the degradation of asphalt durability (Petersen 2000, Petersen 2009).

The asphalt binder reacts with oxygen in the environment during its serviceability, which degrades the properties of asphalt. During the oxidation process, the asphalt becomes harder and brittle (Corbett 1970). The movement of the nonpolar components of asphalt to the polar components of asphalt occurs (Knotnerus 1972). It was found that the polar component of asphalt is the fraction responsible for the aging of asphalt in such a way that the asphaltene fraction of asphalt increases during the aging of asphalt. After the reaction of asphalt with oxygen, the functional group of Ketone, Anhydride, Carboxylic Acid, and Sulfoxide is produced (Rostler and White 1960). The asphalt molecules are grouped into several classes according to their chemical functionality to have a better understanding of the chemical components of asphalt (Petersen 2009). It was shown that carbonyl (C), which is typically the carbonyl chromophore associated with Ketone formation, along with sulfoxide (S) formation, are the main productions of asphalt oxidation (Institute and Administration 2011). Previous studies indicated that there is a linear relationship between the logarithm of dynamic shear modulus (G*) and carbonyl plus sulfoxide absorbance peaks (C+S) (Institute and Administration 2011, Elwardany et al. 2017). Moreover, a linear negative correlation was found between log G* or shear phase angle (δ) with asphalt binder carbonyl. Specifically, the carbonyl content was able to accurately predict the age of asphalt.

The function of RAs in the recycling of RAP is to replace the oxidized, evaporated, or absorbed molecules of asphalt binder by aggregates. This material is added to the RAP to restore and recover the chemical and rheological properties of the aged binder. RA consists of a highly naphthenic or aromatic fraction. However, aromatics and saturates are the main combinations of RAs that are important in the diffusion of RA into the RAP (Cong et al. 2016). The RA molecular structure can have a single component or composite component, which consists of different aromatic and resin components that increase the effectiveness of RA (Zhao et al. 2016). Most RAs have a lightweight and high aromatic content. They have a smaller molecule size and weight to increase the mobility of RA and improve the diffusion of rejuvenator into the RAP. Xu et al. (2018) developed a molecular dynamics model to study RA diffusion behavior. They found that RA can restore the thermodynamic properties and molecular structures of RAP to some extent.

In recent studies, the durability and aging of recycled asphalt using RAs were studied, and it was concluded that the recycled asphalt has a slower aging rate compared to a virgin binder, and it is as durable as an original binder (Shen et al. 2007, Singh et al. 2012). However, the aging rate and durability of recycled asphalt were mostly investigated during the earlier stages of the aging of recycled asphalt with a focus on the rheological properties of recycled asphalt. More specifically, there are only a few studies on the chemical changes of recycled asphalt with aging, which is the essential factor in the durability of recycled asphalt.

1.8.2 Application of Nanomodified Asphalt

Due to the increase in traffic loads and volume, conventional asphalt pavements are not suitable for substantial high traffic conditions. Therefore, there is a need for new pavement with special properties that are suitable for these conditions (Ali & Baqersad 2018). In this case, different modifiers have been introduced to improve pavement performance, such as crumb rubber, styrene-butadiene-rubber (SYP), polymer modifiers, etc. One of the newest materials that has been introduced is nanoparticles, which has had a significant effect on the technology and pavement industries. Pavement engineers have attempted to identify and prove the special properties of nanoparticles and their effects on pavement performance.

Nanoparticles are materials with a 1 to 100 nm size in at least one dimension (Li et al. 2017). Some of the properties of nanoparticles are their high-temperature sensitivity, high ductility, high strain resistance, and low electrical resistivity. Due to these properties, nanoparticles have been introduced as asphalt pavement modifiers and additives (Zhou et al. 2014, Veytskin et al. 2015). As asphalt is a visco-elastic material, nanoparticles produce essential effects on asphalt high temperature, medium temperature, longevity, fatigue, and moisture performance (Amirkhanian et al. 2011, Xiao et al. 2011).

There are different kinds of nanoparticles available in the market, including nanocarbon, nanoclay, nanofibers, etc. Nanoclay (layered silicate) is the nanoparticle mostly used as a material and asphalt modifier due to its low cost and abundance. The previous study identified nanoclay's positive effects on the mechanical and rheological properties of asphalt pavement (Abdelrahman et al. 2014). This study used scanning electron microscopy (SEM) and FTIR and demonstrated the effects of nanoparticles in the improvement of pavement aging and longevity. It was also shown that nanoparticles improved the visco-elastic properties of asphalt with the increase in complex shear modulus (G*) and phase angle (δ) of asphalt. (You et al. 2011) (Yao et al. 2012). However, different studies resulted in different levels of improvement. These various percentages of changes demonstrated that the content and type of nanoparticles play an essential role in the properties of nanomodified pavement properties. Furthermore, another study modified five different neat asphalt binders using the same nanoparticles to evaluate the effects of a neat binder on the properties of a nanomodified binder (Yang and Tighe 2013). The statistical analysis results of this study identified that binder performance grade (PG) has an important impact on the properties of produced nanomodified asphalt, especially in asphalt deflection, stiffness, and m-value properties.

Nanoparticles can improve the asphalt pavement rutting performance compared to neat asphalt (Khattak et al. 2012). This improvement, which is similar to asphalt visco-elastic properties, is highly dependent on the nanoparticle type (Amirkhanian et al. 2010, Faramarzi et al. 2015). Similarly, the low-temperature properties of nanomodified asphalt pavement improved significantly compared to the nonmodified asphalt (Fang et al. 2016). The creep stiffness (S) of the nanomodified binder decreased in comparison to nonmodified asphalt, which led to the enhancement of low-temperature cracking resistance. However, this improvement was not significant in another study with different nanoparticles (Yao et al. 2012). Another study by Abdelrahman et al. (2014) conversely resulted in a creep stiffness increase and a decrease in the m-value of nonmodified asphalt compared to unmodified asphalt.

Asphalt binder reacts with oxygen in the environment during its serviceability, which degrades the properties of asphalt. Studies on nanomodified asphalt pavement proved the anti-aging property of nanoparticles. However, different studies used different nanoparticles and aging indicators. Remarkably, all studies proved the antiaging feature of nanomodified asphalt (Sun et al. 2008, Jahromi and Khodaii 2009). Also, studies on the fatigue performance of nanomodified asphalt pavement proved the improvement in the long-term performance of nanomodified asphalt compared to unmodified asphalt pavement (Khattak et al. 2013, Kavussi and Barghabany 2015).

The mixing of nanoparticles with asphalt binder is an issue due to the possibility of the formation of clumps and agglomeration of particles during the mixing process. This agglomeration can be caused due to the high viscosity of the binder or solubility of nanoparticles. Some believe van der Waal's attraction is the reason for agglomeration (Yao et al. 2012). Without considering the reason for clump formation, this issue can adversely affect the positive impacts of nanoparticles in asphalt pavement. In this case, it is necessary to select the proper mix procedure that can disperse nanoparticles in asphalt and prepare a homogeneous nanomodified asphalt. Two different mixing procedures are available: a dry blending method or solvent blending method. The solvent blending method has better function. However, it is not environmentally friendly (Yao et al. 2012). The dry blending method is efficient and simple. This method has two steps: adding nanoparticles to the asphalt binder and then mixing the nanoparticles. However, due to the sensitivity of asphalt to temperature, this method is highly dependent on the mixing temperature since it can cause ductility problems (Polacco et al. 2008, Zare-Shahabadi et al. 2010, You et al. 2011).

It can be demonstrated that the nanoparticles have special positive impacts on the properties of asphalt pavement. However, these impacts are highly dependent on the neat asphalt binder and nanoparticles' properties. Hence, this study was conducted to investigate the effects of nanoclay particles on the asphalt binder commonly used in Florida. It is expected that nanoparticles enhance asphalt pavement properties, especially long-term and fatigue performance, which is a concern. The results can be used to pave the path to using nanomodified asphalt pavements in the construction of new roads and the recycling of reclaimed asphalt pavement (RAP).

1.8.3 Recycling of Asphalt Using Nanomodified Asphalt

The use of Reclaimed Asphalt Pavement (RAP) in the construction of roads and resurfacing of existing roads is economically beneficial and environmentally sound (Xiao et al. 2007).

The service life and performance of asphalt pavement decreases during its serviceability. However, pavement material (RAP) still has value and can be recycled and used in the construction of new roadways. The recycling of RAP can save energy, materials, and money. In the recycling process of asphalt pavement, it is possible to use RAP material with virgin asphalt or recycling agents (rejuvenators) to improve the properties of RAP material. In this way, the recycled pavement should have the same or better performance than the virgin asphalt mix. One verified improvement in the incorporation of RAP in recycled asphalt pavement compared to asphalt pavement with no RAP is in the rutting performance of recycled asphalt pavement. The increase in the incorporation of RAP in the recycling of asphalt pavement results in an increase in the stiffness of asphalt, causing enhancement in the rutting performance of asphalt pavement. Rutting is the densification and longitudinal pressing of asphalt pavement in the path of traffic wheels due to traffic loads (Chen et al. 2004).

The recycling of asphalt pavement using a relatively low percentage of RAP can be conducted without performing tests on the asphalt binder. However, when the incorporation of RAP in the recycled asphalt pavement is increased, it is necessary to perform asphalt binder property and performance tests to determine the inclusion of RAP effects and verify the short- and long-term performance of recycled asphalt pavement (McDaniel et al. 2000). Although increasing the inclusion of RAP can improve the rutting resistance of asphalt pavement, there is some uncertainty in the long-term and longevity performance of asphalt pavement.

Despite improving the rutting resistance of asphalt pavement with the incorporation of RAP material, increasing the RAP content in asphalt pavement has negative impacts on the cracking resistance and long-term performance of asphalt pavement (Kennedy et al. 1998, Lee et al. 1999). To address this cracking resistance issue, specific asphalt modifiers like polymer modifiers (such as SBS polymer asphalt modifiers) were introduced to improve the cracking and rutting resistance of asphalt pavement (CAROFF 1994, Kim and Sargand 2003, McDaniel and Shah 2003). It was shown that using an SBS polymer modified binder such as a virgin binder in the recycling of RAP material could improve rutting resistance and cracking failure resistance (Huang et al. 2005). However, due to these uncertainties in the short- and long-term performance of recycled asphalt pavement, state departments of transportations (DOTs) limited the content of RAP material in recycled asphalt pavement. For instance, the Florida DOT limited the content of RAP material in asphalt pavement up to 20% by the weight of total aggregates (FDOT 2017). The Kim and Sargand (2003) study on the recycling of RAP material using polymer modified asphalt showed that the G*.sinδ and G*/sinδ parameters are representative of RAP content in pavement. These two parameters, which are representative of fatigue and the rutting performance of asphalt, were increased with the increase of RAP content in recycled asphalt (Roque et al. 2015). Also, the RAP and binder source had a significant effect on the fatigue and rutting performance of the asphalt binder (Stroup-Gardiner 2016).

The incorporation of modifiers and modified binders in the recycling of asphalt pavement is commonly due to improving the long-term and fatigue performance of recycled asphalt pavement. Fatigue performance and cracking resistance of asphalt pavement is associated with the repetitive axle load application on roadways. In this case, the modifiers help to improve the cracking resistance of recycled pavement. Another innovative asphalt modifier is the nanoparticle. It has been shown that incorporation of nanoparticles in asphalt pavement resulted in enhancing asphalt pavement performance, especially in rutting and fatigue performance. The laboratory test results of the nanomodified asphalt binder and mixture showed improvement in the fatigue and rutting performance of asphalt pavement compared to the unmodified asphalt pavement. A recently limited study on the conditioned and unconditioned recycled asphalt pavement mixtures using nanomodified asphalt also showed a significant improvement in the pavement's fatigue life (Sarsam and Shujairy 2015).

The unique and special features of nanoparticles make them one of the best asphalt modifiers that can answer the demand for the pavement of highways with a required particular behavior (Yu et al. 2009, Santagata et al. 2012, Yao et al. 2012, Yusoff et al. 2014). As a result, different studies have been conducted to verify the improvements in nanomodified asphalt compared to unmodified asphalt. However, few studies have been conducted with the use of nanomodified asphalt in the recycling of RAP materials. Therefore, in this study, different percentages of RAP binder were added to the nanomodified and unmodified binder to determine the capability of using a nanomodified binder in the recycling of asphalt pavement.

2 CHAPTER 2: METHODOLOGY

2.1 Recycling of Asphalt Using Recycling Agents

In this study, the Superpave PG standard tests and aging process were conducted. In the first step of the aging process and recycling of the aged binders, the two virgin binders were aged separately using a Rolling Thin-Film Oven (RTFO) and two cycles of Pressurized Aging Vessel (PAV) to prepare the aged binders (Figure 2-1). Then, the aged binders were mixed with RAs to recover the aged binders' properties. Finally, the recycled binders were aged using RTFO and two cycles of PAV to simulate the aging of recycled binders.

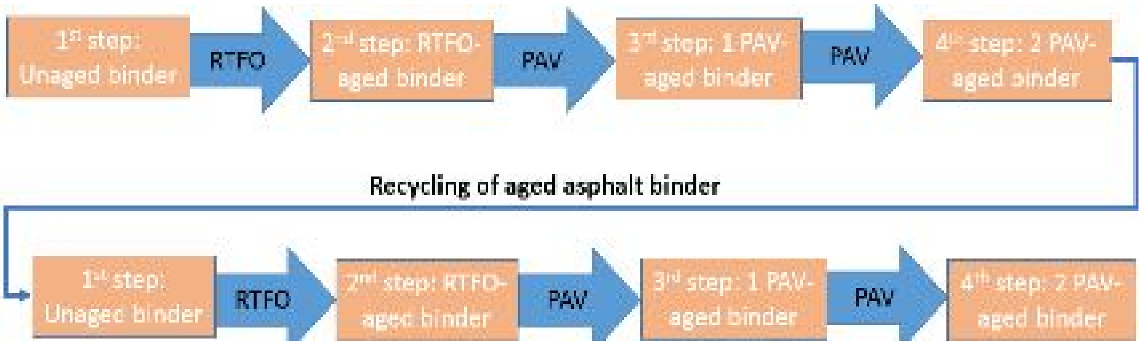


Figure 2-1: Aging and recycling process of binders

In the RTFO aging procedure, the asphalt binder is exposed to heat and airflow to simulate the manufacturing and placement aging of asphalt binder. In this aging procedure, the unaged binder samples were placed in the oven at 163 ˚C for 85 minutes (AASHTO T240) (AASHTO T 240 2013). In the PAV procedure, the asphalt binder was exposed to heat and pressure to simulate the in-service aging of the asphalt binder. Each standard PAV aging cycle included exposing the samples to 2.1 MPa pressure and 100 ˚C heat for 20 hours. In this study, two PAV cycles were used to age the asphalt binder. In the first step, the RTFO-aged binder samples were placed in the PAV for 20 hours. Then, for another 20 hours, the 1 PAV-aged samples were placed in the PAV to be aged again (AASHTO R 28) (AASHTO R 28 2012). Each PAV cycle simulated a 7- to 10-year period of in-service aging of asphalt binder. The study by FDOT concluded that each PAV cycle was equivalent to eight years of pavement service life (Bahia and Anderson 1995).

The recycling process of the asphalt binder using RAs included heating the 2 PAV-aged asphalt binder to 155±5 ˚C, and then adding a desired amount of RA and mixing them for two minutes. The RA and aged binder were mixed using a spatula. The amount of RA was defined to decrease the high-temperature PG grade of the aged binder to that of the original grade of the virgin binder. For this purpose, a softening curve for each RA was established. To establish the softening curve, the aged binder was mixed with a different amount of RA and its high PG was measured. Then, the softening curve for each RA was drawn based on the RA percentage and high PG grade. Finally, the amount of RA was selected from the curve based on the desired high PG grade.

The testing procedure shown in Figure 2-2 was followed to evaluate the asphalt binder performance and define its rheological and chemical properties in each aging step. In this evaluation, the Fourier Transform Infrared Spectroscopy-Attenuated Total Reflection (FTIR-ATR), Dynamic Shear Rheometer (DSR) and Bending Beam Rheometer (BBR) tests were conducted. The standard specifications used to perform these tests are shown in Table 2-1.

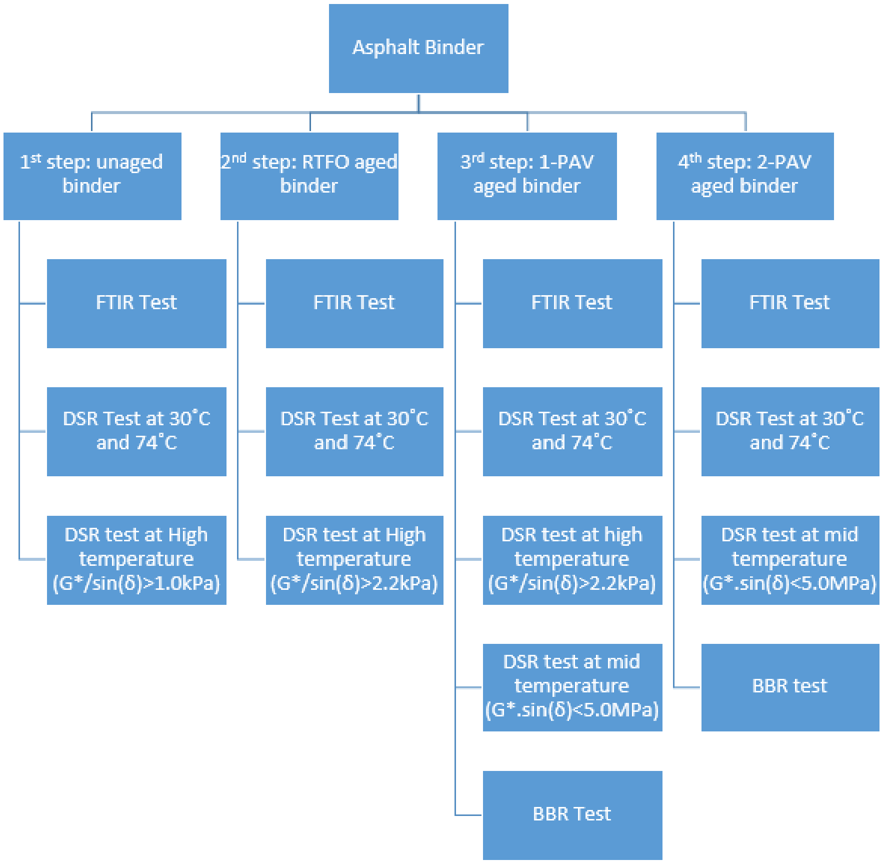


Figure 2-2: Testing procedure of binders

Table 2-1: Standard specifications to perform DSR and BBR tests

Specification	Aging Step	Criteria	Comment
AASHTO T315 Criterion(AASHTO T315 2012)	Unaged	G*/sinδ>1.0 kPa	DSR test at high temperature and 10 rad/s
	RTFO-aged	G*/sinδ>2.2 kPa	DSR test at high temperature and 10 rad/s
	1 PAV-aged	G*.sinδ<5 MPa	DSR test at mid-temperature and 10 rad/s
	2 PAV-aged	G*.sinδ<5 MPa	DSR test at mid-temperature and 10 rad/s
AASHTO T313 Criterion(AASHTO T313 2012)	1 PAV-aged	S<300 MPa m>0.30	BBR test at -12 ˚C and 60 seconds
	2 PAV-aged	S<300 MPa m>0.30	BBR test at -12 ˚C and 60 seconds

The FTIR test was conducted to measure the chemical indices of binders in different aging steps. The FTIR is a device that measures the light absorbed by a solid or liquid. It evaluates the absorption of the infrared spectrum of a sample using high spectra and defines the intensity of absorption over a wavelength. It can absorb waves in a wide range of spectra (400-4000 cm^{-1}), and the peaks in spectra data can be used to detect the chemical composition functional group of asphalt bonds. Different asphalt bonds can absorb various infrared frequency wavelengths. The chemical functional groups that contain oxygen and change during the oxidation of asphalt are carbonyls and sulfoxides. The carbonyls and sulfoxides can be detected approximately at wavelength number 1702 cm^{-1} and 1032 cm^{-1}, respectively. This test was conducted on binders from wavenumbers 400 to 4000 cm-1, with a resolution of 8 cm-1. The diamond crystal of FTIR was cleaned after each test with ethanol.

The DSR is a device that measures the viscous and elastic behavior of asphalt binder. Complex shear modulus (G*) and phase angle (δ) are the two viscous properties of asphalt binder that can be measured by the DSR test. This test can be conducted at a high and mid-temperature to determine the asphalt binder Superpave high and mid-temperature PG grade. The high PG grade of the binder was calculated using Equation 2-1 and by conducting two DSR tests at two different high temperatures. Similarly, the mid-temperature PG grade of the binder was calculated using Equation 2-1 and by conducting two DSR tests at two different medium temperatures. In addition, to correlate the rheological and chemical properties of asphalt binder in different aging steps, the DSR test was conducted at 30 ˚C and 74 ˚C as a representative of medium and high temperatures of asphalt binder performance. It should be noted that the rutting performance of asphalt pavement can be evaluated using G*/sinδ by performing the DSR test at a high temperature. However, the fatigue performance of the binder can be identified using G*sinδ by performing the DSR test at an intermediate temperature. As rutting performance of asphalt pavement is only a concern in early aged pavement, the high-temperature DSR test should be conducted on unaged and RTFO-aged binders. This means that the binder should have recoverable behavior in the early stage of aging. Therefore, the elastic portion of the complex shear modulus G*/Sinδ should be high in the early aged binder. On the other hand, the fatigue performance of asphalt pavement is only a concern in the long-term aged binder. This indicates that the binder should dissipate energy due to an applied load with no cracking. Therefore, the DSR test at mid-temperature should be conducted on the 1 PAV- and 2 PAV-aged binders.

Furthermore, the low-temperature cracking resistance of asphalt binder can be indicated using the BBR test. The BBR test measures the creep stiffness (S) and m-value. Creep stiffness is an indication of thermal stresses in the binder due to contraction, and the m-value is the slope of the master stiffness curve, which determines the rate of stress relief. The low-temperature grade of samples in each aging step can be calculated using the extrapolation of BBR results at two different low temperatures through the use of Equations 2-2 and 2-3. Since the low-temperature grade of binders is a concern for long-term aged binders, the BBR test was conducted on 1 PAV- and 2 PAV-aged samples.

$$T_c = T_1 + [\frac{Log(1.0) - Log(\frac{G_1^*}{sin\delta_1})}{Log\left(\frac{G_1^*}{sin\delta_1}\right) - Log(\frac{G_2^*}{sin\delta_2})} \tag{Equation 2-1}$$

$$T_c = T_1 + \left[\frac{Log(300) - Log(S_1)}{Log(S_1) - Log(S_2)}\right] - 10 \tag{Equation 2-2}$$

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

2.2 Applicability of Nanomodified Binder in Recycling Process

In this study, the Superpave PG standard tests and aging process were conducted. In the first step of the aging process, the two nanomodified and neat (unmodified) asphalt binders were aged separately using a Rolling Thin-Film Oven (RTFO) and two cycles of Pressurized Aging Vessel (PAV) to prepare the aged binders (Figure 2-3). In each step of the aging process, the rheological and chemical tests were conducted to monitor the changes in binder performance and properties.

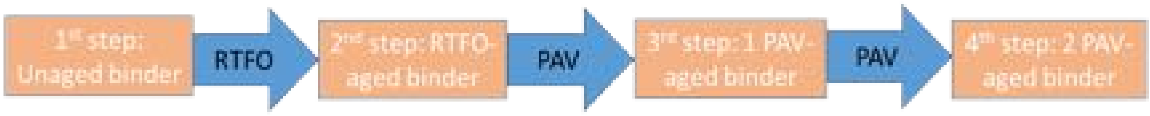


Figure 2-3: Aging and recycling process of binders

The testing procedure shown in Figure 2-2 was followed to evaluate the performance of the asphalt binder and define its rheological and chemical properties in each aging step. In this evaluation, the Fourier Transform Infrared Spectroscopy-Attenuated Total Reflection (FTIR-ATR), Dynamic Shear Rheometer (DSR), and Bending Beam Rheometer (BBR) tests were conducted. The standard specifications used to perform these tests are shown in Table 2-1.

2.3 Recycling of Asphalt Using Nanomodified Binder

In this study, the Superpave PG standard tests and aging process were conducted. In the aging process of the binder, the virgin binder was aged using a Rolling Thin-Film Oven (RTFO) and two cycles of Pressurized Aging Vessel (PAV) to prepare the aged binder (Figure 2-4). Then, the different percentages of the aged binder were mixed with a soft virgin binder or nanomodified binder to partially recycle the aged binder. Finally, the partially recycled binders with different percentages of RAP binder were aged using RTFO and two cycles of PAV to artificially simulate the aging of recycled binders.

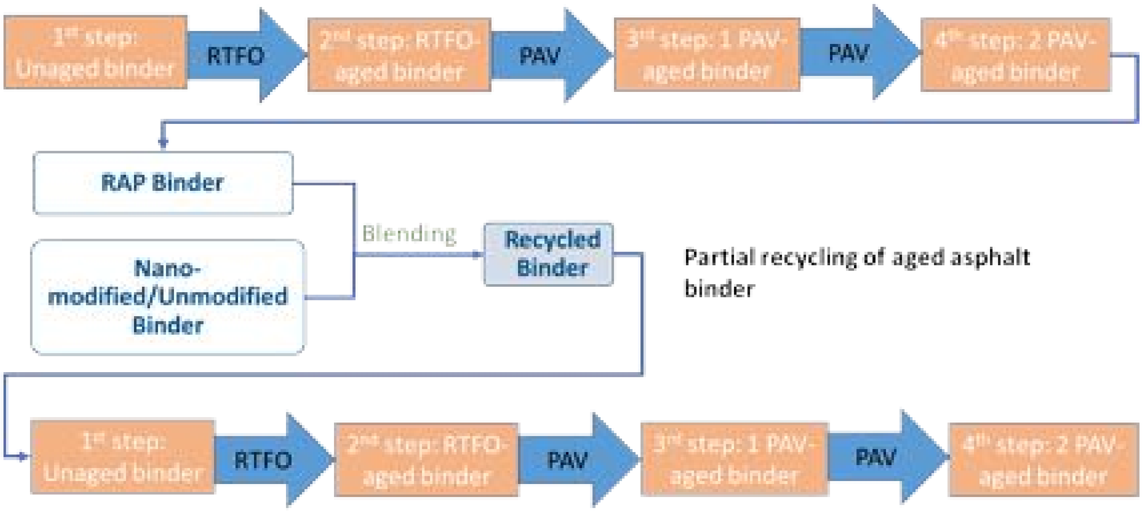


Figure 2-4: Aging and recycling process of binders

The recycling process of the asphalt binder included heating the aged, nanomodified, and unmodified asphalt binders to 155±5 ˚C, and then adding the desired amount of aged binder to the modified or unmodified binders and mixing them for two minutes. The soft binders and aged binders were mixed using a spatula. The 10%, 20%, and 30% aged asphalt binders were added to the soft binders. The percentage of aged binders (RAP binder) is different from the content of RAP materials in the asphalt mixture. By considering a 6% asphalt binder in an asphalt mixture by the total amount of aggregates, the equivalent amount of RAP material mixtures is calculated in Table 2-2. This means that mixing the 10% asphalt binder with the soft binder is equal to the incorporation of 17% of RAP material in the recycled asphalt pavement mixture.

Table 2-2: RAP materials incorporation in the recycling process

RAP binder percentage by mass	RAP
10%	17%
20%	33%
30%	50%

The testing procedure shown in Figure 2-2 was followed to evaluate the asphalt binder performance and define its rheological and chemical properties in each aging step. In this evaluation, the Fourier Transform Infrared Spectroscopy-Attenuated Total Reflection (FTIR-ATR), Dynamic Shear Rheometer (DSR), and Bending Beam Rheometer (BBR) tests were conducted.

3 CHAPTER 3: RHEOLOGICAL AND CHEMICAL CHARACTERISTICS OF ASPHALT BINDERS RECYCLED USING DIFFERENT RECYCLING AGENTS

3.1 Introduction

The incorporation of reclaimed asphalt pavement (RAP) in the construction of new asphalt pavement is limited due to issues with the short-term and long-term performance of pavement. To enhance the aged binder properties, recycling agents (RAs) are added to RAP mixtures. There are different RAs available, and they impact the asphalt binder properties differently, resulting in various aging rates of recycled asphalt. Therefore, in this study, the rheological and chemical characteristics of 100% recycled asphalt binder using different RAs were investigated using the Dynamic Shear Rheometer (DSR), Bending Beam Rheometer (BBR), and Fourier-Transform Infrared Spectroscopy - Attenuated Total Reflection (FTIR-ATR) tests. The Rolling Thin-Film Oven (RTFO) and Pressure Aging Vessel (PAV) were used to simulate the short-term and long-term aging of asphalt binder, respectively. The long-term aging of the binder was simulated by aging asphalt binder with two PAV cycles. This study aims to identify the proper RA for the recycling of binders according to the aging rate, and the short- and long-term performance of recycled asphalt binder. The results showed that the recycled binders with a specific RA that have the minimum initial concentration of sulfoxide and carbonyl will provide better performance compared to recycled binders with other RAs. This result can be used to predict the long-term performance of recycled asphalt, in addition to identifying the proper RA in the recycling process of asphalt pavement.

3.2 Objectives

Different studies have been conducted on the recycling of a 100% RAP asphalt binder using different RAs. The results showed that RAs can be used to recycle 100% RAP. Various RAs have different impacts on the RAP properties, resulting in different aging rates of recycled asphalt during the service life of pavement. Moreover, the relationship between the chemical composition and physical properties of the recycled binder have followed different trends. Therefore, in this study, the chemical (C and S) and rheological (G*, δ, and creep stiffness) properties of recycled binders and their changes during the aging process were investigated. Different RAs and virgin binders were used to identify the aging rate of binders to select proper RAs in the recycling of asphalt pavement by considering pavement performance. In this evaluation, the rutting, fatigue, and low-temperature cracking resistance of asphalt binder were considered. This study's results could also be used to predict the performance of asphalt pavement using the unaged binder chemical properties of recycled asphalt, as well as identify the maintenance and treatment time needed for pavement.

3.3 Materials

Bitumen chemical properties are dependent on the source of the binder, resulting in different molecule components for different types of binders. As a result, two kinds of virgin asphalt binders with the PG 67-22 grade from two different sources that are commonly used in Florida were selected. It was expected that the high-temperature PG of virgin (original) binders were higher than 67 ˚C (AASHTO M320). The critical low temperature of binders was also expected to have an m-value less than 0.3 and a creep stiffness (S) more than 300 MPa at -12 and 60 seconds (AASHTO M320).

Several RAs with different chemical and physical properties were available in the market at the time of this study; six RAs were selected from various sources. Eleven RAs were screened and then chosen if they met the Florida Department of Transportation (FDOT) specification requirements (FDOT 2017). The properties of RAs are shown in Table 3-1. As shown, RAs were naphthenic-, paraffinic-, and biomaterial-based to cover different RAs with different features.

Table 3-1: Properties of RAs

RA	Product Name	Viscosity	Flashpoint COC (˚C)	Appearance
RA 1	Heavy paraffinic distillate solvent extract	100 (cSt) at 60 ˚C	210 min	Dark yellow, heavy oil
RA 2	Solvent-dewaxed heavy paraffinic distillate	22 (cSt) at 60 ˚C	254	Dark yellow, light oil
RA 3	Bio-RA, plant material base	25 (cSt) at 60 ˚C	221	Green
RA 4	Polyol ester oil	40 (cSt) at 40 ˚C	280	Clear and bright
RA 5	Heavy naphthenic distillated solvent	25 (cSt) at 60 ˚C	182	Brown liquid
RA 6	Heavy paraffinic distillate solvent extract	92 (cSt) at 40 ˚C	420	Opaque liquid

3.4 Results

3.4.1 Virgin Binder

Two kinds of virgin binders from two sources were selected. The high PG of binders A and B in the different steps of the aging process were measured using the DSR test at high temperatures, as shown in Table 3-2. As shown, the high PG grade of binders A and B increased 17.5 and 16 degrees during the aging process, respectively. These changes can demonstrate that binder B’s performance with its high PG is better than that of binder A, due to its lower grade increment as it ages.

Table 3-2: High PG of virgin binders in different steps of aging

Aging Step	Binder A				Binder B			
	Test Temperature (˚C)	δ (˚)	G*/sin δ (kPa)	High PG (℃)	Test Temperature (˚C)	δ (˚)	G*/sin δ (kPa)	High PG (℃)
Unaged	70	84	2.20	74.54	70	85	1.27	72.05
	76	87	0.78		76	87	0.63	
RTFO-aged	74	82	2.84	75.99	74	87	2.10	73.63
	80	85	1.32		80	86	0.99	
1 PAV-aged	82	81	2.51	83.19	74	79	4.35	80.08
	88	84	1.30		80	82	2.22	
2 PAV-aged	82	75	7.11	92.05	80	75	7.84	88.05
	88	78	3.53		86	80	3.04	

The measured mid-temperature PG grade of binders A and B are shown in Table 3-3. As shown, both virgin binders met the fatigue performance criteria ($G^*.\sin\delta<5$ MPa at 26.5 ˚C). Also, it can be concluded that binder B had better fatigue performance compared to binder A, due to its lower $G^*.\sin\delta$ value.

Table 3-3: Mid PG of virgin binders in different steps of aging

Aging Step	Binder A				Binder B			
	Test Temperature (˚C)	δ (˚)	G*sin δ (MPa)	Mid PG (℃)	Test Temperature (˚C)	δ (˚)	G*sin δ (MPa)	Mid PG (℃)
Unaged	24	63	1.44	16.06	24	64.5	0.96	14.19
	30	68	0.56		30	69	0.35	
RTFO-aged	24	52	2.78	17.70	24	53.2	1.81	15.81
	30	57	1.59		30	66.2	0.86	
1 PAV-aged	24	44	3.92	21.24	24	43.9	2.79	19.03
	30	49	2.30		30	49.9	1.38	
2 PAV-aged	24	41	5.70	25.36	24	41.2	3.94	22.11
	30	45	3.20		30	46.1	1.85	

The low-temperature cracking resistance of the asphalt binder was measured and is displayed in Table 3-4. As shown, both binders met the cracking resistant criteria ($S<300$ MPa, $m>0.3$ at -12 ˚C and 60 seconds) based on AASHTO T31. Also, the low-temperature cracking resistance of binders A and B was almost the same because their low-temperature grades were similar. Also, the low-temperature performance of binders degraded with aging due to the decrease in the low-temperature grade of the binders. Specifically, the critical temperatures calculated from the m-value were more critical than those calculated from creep stiffness because of the higher low-temperature grade results.

Table 3-4: BBR test results for virgin binders at 60 seconds

Binder	Aging Step	Temperature (˚C)	Stiffness (MPa)	m-value	Critical Temperature (Stiffness)	Critical Temperature (m-value)
A	1 PAV Cycle	-12	185	0.329	-27.84	-25.48
		-18	304	0.279		
	2 PAV cycle	-12	267	0.311	-23.79	-22.66
		-18	395	0.211		
B	1 PAV Cycle	-12	181	0.338	-26.68	-24.48
		-18	346	0.246		
	2 PAV cycle	-12	205	0.311	-25.92	-22.81
		-18	367	0.23		

Figure 3-1 indicates the changes in the sulfoxide and carbonyl indexes with aging that were measured using the FTIR test. As shown, the sulfoxide and carbonyl indexes increased with the aging of the binders. Also, the concentration of sulfoxide and carbonyl of binder B was lower than that of binder A. In Figure 3-2, the correlation between carbonyl plus sulfoxide and log G* and phase angle (δ) of the binders is displayed. As expected, there was a strong correlation between the chemical and rheological properties of the binders during the aging of the virgin binders.

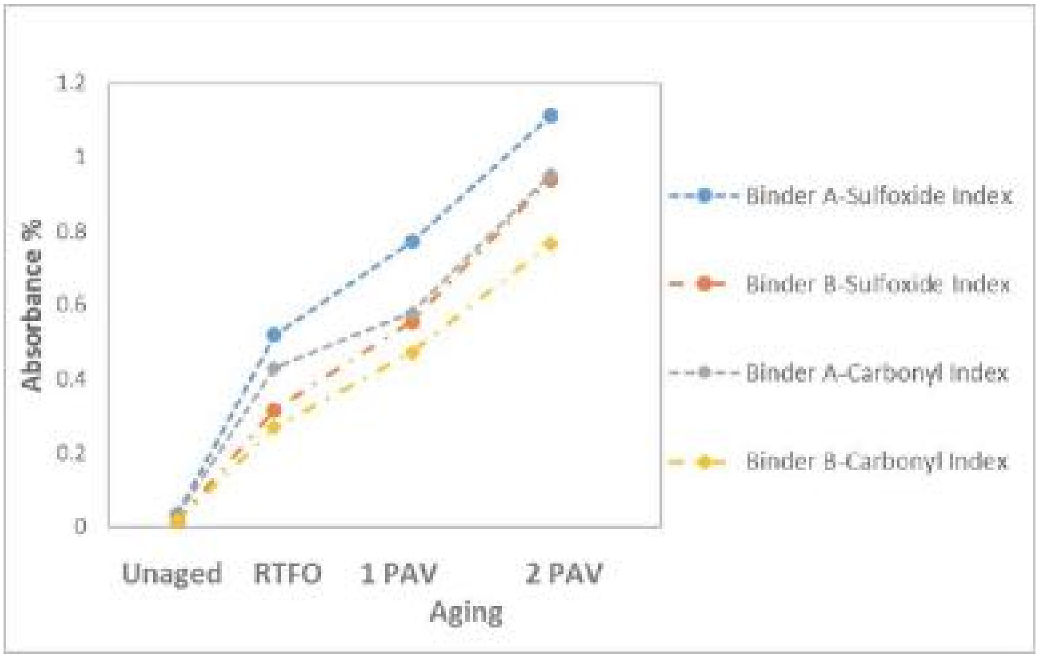


Figure 3-1: The change in carbonyl and sulfoxide indexes with aging

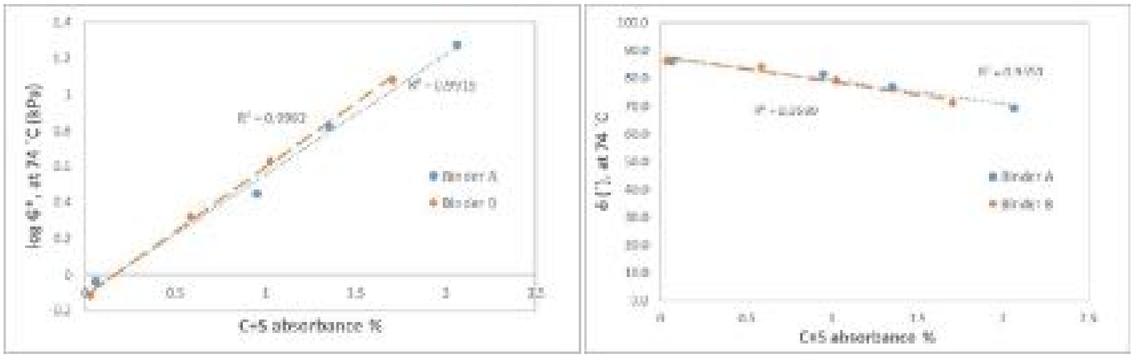


Figure 3-2: Correlation between rheological and chemical properties of binders

3.4.2 Softening Curves

The aged binder was mixed with different percentages of RAs to construct the softening curves. Figure 3-3 shows the softening curves for aged binders A and B, which were recycled using different percentages of RAs. These curves were constructed based on a high PG of recycled binders with different RA content. The goal of this procedure was to define the amount of RA that can recover the high PG of an aged binder to its original grade (virgin binder with a high PG grade). The virgin binders A and B high PG were 74.5

˚C and 72 ˚C, respectively (Table 3-2). Accordingly, the required amount of RA to soften aged binders A and B to recover binder A to a high PG of 74±1 ˚C and binder B to 72±1 ˚C is displayed in Table 3-5. As shown in Table 3-5, the amount of RA required to soften the aged binder B was less than the amount needed for the aged binder A.

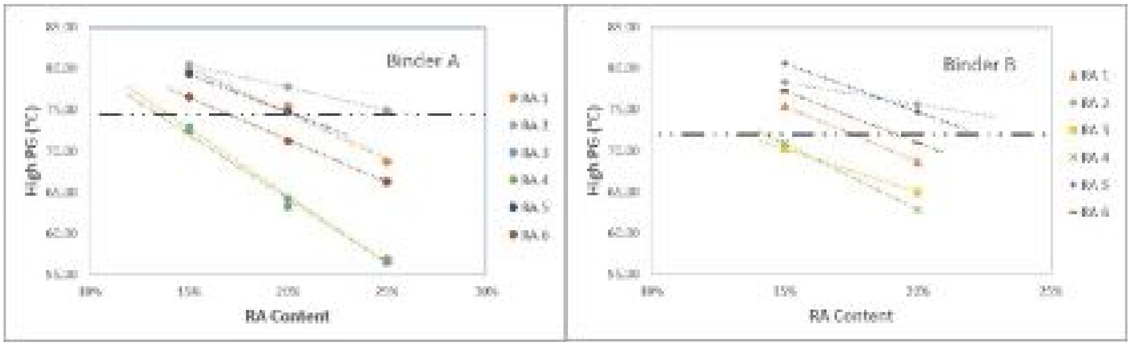


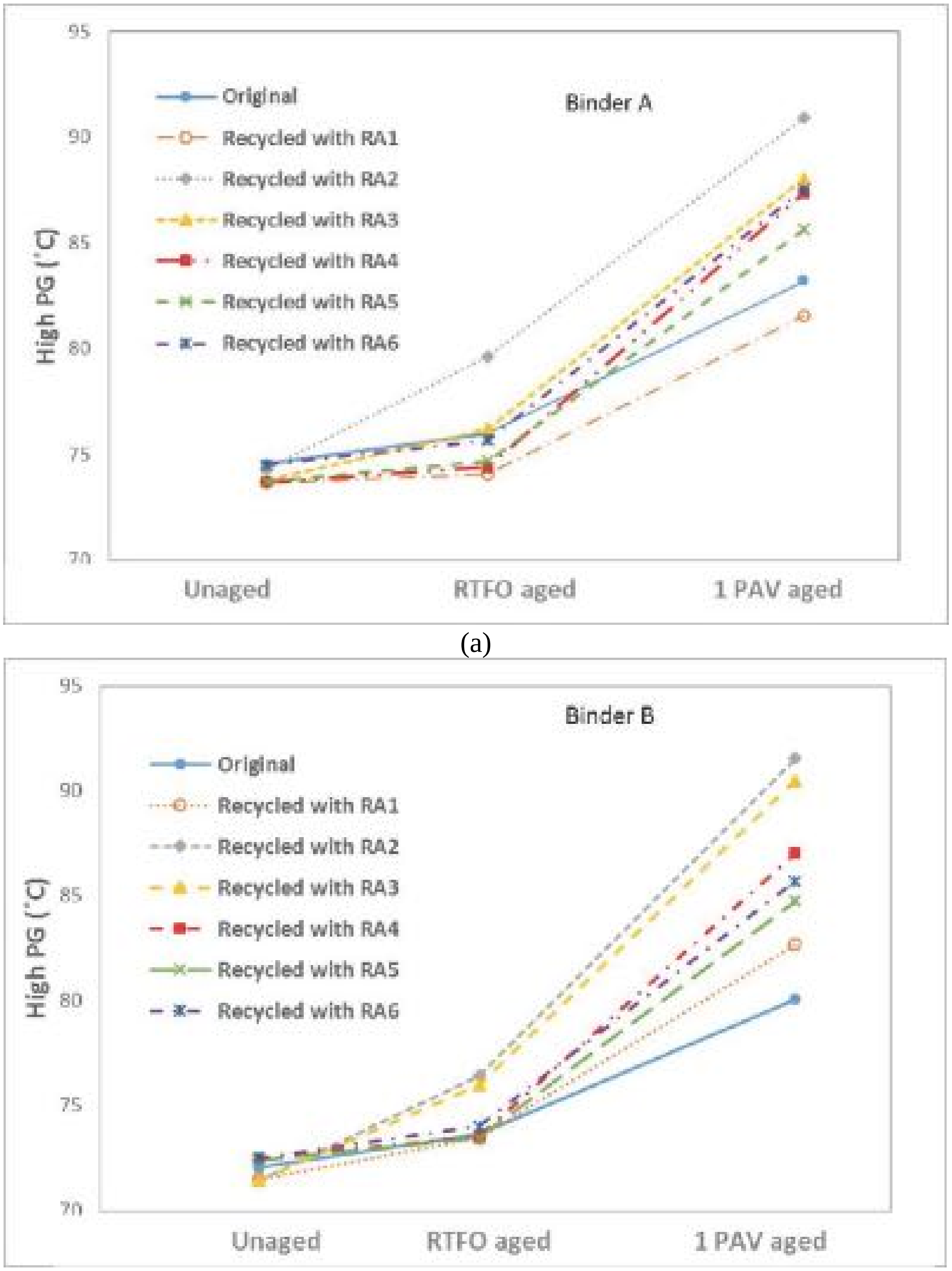
Figure 3-3: RA softening curves for binders A and B

Table 3-5: Required RA content to soften the aged binder to original PG

Binder	RA1	RA2	RA3	RA4	RA5	RA6
A	21%	26%	14%	15%	23.5%	18.5%
B	20%	24%	13%	14%	22%	17.5%

3.4.3 High-Temperature Performance of Recycled Binders

The aged binders were recycled using six different RAs and were then re-aged. To evaluate the rutting performance of the recycled binders, the DSR test was performed at high temperatures in unaged and RTFO-aged and the 1 PAV-aged recycled binders; the results are displayed in Figure 3-4. As shown, the recycled binder A with RA2 and RA3 had better rutting performance than the original binder because the slope of their high PG change is higher than the original binder. Similarly, recycled binder B with RA2 and RA3 has a better rutting performance than the original binder. Also, the recycled binder with RA1, RA4, and RA5 had the worst rutting performance compared to the original binder performance.

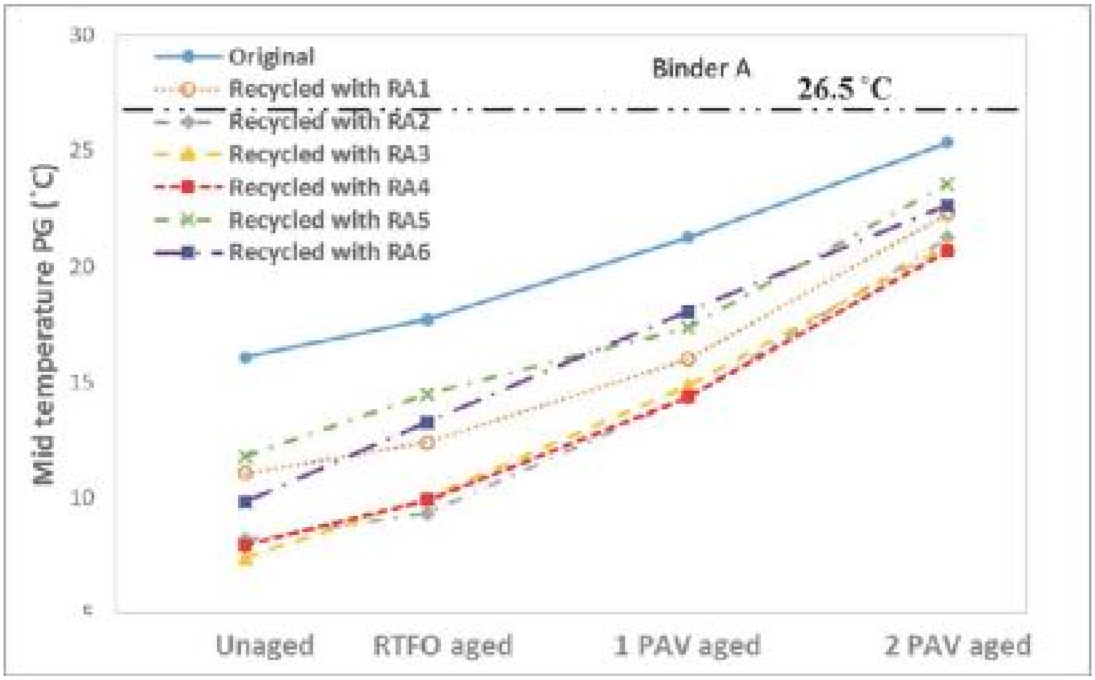

(a)

(b)

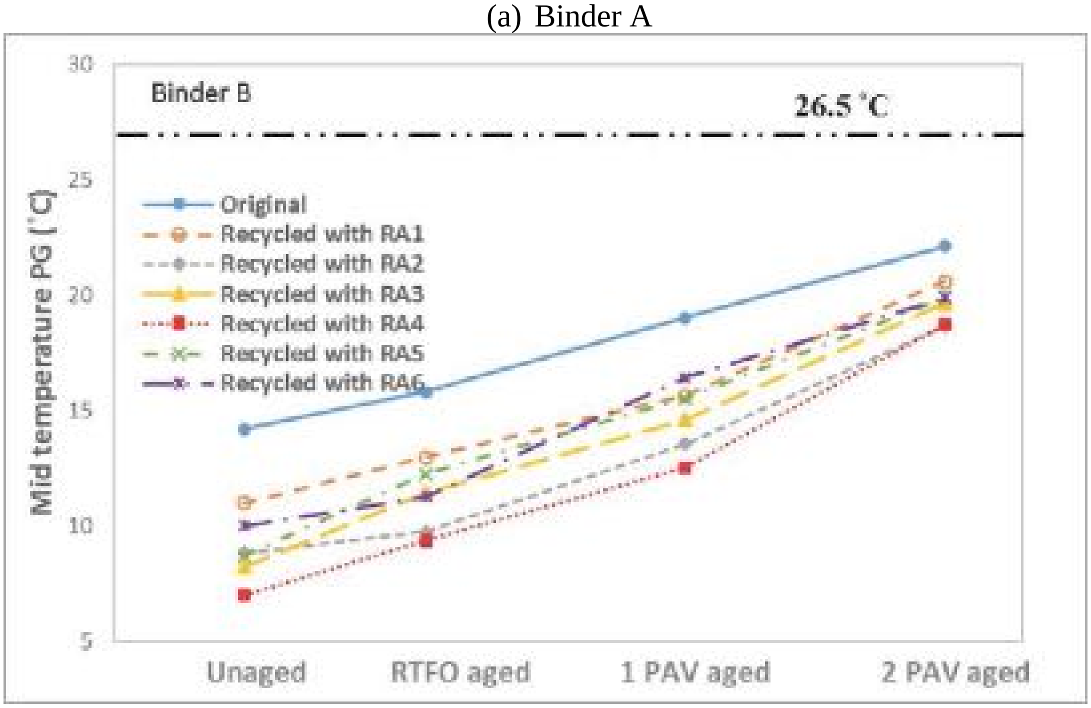
Figure 3-4: High-temperature PG of recycled binder a) A and b) B with different RAs

3.4.4 Mid-Temperature Performance of Recycled Binders

The mid-temperature grade of recycled binders with different RAs in the various steps of aging were measured using the DSR test. As shown in Figure 3-5, all of the RAs could recover the mid-temperature of recycled binders. In fact, the properties of binders at the 1 PAV- and 2 PAV-aged steps were more important because the mid-temperature of the binder is representative of the long-term and fatigue performance of the binder. Accordingly, the recycled binders' fatigue performance was not a concern because they had a lower mid-temperature PG than the original binder. Also, by considering the mid-PG of the recycled binder at 1 PAV and 2 PAV and 26.5 ˚C as the PAV failure point according to AASHTO T315, it was possible to determine the PAV time that each binder took to reach the failure point. Consequently, a durability index was introduced as the PAV failure time of the recycled binder to the corresponding original binder. This index can demonstrate the effectiveness and durability of the recycled binder compared to the virgin binder's performance. As shown in Table 3-6, the PAV failure time and durability of all of the recycled binders were higher than the original binders. However, the performance of the recycled binder with RA2 and RA3 was better than the other recycled binders.



(a) Binder A



(b) Binder B

Figure 3-5: Mid-temperature PG of recycled binders A and B with different RAs

Table 3-6: Durability and PAV failure time of recycled binders

Longevity		Original	RA1	RA2	RA3	RA4	RA 5	RA6
Binder A	PAV failure time	46.5	58	61	62.7	57.6	55	56.3
	Durability Index	1.00	1.25	1.30	1.35	1.26	1.18	1.21
Binder B	PAV failure time	62.3	73	76	75	75.3	75	69.4
	Durability Index	1.00	1.17	1.22	1.20	1.16	1.14	1.11

3.4.5 Low-Temperature Performance of Recycled Binders

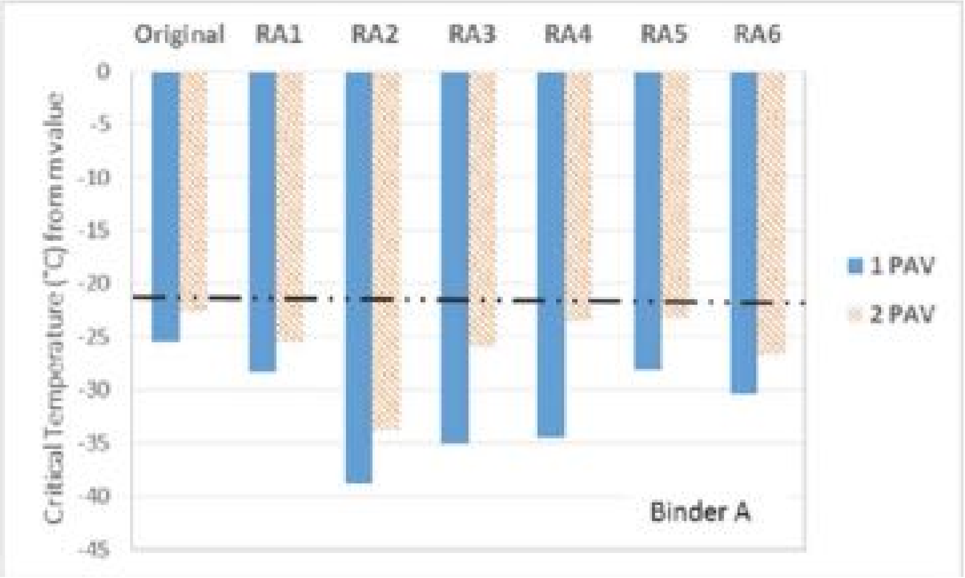
Also, the cracking resistance of binders at low temperatures was evaluated using the BBR test. The m-value and creep stiffness of virgin binders and recycled binders in the 1 PAV- and 2 PAV-aged steps were measured and are shown in Tables 3-7 and 3-8. All of the recycled binders met the low-temperature cracking criteria of AASHTO T 313 ($S<300$ MPa and $m>0.3$ at 60 seconds and -12 ˚C). Also, the recycled binders were significantly softer and had lower creep stiffness compared to the original binders. The critical low temperatures calculated using the m-value were more critical than those calculated by stiffness and resulted in higher low-temperature grades. As the binders aged, the stiffness increased. Adversely, the m-value decreased with aging. In addition, the low-temperature grades of recycled binders calculated using the m-values were compared in Figure 3-6. As shown, all of the recycled binders had better low-temperature performance than that of the original binders because their low temperature was lower than that of the original binders.

Table 3-7: Binder A's BBR test results at 60 seconds

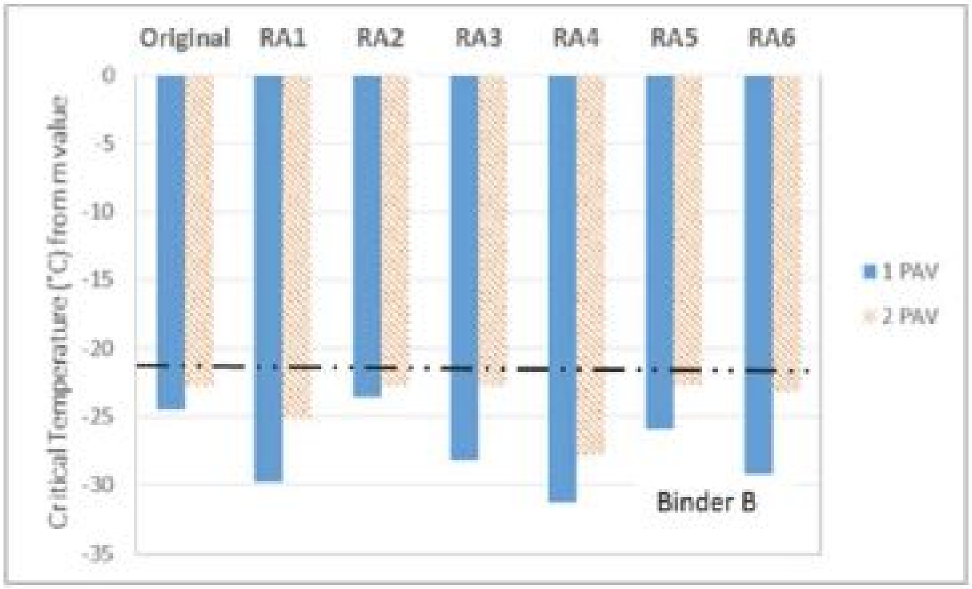
RA	Aging Step	Temperature (˚C)	Stiffness (MPa)	m-value	Critical Temperature (Stiffness)	Critical Temperature (m-value)
Original	1 PAV Cycle	-12	185	0.329	-27.84	-25.48
		-18	304	0.279		
	2 PAV cycle	-12	267	0.311	-23.79	-22.66
		-18	395	0.211		
RA 1	1 PAV Cycle	-12	59.2	0.34	-34.38	-28.32
		-18	130	0.302		
	2 PAV cycle	-12	181	0.324	-29.65	-25.43
		-18	269	0.282		
RA 2	1 PAV Cycle	-12	34	0.398	-46.54	-38.80
		-18	57.9	0.363		
	2 PAV cycle	-12	40.6	0.351	-41.47	-33.77
		-18	75.2	0.325		
RA 3	1 PAV Cycle	-12	70.3	0.368	-49.76	-35.16
		-18	96.2	0.337		
	2 PAV cycle	-12	131	0.325	-31.12	-25.95
		-18	226	0.287		
RA 4	1 PAV Cycle	-12	36.6	0.352	-42.93	-34.48
		-18	66.9	0.327		
	2 PAV cycle	-12	237	0.318	-25.19	-23.57
		-18	369	0.249		
RA 5	1 PAV Cycle	-12	141	0.385	-30.45	-28.14
		-18	241	0.302		
	2 PAV cycle	-12	202	0.315	-24.66	-23.29
		-18	493	0.245		
RA6	1 PAV Cycle	-12	61.9	0.352	-36.68	-30.43
		-18	118	0.315		
	2 PAV cycle	-12	149	0.323	-29.76	-26.60
		-18	256	0.293		

Table 3-8: Binder B’s BBR test results at 60 seconds

RA	Aging Step	Temperature (˚C)	Stiffness (MPa)	m-value	Critical Temperature (Stiffness)	Critical Temperature (m-value)
Original	1 PAV Cycle	-12	181	0.338	-26.68	-24.48
		-18	346	0.246		
	2 PAV cycle	-12	205	0.311	-25.92	-22.81
		-18	367	0.23		
RA 1	1 PAV Cycle	-12	88.9	0.403	-33.57	-29.73
		-18	167	0.323		
	2 PAV cycle	-12	127	0.334	-31.23	-25.14
		-18	222	0.269		
RA 2	1 PAV Cycle	-12	39.9	0.317	-48.05	-23.57
		-18	63.5	0.252		
	2 PAV cycle	-12	46.1	0.309	-43.42	-22.83
		-18	77.9	0.244		
RA 3	1 PAV Cycle	-12	115	0.333	-35.52	-28.19
		-18	176	0.301		
	2 PAV cycle	-12	276	0.305	-23.06	-22.77
		-18	442	0.266		
RA 4	1 PAV Cycle	-12	27.5	0.382	-42.74	-31.28
		-18	54.9	0.329		
	2 PAV cycle	-12	95.1	0.329	-38.08	-27.80
		-18	146	0.299		
RA 5	1 PAV Cycle	-12	106	0.328	-36.72	-25.91
		-18	162	0.285		
	2 PAV cycle	-12	282	0.307	-23.82	-22.72
		-18	346	0.249		
RA6	1 PAV Cycle	-12	86.2	0.361	-34.10	-29.96
		-18	160	0.315		
	2 PAV cycle	-12	205	0.303	-28.28	-23.20
		-18	295	0.288		



(a)



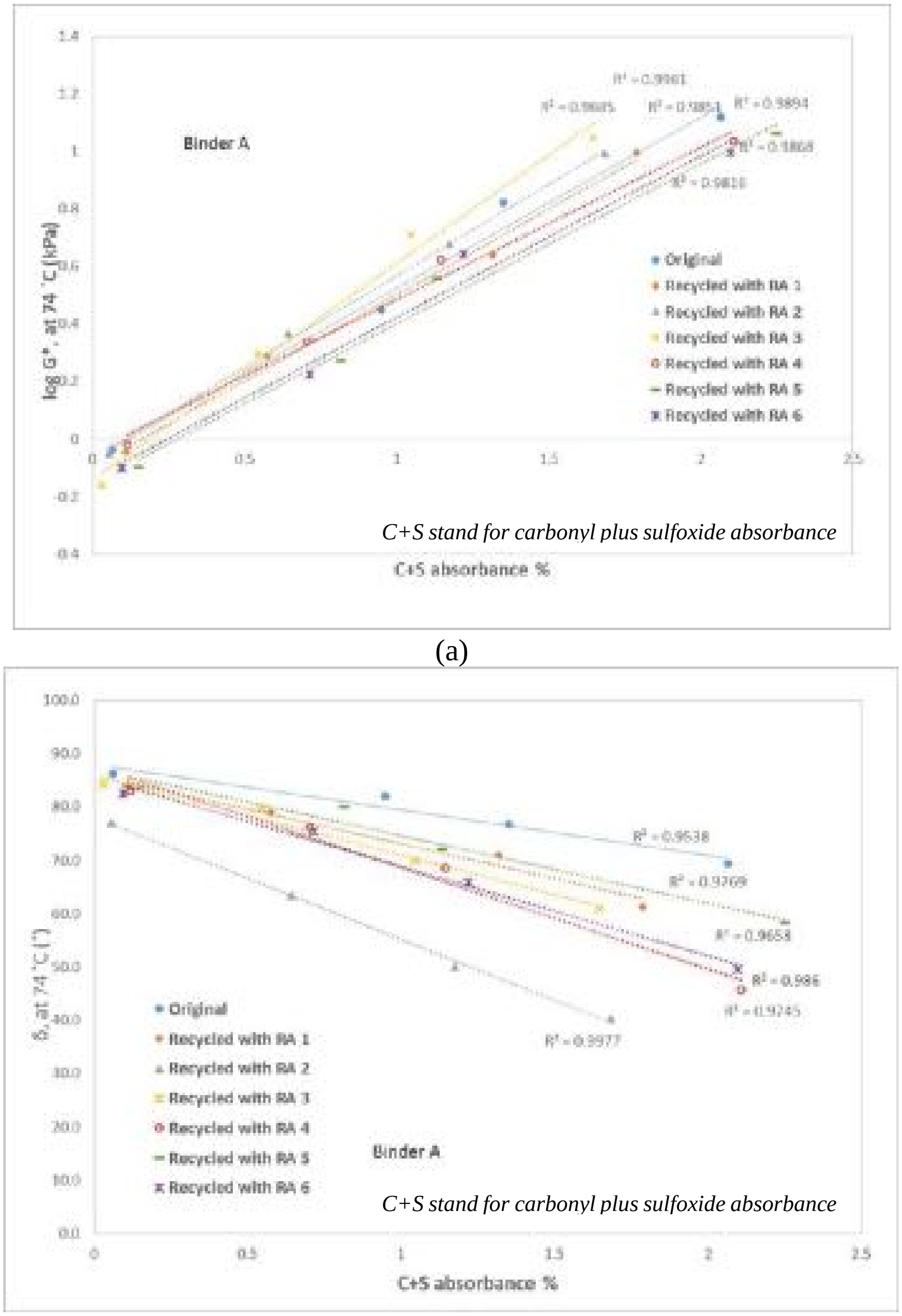
(b)

Figure 3-6: Calculated critical low temperature of a) recycled binder A and b) recycled binder B using m-values

3.4.6 Chemical Indexes Changes of Recycled Binders

The correlation between the chemical and rheological indexes of recycled binders was measured and is shown in Figures 3-7 and 3-8 for binders A and B, respectively. In these figures, G* and δ were measured at 74 ˚C in the unaged, RTFO-aged, and 1 PAV- and 2

PAV-aged binders recycled with different RAs. Also, the carbonyl (C) and sulfoxide (S) indexes were measured using the FTIR test in different aging steps. As shown, there was a strong correlation between chemical indexes (C+S) and rheological indexes (logG* and δ). Also, the production of C and S with aging and logG* increased. This increment indicates that C and S were indicative of the binder's age. However, the δ were decreased by aging and the increase of C and S. The recycled binders with RA2 and RA3 have a steeper slope compared to virgin binders. By considering the logG* and the chemical indexes figure for binders A and B, the recycled binders with RA2 and RA3 started below the original binder, and then crossed the original binder and went up. This trend means that the concentration of C and S and magnitude of G* were less than the original binder at the unaged step. Similarly, by considering the δ and the chemical indexes (Figure 3-7, 3-8) for binders A and B, it can be demonstrated that all of the recycled binders' phase angles were less than that of the original binders. This means recycled binders had more elastic behavior compared to the virgin binders. It should be noted that the similar trend that was shown in Figures 3-7 and 3-8 was also shown with the absorbance of carbonyl or sulfoxide separately. Due to the following of a similar trend, these figures were not shown here.



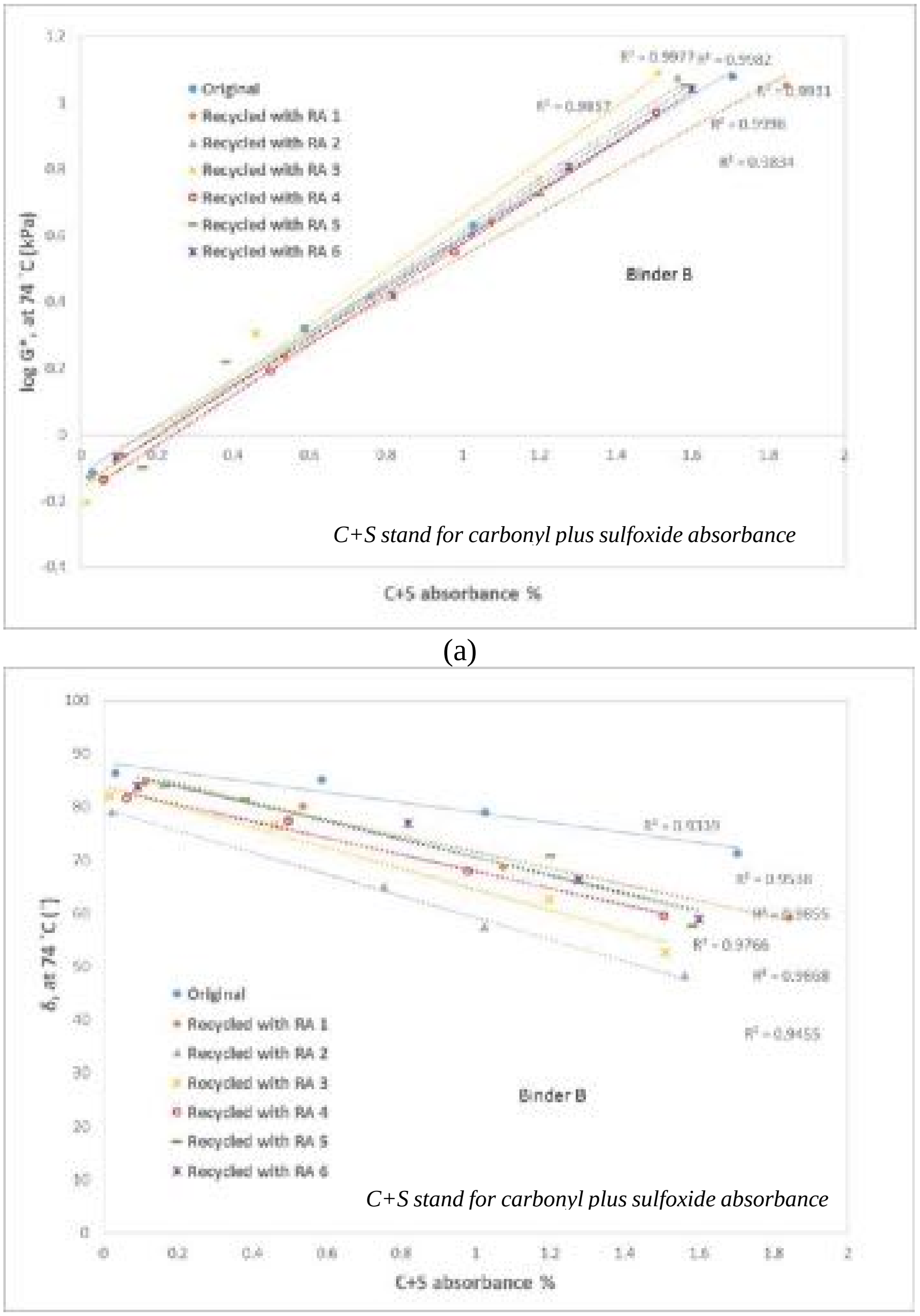
C+S stand for carbonyl plus sulfoxide absorbance

(a)

C+S stand for carbonyl plus sulfoxide absorbance

(b)

Figure 3-7: Correlation between chemical and rheological indexes measured at 74 ˚C. Correlation between (a) C+S absorbance and log G, (b) C+S absorbance and δ (˚) of recycled binder A with different RAs*



C+S stand for carbonyl plus sulfoxide absorbance

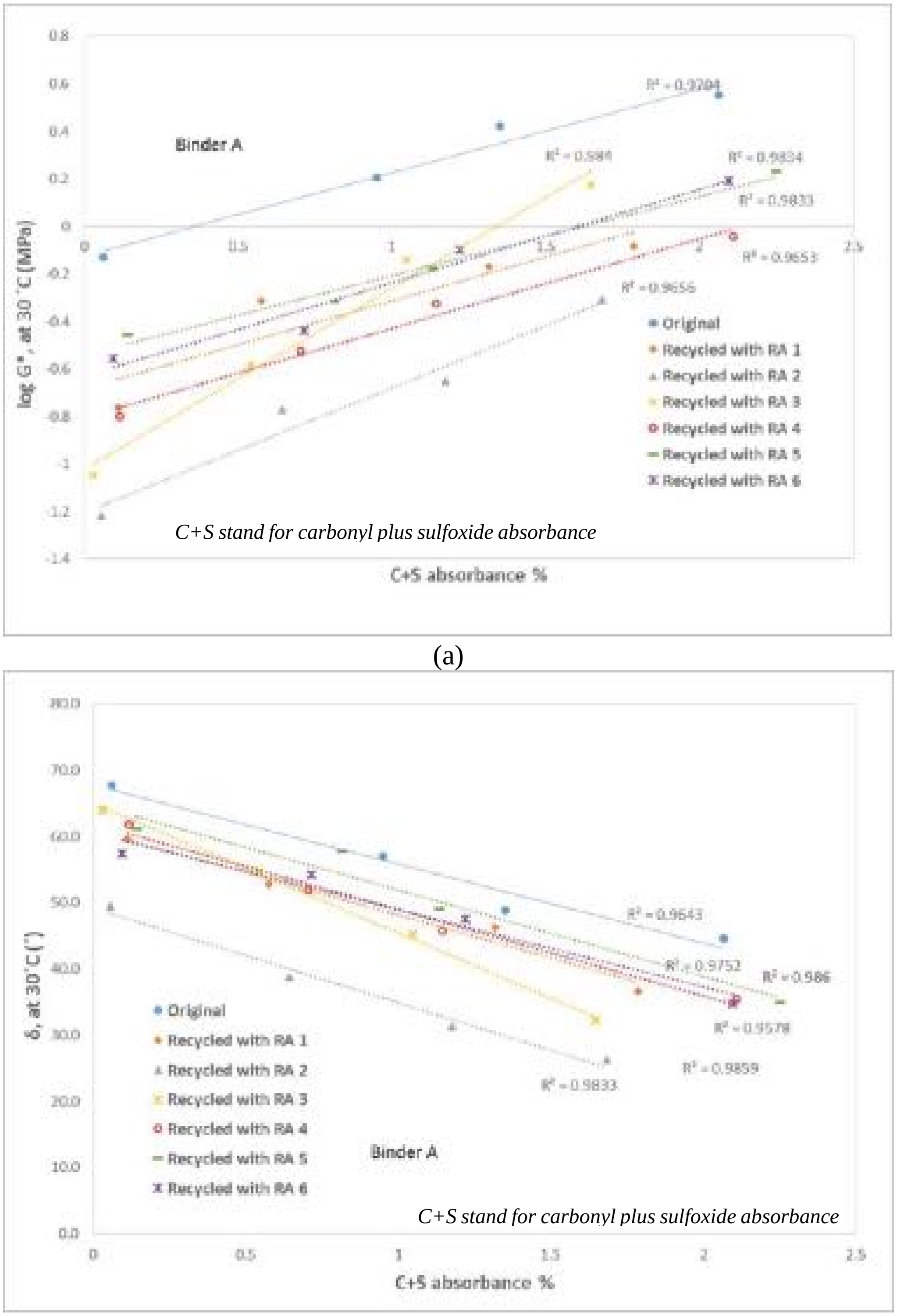
(a)

C+S stand for carbonyl plus sulfoxide absorbance

(b)

Figure 3-8: Correlation between chemical and rheological indexes measured at 74 ˚C. Correlation between (a) C+S absorbance and log G, (b) C+S absorbance and δ (˚) of recycled binder B with different RAs*

Figures 3-9 and 3-10 also show the strong correlation between logG* and δ measured at mid-temperature (30 ˚C) with chemical indexes (C and S). The recycled binders have significantly lower G* and δ than the original binders, especially in the recycled binders with RA2 and RA3. Also, the trends that were followed during the aging of the recycled binders with RA2 and RA3 were different from the original binder and other recycled binders. This trend was followed in both binders A and B. This is similar to the trend shown in Figures 9 and 10, but is more significant. Also, the decrease of δ with aging for the recycled binders with RA2 and RA3 was more significant than other recycled binders. It should be noted that the similar trend that was shown in Figures 3-9 and 3-10 was also shown with the absorbance of carbonyl or sulfoxide separately. Due to the following of a similar trend, these figures were not shown here.



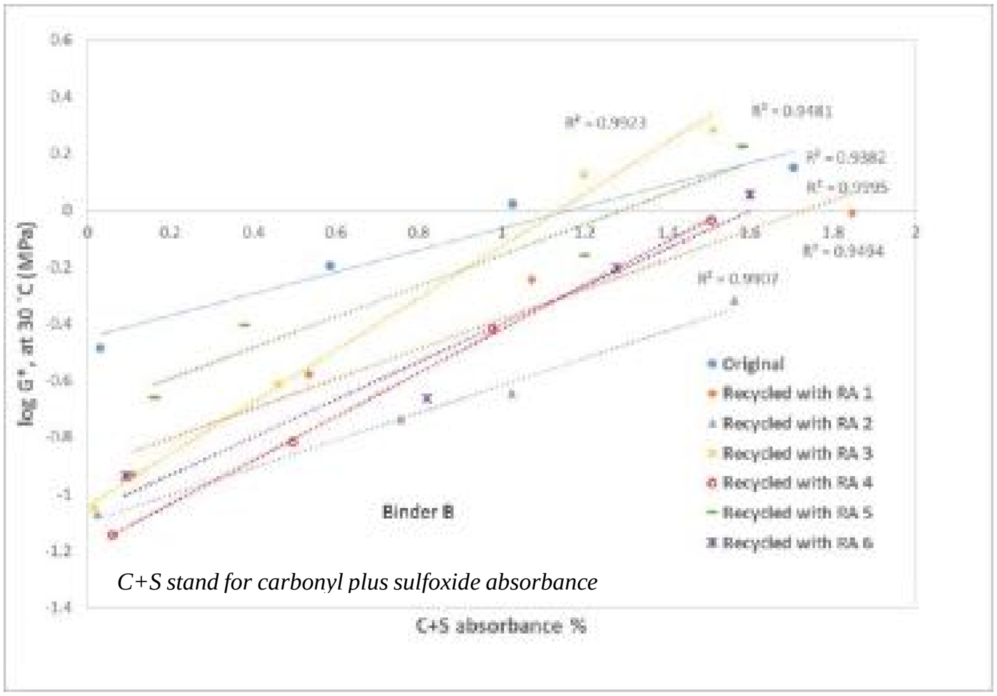
C+S stand for carbonyl plus sulfoxide absorbance

(a)

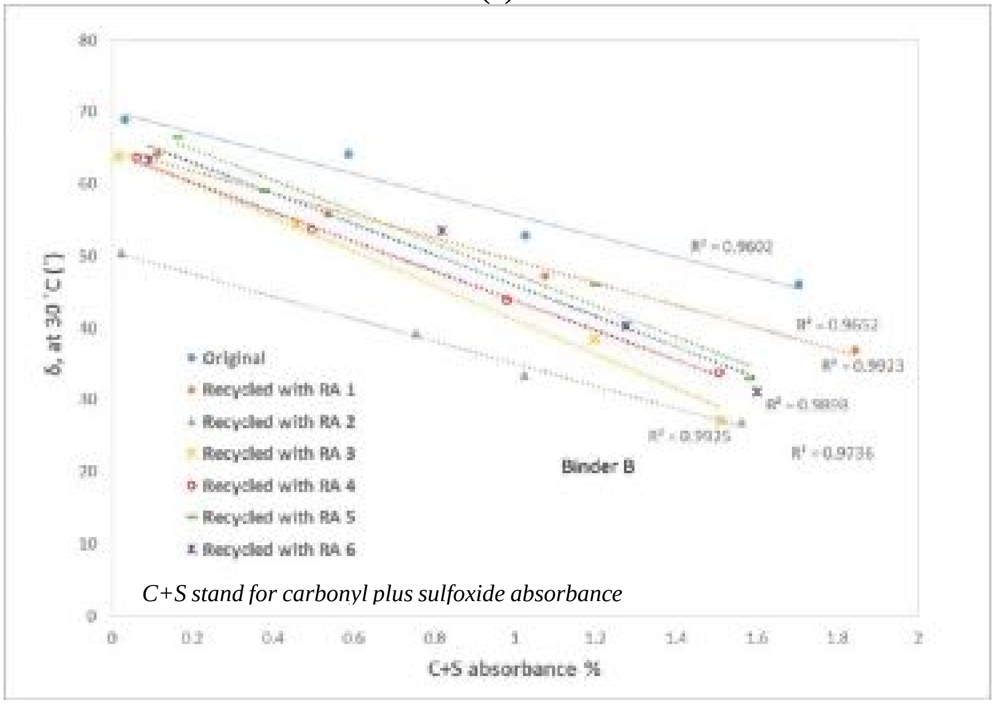
C+S stand for carbonyl plus sulfoxide absorbance

(b)

Figure 3-9: Correlation between chemical and rheological indexes measured at 30 ˚C. Correlation between (a) C+S absorbance and log G, (b) C+S absorbance and δ (˚) of recycled binder A with different RAs*



(a)



(b)

Figure 3-10: Correlation between chemical and rheological indexes measured at 30 ˚C. Correlation between (a) C+S absorbance and log G, (b) C+S absorbance and δ (˚) of recycled binder B with different RAs*

3.4.7 Softening Curve According to the Chemical Indexes

In Section 3.6.2, the softening curves were established to identify the amount of RA that is needed to recycle the aged binder according to the changes in the hot temperature PG of asphalt binder. Also, it was shown in previous sections that changes in the chemical index of asphalt binders have a strong correlation with the rheological properties of asphalt binders. Accordingly, in this section, the changes of chemical indexes of recycled asphalt with different amounts of RA are investigated to establish a softening curve according to the chemical indexes. Figure 3-11 shows the changes in carbonyl plus sulfoxide (C+S) absorption in recycled binders A and B when the amount of RA2 and RA3 is increased in the RAP binder. This figure indicates a strong correlation between the chemical indexes of recycled binders with an increase in RA content in the RAP binder. The concentration of chemical indexes decreased with the increase of RA. Therefore, it can be concluded that it is possible to establish a softening curve based on changes in the chemical indexes of asphalt binders, and the amount of RA in the recycling of aged binders is identified based on chemical indexes. Using this method is much more economical than using the PG of binders in constructing the softening curves.

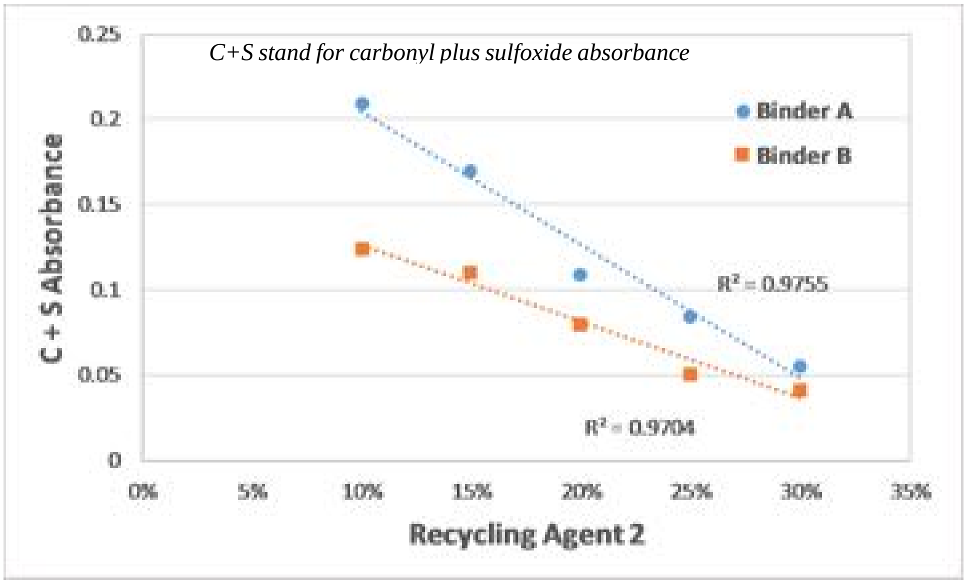


Figure 3-11: Changes in carbonyl plus sulfoxide absorbance in the unaged binder of A and B with adding of different percentages of RA2

3.5 Discussion

Although two different virgin binders from two different crude oil sources were selected for use in this study, the binders' low-temperature, rutting, and fatigue performance were approximately the same. The differences between the two binders' rheological performances varied enough to compare them and select the binder with better performance. Binder B's resistance to aging and its fatigue performance were better than binder A. Also, the virgin binders' chemical indexes (C, S or C+S peaks) were a distinctive representation of binder age and had a strong correlation with the rheological properties of binders, which is consistent with previous studies (Elwardany et al. 2017). More specifically, the lower levels of carbonyl and sulfoxide in binder B compared to binder A were proper indicators for the selection of a virgin binder with better initial and long-term performance.

In previous studies, the amount of RA used to recycle the aged binders was generally selected without creating a softening curve (Mohammadafzali et al. 2015). This procedure resulted in recycled binders with different initial rheological properties. However, in this study, the softening curve was established to recycle binders with the same initial high-temperature grade to make the comparison between the recycled binders' and virgin binders' properties easier. Also, the lower level of change between the unaged and 2 PAV-aged high PG of binder B compared to binder A resulted in a smaller amount of RA required to soften the aged binder B.

As studied by other researchers, the rutting performance of recycled asphalt using RA improved compared to the virgin binder (McGovern et al. 2018). This improvement was also observed for recycled binders from the aged binder B. However, this rutting performance improvement in recycled binders was found in recycled aged binder A with RA2, RA3, and RA6. This can be caused by the different properties of virgin binders A and B. It should be noted that most of the previous studies were conducted with a prespecified amount of RA in the recycling of aged binders. In this case, the rutting performance of recycled binders was improved because the initial properties of recycled binders and virgin binders were not comparable. This means that the recycled binders and virgin binders had a different high PG at the unaged step.

The fatigue performance (which is $G^*.\sin\delta$) of all of the recycled binders was better than that of the virgin binders. As described, the fatigue performance of binders was used to predict the service life of binders and calculate their durability index. This indicates that recycled binders have a better service life than virgin binders. The durability index of recycled binders with RA2 and RA3 was better than other recycled binders and significantly increased the service life of pavement. Similarly, the low-temperature performance of all recycled binders was better than that of the virgin binders. A study conducted on the BBR results showed that the creep stiffness (S) of binders is the main factor that controls thermal stress development (Marasteanu 2004). This study showed that the recycled binders had a stiffness level that was significantly lower than that of the virgin binders. These results demonstrate that the level of thermal stress that developed in the recycled binders due to cold weather is considerably lower than in the virgin binders.

When rutting performance is a concern, the binder should be stiff and elastic. In this case, G* should be high enough to provide a stiff binder, and δ should be low enough to increase the elastic portion of G*. By considering the rheological properties of the binder measured at 74 ˚C, all recycled binders had lower δ compared to the virgin binder. However, only recycled binders with RA2 and RA3 had a higher G* than the virgin binders. This trend indicates that only recycled binders with RA2 and RA3 are stiffer and more elastic than the virgin binders in their early age when rutting is a concern. When the long-term and fatigue performance of a binder is a concern, the binder should be elastic and not too stiff. Therefore, the G* and δ should be low. By considering the rheological properties of the binder measured at 30 ˚C, all recycled binders had a better long-term performance than the virgin binders. In summary, by considering the rutting, fatigue, and low-temperature cracking performance of binders, it can be concluded that the recycled binders with RA2 and RA3 had better performance than the virgin binders.

The chemical indexes measured in virgin and recycled binders throughout the different aging steps defined the increase of carbonyl and sulfoxide (Table 3-9). This trend is in line with previous studies. However, the rate of these parameter changes for different binders varied. This rate showed that the virgin and recycled binders with a lower amount of carbonyl and sulfoxide at an unaged step had better short-term and long-term performance. As shown, the rate of chemical indexes changes with aging for the recycled binders using RA2, and RA3 is less than that of the virgin binders and had an initial lower concentration at the unaged step. This trend and conclusion can be used to select the best RA for the recycling of RAP. Remarkably, the cost of measuring the chemical indexes of binders is significantly cheaper than defining the rheological properties of binders. As there are different RAs with different unknown properties, this result can be applied to categorize the effect of RAs on the short- and long-term performance of recycled RAP.

Table 3-9: Rheological properties of recycled binders based on chemical indices

RAP binder	Temperature	RA3	RA2
A	74 ˚C	Log G* = 0.75 - 0.14 (C+S)	Log G* = 0.64 - 0.07 (C+S)
		δ = -14.89 – 85.4 (C+S)	δ = -22.9 – 78.01 (C+S)
	30 ˚C	Log G* = 0.76 - 1.02 (C+S)	Log G* = 0.52 - 1.2 (C+S)
		δ = -19.61 + 65.05 (C+S)	δ = -14.27 + 49.19 (C+S)
B	74 ˚C	Log G* = 0.82 - 0.17 (C+S)	Log G* = 0.77 - 0.15 (C+S)
		δ = -19.34 + 83.93 (C+S)	δ = -20.31 + 79.54 (C+S)
	30 ˚C	Log G* = 0.92 - 1.04 (C+S)	Log G* = 0.48 - 1.1 (C+S)
		δ = -23.8 + 64.9 (C+S)	δ = -15.6 + 50.65 (C+S)

To facilitate the use of chemical indexes in the recycling of asphalt binder, the possibility of using chemical indexes to establish the softening curves was investigated. The results showed that it is possible to use chemical indexes to select the proper amount of RA based on the chemical indexes of recycled binders. This result can reduce the use of rheological tests in the recycling process of asphalt pavement.

3.6 Development Method in Defining RA Dosage

The results of this research show that the performance of recycled binders is highly dependent on the type of RA. Based on this research, RA2 and RA3 are among the proper RAs available on the market. In order to define the required amount of RA% in the recycling of aged binders, softening curves were constructed based on the high-temperature PG value of binders. However, the relationship between the percentage of RA and concentration of carbonyl plus sulfoxide in recycled binders indicated that it is possible to define the RA percentage based on chemical indices (Figure 3-11). Also, the correlation between chemical indices and rheological properties of binders demonstrated that it is possible to predict recycled binder rheological properties based on chemical indices. Based on these results, a procedure was developed to define RA% in the recycling of pavement, predict pavement performance, and adjust the dosage of required RA% based on asphalt chemical properties (Figure 3-12). A comparison between conducting FTIR tests and DSR tests showed that the FTIR tests yielded a 60% savings in time and costs.

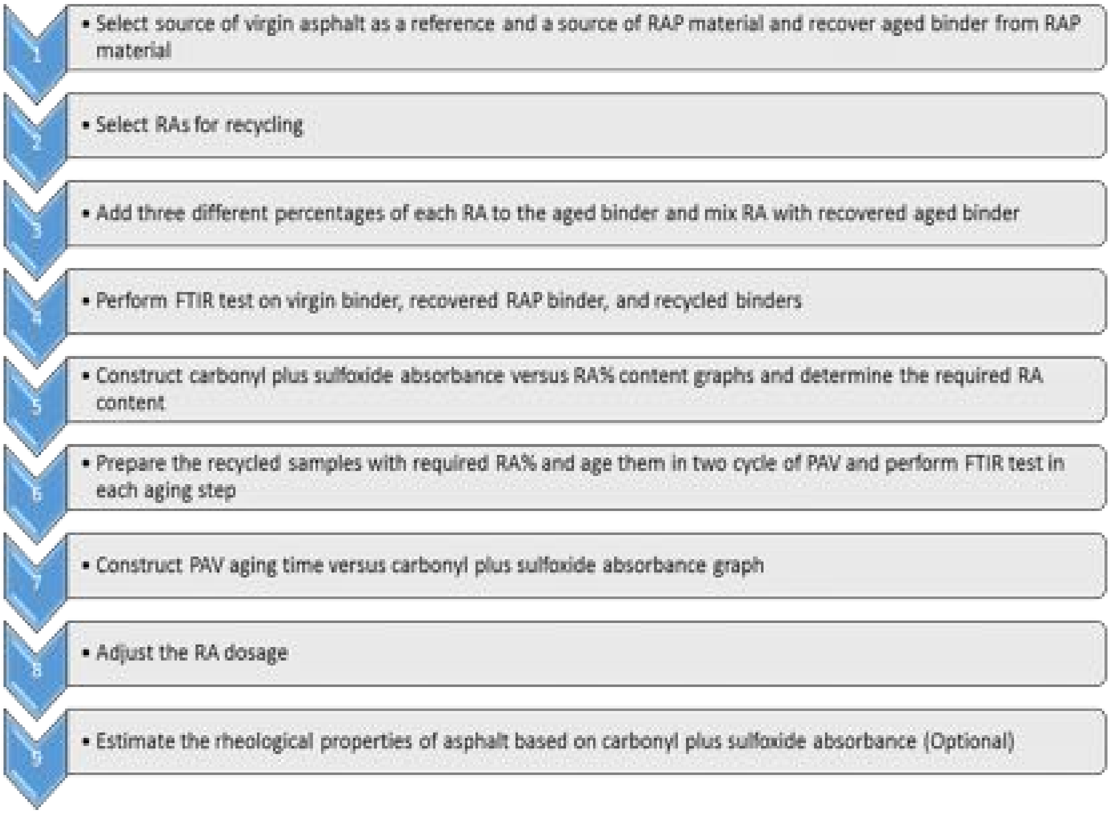


Figure 3-12: Procedure to define the RA percentage in RAP recycling

1- A source of virgin asphalt as a reference and a source of RAP material should be selected. The aged binder from RAP material should be extracted.

2- Select several RAs to rejuvenate RAP material.

3- Three different percentages of each RA should be added to the aged binder. For example, consider two different sources of RAs. Therefore, six samples with different percentages of RA (for instance, 10%, 15%, and 20%) should be prepared (Table 3-10).

Table 3-10: Required samples to select the proper RA

Recycling Agent	RA Percentage		
	10%	15%	20%
RA2	Sample RA2-1	Sample RA2-2	Sample RA2-3
RA3	Sample RA3-1	Sample RA3-2	Sample RA3-3

4- Conduct the FTIR test on the virgin binder, RAP binder, and recycled binders with three different percentages of RA. By performing this test, the carbonyl plus sulfoxide absorbance of samples will be determined (Table 3-11 and Figure 3-13).

Table 3-11: Chemical indices of virgin and RAP binder

Binder	Aging condition	C+S absorbance%
Virgin binder	Unaged	0.062
RAP binder	Aged	2.06

5- The carbonyl plus sulfoxide absorbance versus RA% graph for the recycled binders should be constructed (Figure 3-13).

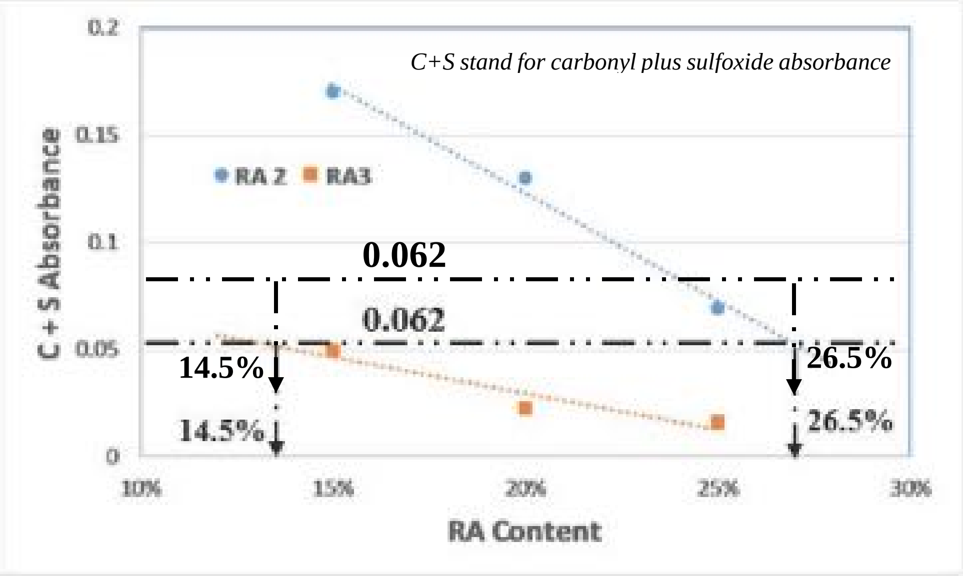


Figure 3-13: Defining the proper amount of RA in the recycling process

To define the required RA% in the recycling process, a horizontal line in Figure 3-13 should be drawn from 0.062 % absorbance (carbonyl plus sulfoxide of virgin binder). Then, the required amount of RA can be extrapolated (Table 3-12).

Table 3-12: Required amount of RA

Recycling agent	Required RA content (%)
RA 2	26.5
RA 3	14.5

6- The recycled binders using the required amount of RA% (Table 3-12) should be prepared. Next, the prepared samples should be aged in RTFO and two cycles of PAV. Then, the FTIR test on the samples aged in RTFO-, 1 PAV-, and 2 PAV-aged should be conducted to measure their carbonyl plus sulfoxide in each aging step.

7- Based on the conducted FTIR test on the samples, the carbonyl plus sulfoxide absorbance versus PAV aging time graph should be constructed (Figure 3-14). It should be noted that each PAV aging cycle is exposure of binder to pressure and heat for 20 hours.

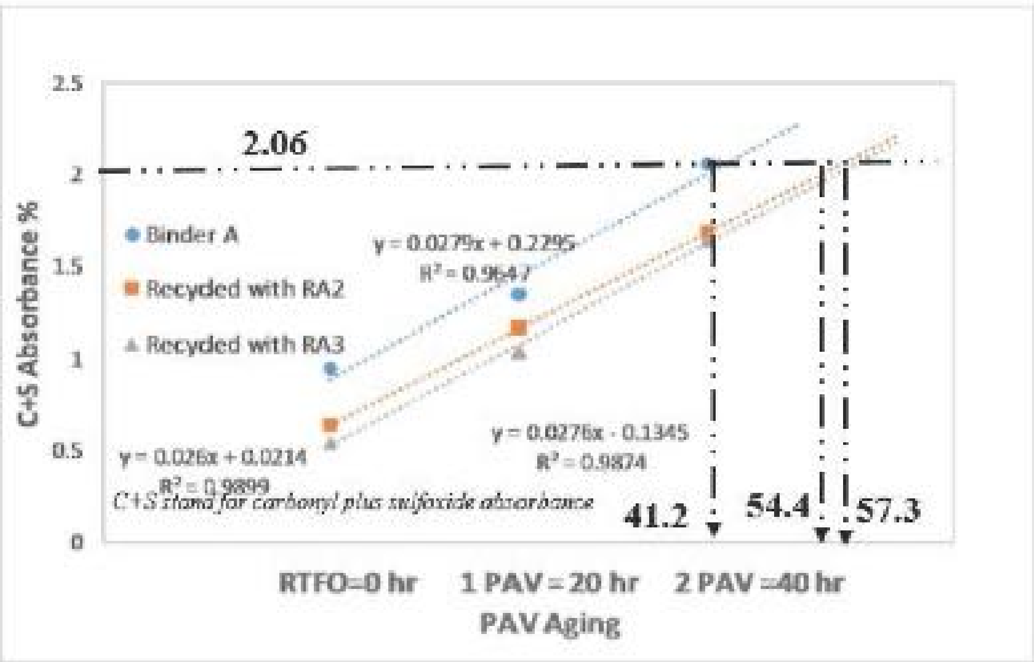


Figure 3-14: Carbonyl plus sulfoxide verses PAV aging time

8- In previous studies, it was shown that each PAV cycle is equal to a 10-year in-service life of asphalt pavement. If the design life pavement is considered to be 20 years, the aging of binders in two cycles of PAV is required to simulate in-service aging (Bahia and Anderson 1995). Therefore, the carbonyl plus sulfoxide concentration of the aged virgin binder after 2 PAV cycles of aging can be considered a failure point of asphalt. In this study, the aged virgin binder A with 2 cycles of PAV aging had a 2.06% concentration of carbonyl plus sulfoxide absorbance. By considering a 2.06% absorbance a failure point, it is possible to calculate the required PAV time to recycle binders to reach the failure point using the equations in Figure 3-14 (Table 2-14). As shown in Figure 3-14 and previous sections, the recycled binders had a lower aging rate in comparison to the virgin binders. Accordingly, the dosage of RA% required for the recycling process can be adjusted so that a recycled binder will have the same aging rate and service life as a virgin binder.

As shown in Table 3-13, the PAV exposure time that binders need to reach to the failure point (2.06% absorbance) was calculated using the equations in Figure 3-14. Also, the aging rate of recycled binders was calculated using Equation 3-1. Then, the adjusted required RA was calculated using Equation 3-2 (Table 3-14).

Table 3-13: Aging rate of binders

Binders	PAV exposure time (hours)	Aging rate
Virgin binder	41.2	-
Recycled with RA2	54.4	1.32
Recycled with RA3	57.3	1.39

$$Aging\ rate = \frac{\text{Recycled binder PAV exposure time}}{\text{Virgin binder PAV exposure time}} \qquad \text{Equation 3-1}$$

Table 3-14: Adjusted required amount of RA

Recycling agent	Required RA content (%)	Adjusted RA content (%)
RA 2	26.5	21
RA 3	14.5	11

$$Asjusted\ required\ RA = \frac{\text{Required RA content}}{\text{aging rate}} \qquad \text{Equation 3-2}$$

9- If prediction of the recycled binder performance is needed, the DSR test on the RTFO-aged and 2 PAV-aged binder is also required. By conducting the DSR test, the G* and δ of binders can be measured. Then, it is possible estimate the rheological properties of asphalt binder by constructing carbonyl plus sulfoxide versus logG* or δ graph (Figure 3-10). The equations that can be used to calculate the rheological properties of a recycled binder with RA2 and RA3 are listed in Table 3-15. Using the calculated rheological properties, it is possible to indicate the fatigue and rutting performance of asphalt.

Table 3-15: Rheological properties of binders based on chemical indices

Temperature	RA3	RA2
74 ˚C	Log G* = 0.75 - 0.14 (C+S)	Log G* = 0.64 - 0.07 (C+S)
	δ = -14.89 – 85.4 (C+S)	δ = -22.9 – 78.01 (C+S)
30 ˚C	Log G* = 0.76 - 1.02 (C+S)	Log G* = 0.52 - 1.2 (C+S)
	δ = -19.61 + 65.05 (C+S)	δ = -14.27 + 49.19 (C+S)

4 CHAPTER 4: RECYCLING OF RAP USING NANOMODIFIED ASPHALT

4.1 Introduction

Due to the special characteristics of nanoparticles, pavement engineers have used nanoparticles as an additive in asphalt pavement to improve pavement performance. This study focuses on characterizing modified asphalt performance using nanoclay particles compared to the unmodified asphalt binder. For this evaluation, 3% of nanoclay particles were added to a neat binder to prepare the nanomodified binder. Then, the modified and unmodified binders underwent a short- and long-term aging process using the Rolling Thin Film Oven (RTFO) and two cycles of Pressure Aging Vessel (PAV). Finally, the Dynamic Shear Rheometer (DSR), Bending Beam Rheometer (BBR), and Fourier-Transform Infrared Spectroscopy - Attenuated Total Reflection (FTIR-ATR) tests were carried out in each aging step to assess and monitor the rutting, fatigue, low-temperature performance, durability, and chemical changes of binders during the aging process. The results of this study proved the enhanced properties and performance of the nanomodified binder compared to the unmodified binder. These results can pave the path with the use of nanomodified binders in the recycling of aged pavement and construction of roads that experience poor weather conditions and high traffic loads.

Furthermore, the use of the nanomodified asphalt binder in the recycling of RAP material to increase the incorporation of RAP percentage in recycled asphalt compared to using neat (unmodified) asphalt binder was investigated. For this evaluation, a nanoclay modified and unmodified binder were used. The RAP (aged) binder was prepared by putting the unmodified binder in the Rolling Thin Film Oven (RTFO) and through two cycles of

Pressure Aging Vessel (PAV). Then, the rutting, fatigue, and low-temperature performance of the recycled binders with different percentages of RAP during the aging of the binder were evaluated. The results demonstrated that the maximum use of a 20% RAP binder (33% RAP material) was allowed in the recycled pavement using a nanomodified binder, while the maximum use of a 10% RAP binder (17 % RAP material) was allowed with the unmodified binder. This increase in the incorporation of RAP percentage in recycled asphalt pavement can pave the path to expand the use of the nanomodified binder in the recycling of asphalt pavement and its usage in severe weather conditions.

4.2 Objectives

The objective of this study was to use nanoclay particles as an additive to the neat asphalt binder and assess the short- and long-term performance of the produced nanomodified binder. In this evaluation, the rheological and chemical characteristics of nanomodified and neat asphalt binders were tested and compared. Moreover, the relationship between the chemical indexes and rheological properties of the nanomodified asphalt binder was investigated. The results can pave the path in using nanomodified asphalt in the asphalt pavement industry, especially in the recycling of asphalt pavement due to the unique properties of nanomodified asphalt. Also, it is possible to identify the age of pavement and predict its performance by selecting the proper treatment.

Another objective of this study was to investigate the use of a nanomodified binder in the recycling of an aged binder. The different percentages of aged asphalt (RAP binder) were mixed with a nanomodified and unmodified binder to compare the performance of the recycled asphalt binder. In determining the asphalt binder’s performance properties, the fatigue, rutting, and thermal cracking resistance of asphalt pavement were evaluated. Also, the Fourier Transform Infrared Spectroscopy (FTIR) test was conducted to monitor the changes in the chemical functionality of asphalt binders. It should be noted that the nanomodified binder was prepared using 3% of nanoclay particles.

4.3 Materials

One neat asphalt binder with the PG 67-22 grade that is commonly used in Florida was selected. It was expected that the high-temperature PG of virgin (original) binder was higher than 67 ˚C (AASHTO M320) (AASHTO M320 2017). The critical low temperature of the binder was also expected to have an m-value of more than 0.3 and a creep stiffness (S) of less than 300 MPa at -12 ˚C and 60 seconds (AASHTO M320). Also, the medium temperature grade of the asphalt binder was expected to meet the AASHTO M320 standard criteria ($G^*.\sin\delta<5$ MPa at 26.5 ˚C).

One type of nanoparticle was also selected. The Bentonite Nanoclay manufactured by Sigma-Aldrich was used as an additive to modify the neat asphalt binder properties. The 3% of nanoclay by weight of the binder was mixed with the neat asphalt binder. This type of nanomaterial and the percentage of nanoparticles were selected because of the previous studies that used it with different binders, which resulted in the enhancement of pavement properties and performance (Li et al. 2017).

The nanoparticles and neat binder were mixed using the dry blending method to prepare the nanomodified binder. To prepare the nanomodified binder, the neat binder was heated to 160 ±5 ˚C, and then the nanoparticles were added gradually to the binder and finally mixed at a speed of 4000 rpm for 45 minutes using a high shear blender. It should be noted that the temperature of the binder kept constant during the mixing time. The blending procedure, including blending duration and speed, was selected according to previous studies to prepare a homogenous nanomodified binder (Polacco et al. 2008, Zare-Shahabadi et al. 2010, You et al. 2011, Ghasemi et al. 2012).

The recycling process of the asphalt binder included heating the aged, nanomodified, and unmodified asphalt binders to 155±5 ˚C, and then adding the desired amount of aged binder to the modified or unmodified binders and mixing them for two minutes. The soft binders and aged binders were mixed using a spatula. The 10%, 20%, and 30% aged asphalt binders were added to the soft binders.

4.4 Comparison of Nanomodified Binder with Unmodified Binder

4.4.1 High-temperature performance (Rutting Performance)

The high-temperature grade (High PG) of modified and unmodified asphalt binders was calculated by conducting DSR tests at high temperatures. The results are summarized in Table 4-1. As shown, with the aging of asphalt, the phase angle of binders decreased and G* increased. Also, the nanomodified binder had a higher high PG grade compared to the unmodified binder. This higher PG was caused by a higher G* and lower δ compared to the unmodified binder. As the rutting performance of asphalt is a concern at high temperatures, the rutting performance of the nanomodified binder was slightly better than the unmodified binder, which had a higher G*/sin δ value.

Table 4-1: High PG grade of nanomodified and unmodified binders in different steps of aging

Aging Phase	Unmodified Binder				Nanomodified Binder			
	Test Temperature (˚C)	δ (˚)	G*/sin δ (kPa)	High Temperature Grade PG (℃)	Test Temperature (˚C)	δ (˚)	G*/sin δ (kPa)	High Temperature Grade PG (℃)
Unaged	70	84	2.20	74.54	70	82	2.34	75.59
	76	87	0.78		76	84	0.94	
RTFO-aged	74	82	2.84	75.99	74	81	3.12	76.94
	80	85	1.32		80	84	1.53	
1 PAV-aged	82	81	2.51	83.19	74	76	6.60	82.91
	88	84	1.30		80	79	3.15	
2 PAV-aged	82	75	7.11	92.05	80	69	9.80	96.02
	88	78	3.53		86	74	5.60	

4.4.2 Mid-temperature performance

The DSR tests at medium temperatures were conducted to evaluate the long-term performance of asphalt binders. The tests results are summarized in Table 4-2. As shown, with the aging of binders, the δ decreased and G* were increased. Also, both modified and unmodified binders met the fatigue performance criteria (G*.sinδ<5 MPa at 26.5 ˚C). The modified binder had lower δ and higher G* compared to the unmodified binder, resulting in a lower mid-PG for the unmodified binder. This lower PG for the nanomodified binder demonstrated that the nanomodified binder had better fatigue performance. Also, by considering the mid-PG of the binders at 1 PAV and 2 PAV and 26.5 ˚C as the PAV failure point according to AASHTO T315, it was possible to determine the PAV time that each binder took to reach the failure point. Accordingly, the PAV failure time of each binder was calculated and summarized in Table 4-3. As shown, the failure time in hours of the nanomodified binder was greater than the unmodified binder. This improvement indicates an 8% enhancement in the durability and service life of the pavement.

Table 4-2: Mid-PG grade of nanomodified and unmodified binders in different steps of aging

Aging Phase	Unmodified Binder				Nanomodified Binder			
	Test Temperature (˚C)	δ (˚)	G*sin δ (MPa)	Mid temperature PG (℃)	Test Temperature (˚C)	δ (˚)	G*sin δ (MPa)	Mid temperature PG (℃)
unaged	24	63	1.44	16.06	24	60	1.44	14.79
	30	68	0.56		30	65	0.64	
RTFO-aged	24	52	2.78	17.70	24	48	2.79	16.85
	30	57	1.59		30	54	1.71	
1 PAV-aged	24	44	3.92	21.24	24	41	4.01	20.46
	30	49	2.30		30	46	2.76	
2 PAV-aged	24	41	5.70	25.36	24	36	5.28	24.63
	30	45	3.20		30	41	3.13	

Table 4-3: Durability and PAV failure time of recycled binders

Durability	Unmodified Binder	Nanomodified Binder
PAV failure time (hr.)	46.5	50.2

4.4.3 Low-Temperature Performance

The low-temperature cracking resistance of the asphalt binder was measured and is displayed in Table 4-4. As shown, by the increase of temperature, the creep stiffness of the binder increased, and the m-value decreased. Also, both binders met the cracking resistant criteria (S<300 MPa, m>0.3 at -12 ˚C and 60 seconds) based on AASHTO T31. The critical low-temperature grade of the binder was calculated using creep stiffness and the m-value. However, the critical temperatures calculated from the m-value were more critical than those calculated from creep stiffness because they resulted in a higher low-temperature grade. More importantly, the calculated critical low-temperature grade of the modified binder was lower than the unmodified binder. This indicates the improvement in the low-temperature performance of the modified binder compared to the unmodified binder. Also, the decrease in creep stiffness and increase in the m-value of the modified binder compared to the unmodified binder was significant.

Table 4-4: BBR test results at 60 seconds

Binder	**Aging Step**	**Temperature (° C)**	**Stiffness (Mpa)**	**m-value**	**Critical Temperature (Stiffness)**	**Critical Temperature (m-value)**
Unmodified	1 PAV Cycle	-12	185	0.329	-27.84	-25.48
		-18	304	0.279		
	2 PAV cycle	-12	267	0.311	-23.79	-22.66
		-18	395	0.211		
Modified	1 PAV Cycle	-12	179	0.362	-28.20	-27.31
		-18	295	0.292		
	2 PAV cycle	-12	241	0.333	-25.39	-24.18
		-18	355	0.242		

4.4.4 Chemical Indexes of Binders

Figure 4-1 shows the changes in the sulfoxide and carbonyl indexes of binders with aging that were measured using the FTIR test. As shown, the sulfoxide and carbonyl indexes increased with the aging of the binders. Also, at the unaged step, the concentration of sulfoxide and carbonyl for the modified binder was higher than the unmodified binder. However, in RTFO-, 1 PAV- and 2 PAV-aged steps, the concentration of sulfoxide and carbonyl for the modified binder was lower than the unmodified binder.

In Figure 4-2, the correlation between carbonyl plus sulfoxide and measured log G* and phase angle (δ) at 74 ˚C of the binders is displayed. As shown, there was a strong positive logarithmic correlation between the carbonyl plus sulfoxide chemical functional groups and complex modulus (G*) with the aging of the modified and unmodified binders. Conversely, there was a strong negative correlation between the carbonyl plus sulfoxide chemical functional groups and phase angle (δ) with the aging of the modified and unmodified binders. Also, the phase angle (δ) of the modified binder was lower than the unmodified binder. Conversely, the complex shear modulus (G*) of the nanomodified binder was higher than the unmodified binder. The same trends were observed for the measured rheological properties of asphalt at 30 ˚C (Figure 4-3).

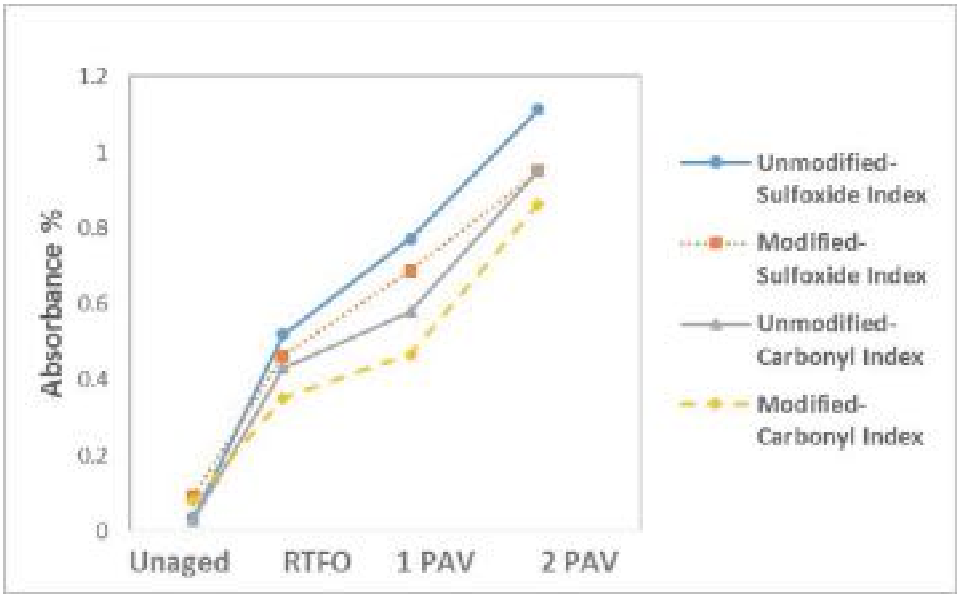
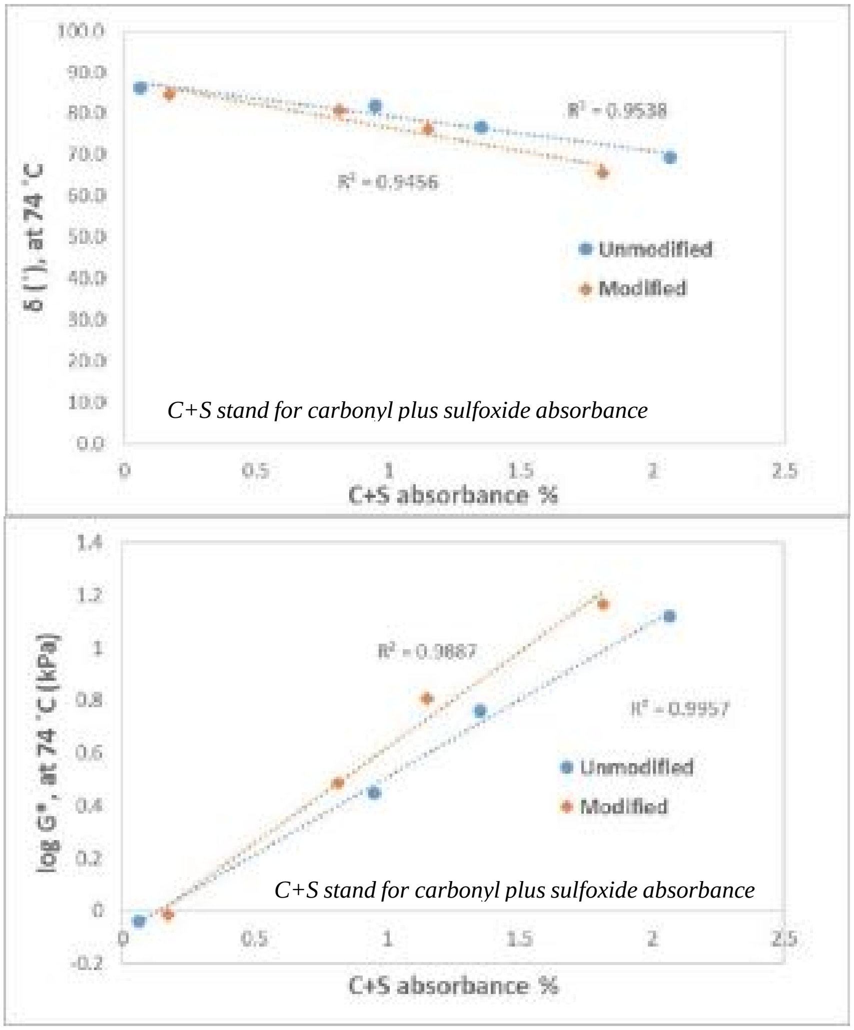


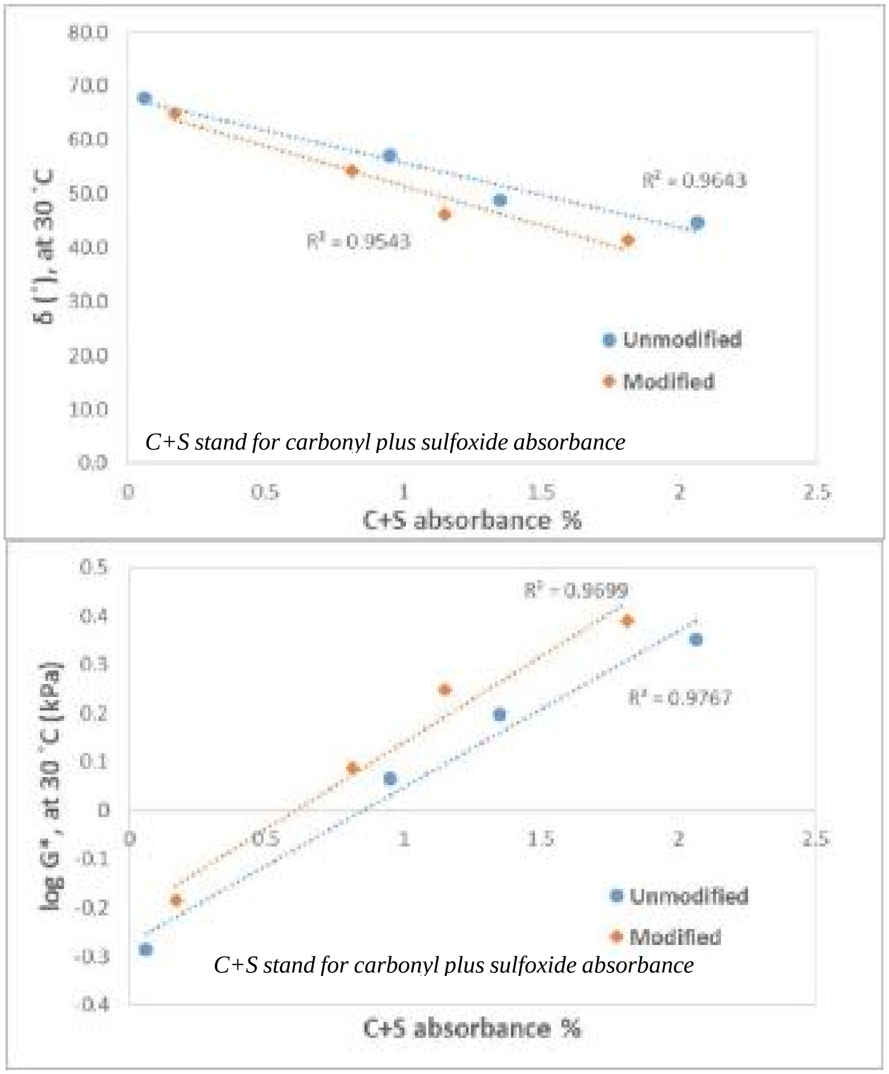
Figure 4-1: The change in carbonyl and sulfoxide indexes with aging



C+S stand for carbonyl plus sulfoxide absorbance

C+S stand for carbonyl plus sulfoxide absorbance

Figure 4-2: Correlation between chemical indexes and measured rheological properties of binders at 74 ˚C



C+S stand for carbonyl plus sulfoxide absorbance

C+S stand for carbonyl plus sulfoxide absorbance

Figure 4-3: Correlation between chemical indexes and measured rheological properties of binders at 30 ˚C

According to the strong correlation between the chemical indexes and rheological properties of unmodified and modified binders (Figures 4-2 and 4-3), models were developed to predict the rheological properties of binders according to their chemical indexes. These models predict complex shear modulus (G*) and the phase angle of asphalt in two temperatures of 30 ˚C and 74 ˚C, as shown in Table 4-5. In these models, C+S is the carbonyl plus sulfoxide of the asphalt binder at any age of asphalt. According to these models, the rheological properties of the asphalt binder can be defined based on the chemical index of asphalt. It should be noted that measuring the chemical indexes of asphalt are not temperature-related, and by defining the chemical index of the asphalt binder, it is possible to identify the rheological properties of asphalt in different temperatures.

Table 4-5: Prediction of rheological properties of unmodified and modified binders according to the chemical indexes of binders during their service life

Temperature	Unmodified Binder	Nanomodified Binder
74 ˚C	Log G* = -0.0766 + 0.5866 (C+S)	Log G* = -0.1088 + 0.73 (C+S)
	δ = 88 - 8.51 (C+S)	δ = 88.18 - 11.546 (C+S)
30 ˚C	Log G* = -0.2731 + 0.3214 (C+S)	Log G* = -0.216 + 0.3558 (C+S)
	δ = 67.81 – 11.975 (C+S)	δ = 66.27 – 14.804 (C+S)

4.4.5 Discussion

The incorporation of nanoparticles in asphalt pavement as an additive has been introduced as a method to modify the properties of asphalt pavement and address the increase of traffic load in roads. This study performed comprehensive experimental tests and literature reviews to evaluate the nanomodified asphalt pavement performance. One essential issue was finding and selecting the best method to mix nanoparticles with the asphalt binder so that it produced a homogenous binder. The dry blending method was used, and the mixing time duration and mixing speed were selected based on previous studies. However, different studies used different times and speeds. Therefore, a study that provides a standard to specify the mixing time duration and speed is deemed necessary.

The incorporation of nanoparticles showed improvement in the rutting and high-temperature performance of the nanomodified binder. This improvement was not significant. However, previous studies showed a significant improvement. The reason for this improvement is because the researchers used different percentages of nanoparticles and binders with different PG grades. Another reason to consider is that rutting performance improvement is not only due to the incorporation of nanoparticles; it can also be caused by the high sensitivity of the binder to heat. The asphalt binder, which is heated during the mixing of the asphalt binder with nanoparticles, can increase the shear modulus of asphalt.

The significant impact of nanoparticles in the improvement of pavement performance was shown in the fatigue and long-term performance of the nanomodified binder. Although the binder was heated during the mixing of nanoparticles with a binder that could produce a negative effect on the fatigue performance of asphalt, the nanoparticles significantly improved the fatigue performance of the asphalt pavement. By considering this improvement, which can increase the service life of pavement by 8%, along with the low cost of nanoclay particles, it can be concluded that the use of nanomodified asphalt is economically beneficial. Also, the decrease of phase angle in the nanomodified asphalt in comparison to the unmodified binder indicated the enhancement of the elastic behavior of asphalt and ability of nanomodified asphalt in recovering the deformation.

The low-temperature performance of the nanomodified binder improved compared to the unmodified asphalt. This improvement was due to the decrease in creep stiffness and increase of the m-value. These changes are ideal because the decrease in creep stiffness is an indicator of improvement in thermal cracking resistance. Also, the increase in the m-value shows improvement in the relief of thermal stress.

The FTIR test showed that the carbonyl and sulfoxide functional groups are good indicators of asphalt age for nanomodified and unmodified asphalt. Also, it showed that nanoparticles have an anti-aging impact in the asphalt pavement because the produced sulfoxide and carbonyl in the RTFO-, 1 PAV-, and 2 PAV-aged steps of the nanomodified binder were lower than the unmodified binder. However, the concentration of sulfoxide and nanoparticles in the unaged step in the nanomodified binder were equal to or more than that of the unmodified binder. This higher concentration can be due to the increase in heat during the mixing of the binder with nanoparticles.

The high correlation between the chemical indexes and rheological properties of the asphalt binder that were measured in high and medium temperatures demonstrated that chemical indexes are good representatives of pavement rutting and fatigue performance. Also, the lower value of the phase angle of the nanomodified asphalt indicated the better elastic behavior of nanomodified asphalt compared to unmodified asphalt. Conversely, the higher complex modulus of the nanomodified asphalt compared to the unmodified binder indicated the stiff behavior of asphalt. This behavior is ideal when the rutting is a concern and asphalt should not deform too much. This means that incorporation of nanoparticles can simultaneously improve the fatigue and rutting performance of asphalt.

Finally, the models were developed to predict the rheological properties of asphalt binders according to the chemical index of the asphalt binder. In this case, it is possible to predict the rheological properties of asphalt in different temperatures, which are representative of the rutting and fatigue performance of asphalt. This indicates that it is possible to use the simple FTIR test to predict the performance of asphalt at different ages.

4.5 Results of Nanomodified Recycled Asphalt

4.5.1 High-Temperature Performance

The DSR test was conducted at high temperatures to measure the rutting performance of binders. Results are summarized in Table 4-6. In this table, the high-temperature PG of binders was calculated using Equation 2-1. As shown, with the increase of RAP binder percentages in recycled binders with nanomodified and unmodified binders, the complex shear modulus (G*) and high PG of asphalt binders were increased. However, the phase angle (δ) of binders decreased slightly with an increase in RAP binder incorporation in the recycled binders. These results indicated that the addition of a RAP binder percentage in the recycled binders could improve the rutting performance of recycled binders. Furthermore, by comparing the recycled nanomodified and unmodified binders, it can be demonstrated that the recycled binders with nanomodified asphalt had better rutting performance due to their higher high-temperature PG. The higher temperature PG of recycled nanomodified binders compared to recycled unmodified binders was caused by an increase in G* and a decrease in δ in the nanomodified binder. It should be noted that the rutting performance of asphalt is a concern in the short-term (unaged- and RFTO-aged)

aged binder. In this case, the high-temperature grade of asphalt binders was measured only in unaged-, RTFO- and 1 PAV-aged binders.

Table 4-6: High-temperature PG of recycled binders

RAP Binder	Aging Step	Unmodified Binder				Modified Binder			
		Test Temperature (˚C)	δ (˚)	G*/sin δ (kPa)	High Temperature PG (℃)	Test Temperature (˚C)	δ (˚)	G*/sin δ (kPa)	High Temperature PG (℃)
0%	Unaged	70	84	2.20	74.54	70	82	2.34	75.59
		76	87	0.78		76	84	0.94	
	RTFO-aged	74	82	2.84	75.99	74	81	3.12	76.94
		80	85	1.32		80	84	1.53	
	1 PAV-aged	82	81	2.51	83.19	74	76	6.60	82.91
		88	84	1.30		80	79	3.15	
10%	Unaged	68	84	2.48	76.75	68	83	2.58	78.36
		74	86	1.33		74	84	1.49	
	RTFO-aged	74	81	3.98	78.61	74	79	4.17	79.59
		80	83	1.84		80	82	2.10	
	1 PAV-aged	74	75	12.08	87.80	74	71	13.83	90.90
		80	79	5.76		80	74	7.20	
20%	Unaged	68	83	2.77	77.97	68	82	2.34	79.82
		74	85	1.50		74	84	1.52	
	RTFO-aged	74	79	4.47	79.89	74	79	4.79	80.88
		80	82	2.17		80	82	2.43	
	1 PAV-aged	74	69	16.32	90.60	74	64	20.50	94.02
		80	74	7.91		80	68	10.50	
30%	Unaged	68	82	3.39	78.17	68	81	3.93	79.98
		74	84	1.65		74	84	1.98	
	RTFO -aged	74	77	7.36	83.21	74	76	8.86	86.22
		80	80	3.35		80	79	4.47	
	1 PAV-aged	74	67	22.77	93.66	74	65	26.20	95.44
		80	71	11.16		80	69	13.10	

Figure 4-4 also shows changes in the high-temperature PG (G*/sinδ) of recycled binders at the RTFO-aged step because rutting is a concern at during this step. As shown, the addition of RAP binder in the recycled asphalt resulted in a higher high-temperature PG of asphalt. The higher the RAP binder incorporation in the recycled asphalt, the higher the high PG grade, and the better the rutting performance. Also, it is obvious that the use of nanomodified asphalt improved rutting performance better than the use of an unmodified binder in recycled asphalt.

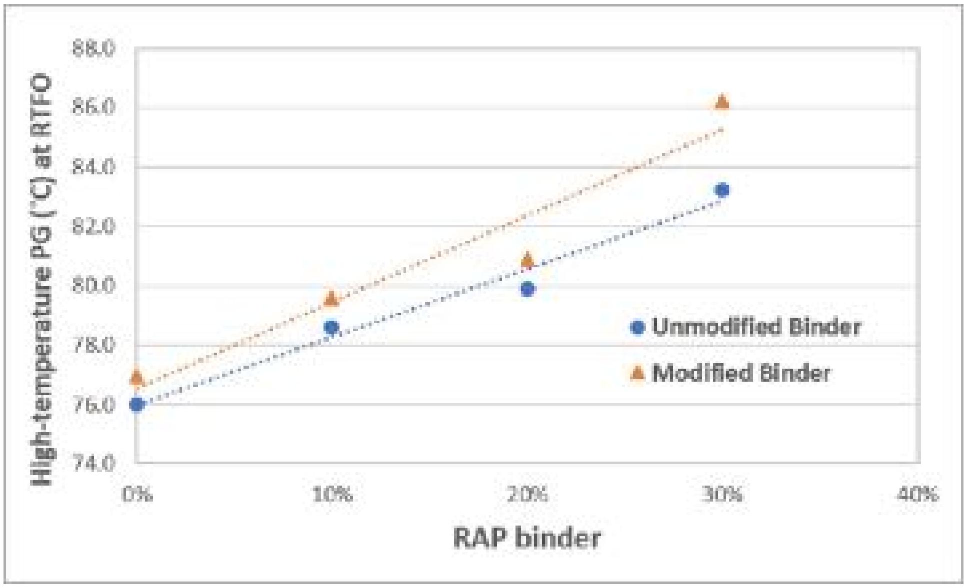


Figure 4-4: Changes in high-temperature PG of binders at RTFO-aged step with RAP binder percentage

4.5.2 Mid-Temperature Performance

The DSR test for each binder at each aging step was conducted at two different medium temperatures and is summarized in Table 4-7. Also, the mid-temperature PG of binders at each aging step was calculated by using Equation 2-1 and by considering criteria from Table 2-2. As shown, by the aging of the binders, the mid-temperature PG and G* of the binders increased. Inversely, the phase angle (δ) of the binders decreased by aging. Also, by increasing the incorporation of the RAP binder in the recycled asphalt, the mid-temperature PG of the binders increased.

As shown in Table 4-7, the original modified binder had lower PG, which shows a better fatigue performance. Consequently, the recycled binders using nanomodified asphalt also had a lower mid-temperature PG. This indicates the lower increase in the rate of a mid-temperature grade with the addition of RAP content in the recycled asphalt. This can be due to the incorporation of nanoparticles in the recycled binders.

Table 4-7: Mid-temperature PG of recycled binders

RAP Binder	Aging Step	Unmodified Binder				Modified Binder			
		Test Temperature (˚C)	δ (˚)	G*sin δ (MPa)	Mid temperature PG (℃)	Test Temperature (˚C)	δ (˚)	G*sin δ (MPa)	Mid temperature PG (℃)
0%	Unaged	24	63	1.44	16.06	24	60	1.44	14.79
		30	68	0.56		30	65	0.64	
	RTFO-aged	24	52	2.78	17.70	24	48	2.79	16.85
		30	57	1.59		30	54	1.71	
	1 PAV-aged	24	44	3.92	21.24	24	41	4.01	20.46
		30	49	2.30		30	46	2.76	
	2 PAV-aged	24	41	5.70	25.36	24	36	5.28	24.63
		30	45	3.20		30	41	3.13	
10%	Unaged	24	60	1.96	16.88	24	57	1.93	15.82
		30	64	0.89		30	60	0.96	
	RTFO-aged	24	49	3.04	18.65	24	45	2.88	16.34
		30	53	1.74		30	50	1.87	
	1 PAV-aged	24	41	4.01	22.12	24	38	3.88	21.48
		30	46	1.98		30	42	2.12	
	2 PAV-aged	24	40	6.07	25.97	24	32	5.38	24.87
		30	43	3.36		30	37	3.25	
20%	Unaged	24	58	2.23	18.32	24	54.5	2.18	16.32
		30	63	0.95		30	60.1	1.14	
	RTFO-aged	24	47	4.41	21.40	24	43.1	4.49	21.63
		30	51	3.30		30	49.2	3.42	
	1 PAV-aged	24	38	5.13	24.39	24	34.8	5.33	24.72
		30	44	3.47		30	40.6	3.12	
	2 PAV-aged	24	38	7.90	27.86	24	25.7	6.82	26.65
		30	40	3.88		30	29.1	3.38	
30%	Unaged	24	55	2.86	20.29	24	54.9	2.92	20.20
		30	59	1.16		30	59.8	1.25	
	RTFO-aged	24	46	5.26	24.77	24	43.6	5.29	24.81
		30	50	3.55		30	48.4	3.49	
	1 PAV-aged	24	37	6.67	27.40	24	36.3	7.03	27.55
		30	42	4.01		30	40.1	3.95	
	2 PAV-aged	24	28	8.35	29.07	24	24.3	7.61	28.80
		30	31	4.55		30	28.7	4.5	

Figure 4-5 shows the changes in the mid-temperature grade of recycled binders with different percentages of the RAP binder at the 2 PAV-aged step. As shown, the recycled nanomodified binder had a lower mid-PG compared to the recycled asphalt using an unmodified binder. According to the AASHTO specification criteria listed in Table 2-2, the mid-temperature grade of binder PG 67-22 should be 26.5 ˚C. In this case, the recycled asphalt with an unmodified binder with 0% and 10% RAP binder met the standard criteria. However, the recycled asphalt with a nanomodified binder with 0%, 10% and 20% RAP binder met the requirements. This indicates that the maximum RAP binder incorporation into the recycled binders with unmodified and nanomodified binders can be 10% and 20%, respectively.

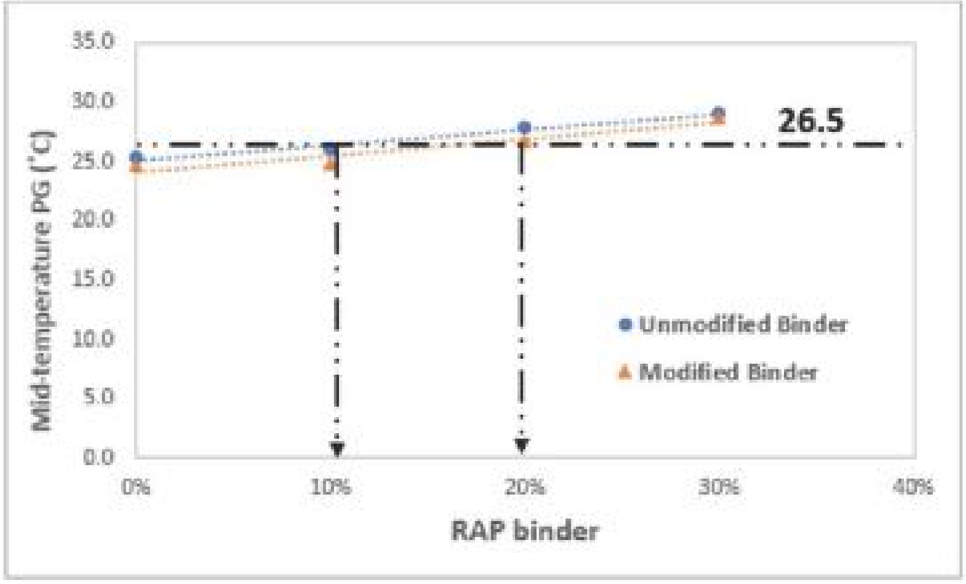


Figure 4-5: Changes in mid-temperature PG of binders at the 2 PAV-aged step with RAP binder percentage

4.5.3 Low-Temperature Performance

The low-temperature performance of recycled asphalt with nanomodified and unmodified binders was evaluated using the BBR test at the 1 PAV and 2 PAV aging steps. Results are summarized in Tables 4-8 and 4-9 for the unmodified and modified binders, respectively. At each aging step, the BBR test was conducted at two different temperatures of -12 ˚C and -18 ˚C. The stiffness and m-value at 60 seconds are listed in Tables 5 and 6. As shown, the critical low temperature (low-temperature PG) of the binders at each aging step was calculated using Equations 2-2 and 2-3. As shown, with the aging of the binders, the low-temperature grade of binders increased and became more critical. With the aging of binders and increase of the RAP binder percentage in the recycled binders, the stiffness of the binders increased. Conversely, the m-value decreased. Also, the higher calculated critical low-temperature of a binder using stiffness and the m-value was selected as the low-temperature PG of the binder. As the original binder grade was PG67-22, it was expected that the low-temperature grade of recycled binders would drop lower than -22 ˚C. Accordingly, the recycled binder using the unmodified binder with a maximum percentage of 10% RAP binder met this criterion. Similarly, for the recycled binder using the nanomodified binder with a maximum of 20% of RAP binder met this criterion. This concept is also shown in Figure 4-6 in which the changes in the low-temperature PG of recycled unmodified and modified asphalt with different percentages of RAP binder are displayed. As indicated, the low-temperature grade of recycled binders increased with the increase of the RAP binder in the recycled asphalt. More significantly, the lower low-temperature grade of recycled binders using nanomodified binders is obvious.

Table 4-8: Low-temperature grade of unmodified recycled binders

RAP Binder	Aging Step	Test Temperature (˚C)	Stiffness (MPa)	m-value	Critical Temperature (Stiffness)	Critical Temperature (m-value)	Low Temperature PG
0%	1 PAV Cycle	-12	185	0.329	-27.84	-25.48	-22.66
		-18	304	0.279			
	2 PAV Cycle	-12	267	0.311	-23.79	-22.66	
		-18	395	0.211			
10%	1 PAV Cycle	-12	213	0.318	-26.76	-23.93	-21.93
		-18	328	0.262			
	2 PAV Cycle	-12	301	0.305	-21.93	-22.28	
		-18	404	0.198			
20%	1 PAV Cycle	-12	255	0.292	-24.78	-20.67	-17.74
		-18	362	0.256			
	2 PAV Cycle	-12	346	0.284	-17.74	-20.99	
		-18	423	0.189			
30%	1 PAV Cycle	-12	308	0.262	-21.51	-17.35	-17.29
		-18	426	0.213			
	2 PAV cycle	-12	384	0.257	-17.29	-19.26	
		-18	526	0.163			

Table 4-9: Low-temperature grade of nanomodified recycled binders

Rap Binder	Aging Step	Test Temperature (˚C)	Stiffness (MPa)	m-value	Critical Temperature (Stiffness)	Critical Temperature (m-value)	Low Temperature PG
0%	1 PAV Cycle	-12	179	0.362	-28.20	-27.31	-24.18
		-18	295	0.292			
	2 PAV Cycle	-12	241	0.333	-25.39	-24.18	
		-18	355	0.242			
10%	1 PAV Cycle	-12	196	0.339	-27.91	-25.71	-22.86
		-18	302	0.276			
	2 PAV Cycle	-12	248	0.312	-24.61	-22.86	
		-18	384	0.228			
20%	1 PAV Cycle	-12	236	0.317	-26.18	-23.59	-22.27
		-18	333	0.253			
	2 PAV Cycle	-12	295	0.305	-22.29	-22.27	
		-18	416	0.194			
30%	1 PAV Cycle	-12	289	0.283	-22.62	-20.50	-18.15
		-18	414	0.215			
	2 PAV Cycle	-12	368	0.252	-18.15	-18.21	
		-18	506	0.176			

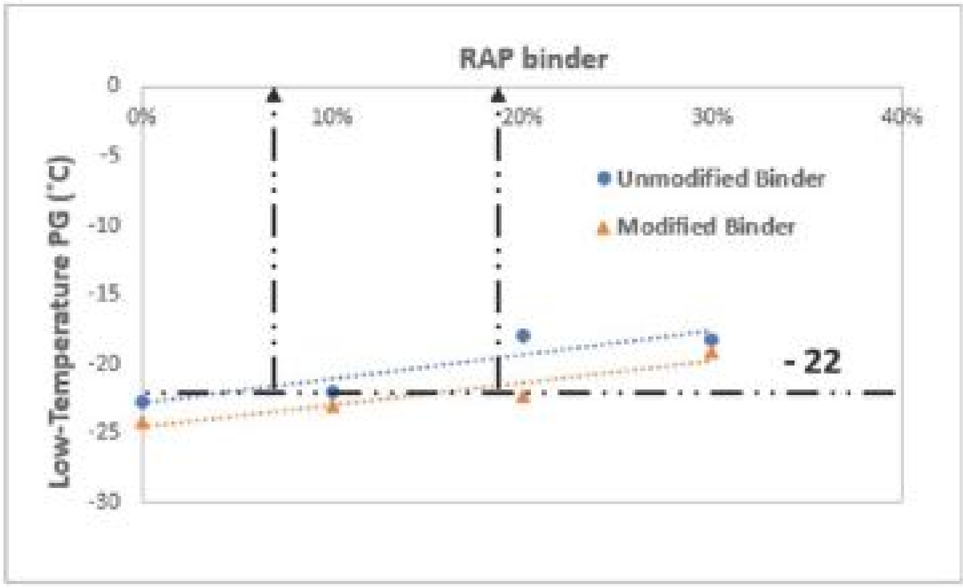


Figure 4-6: Low-temperature PG of recycled binders at 2 PAV-aged step

4.5.4 Chemical Properties of Binders

The functional chemical composition of binders in each aging step was measured using the FTIR test. The chemical compositions that change with the aging of asphalt are Carbonyl (C) and Sulfoxide (S). The changes in S plus C with aging for recycled modified and unmodified binders are shown in Figure 4-7. As shown, the C+S absorbance of binders increased with aging. This trend was also demonstrated with the changes that occurred in S or C separately. However, because the pattern was the same, the results are not shown.

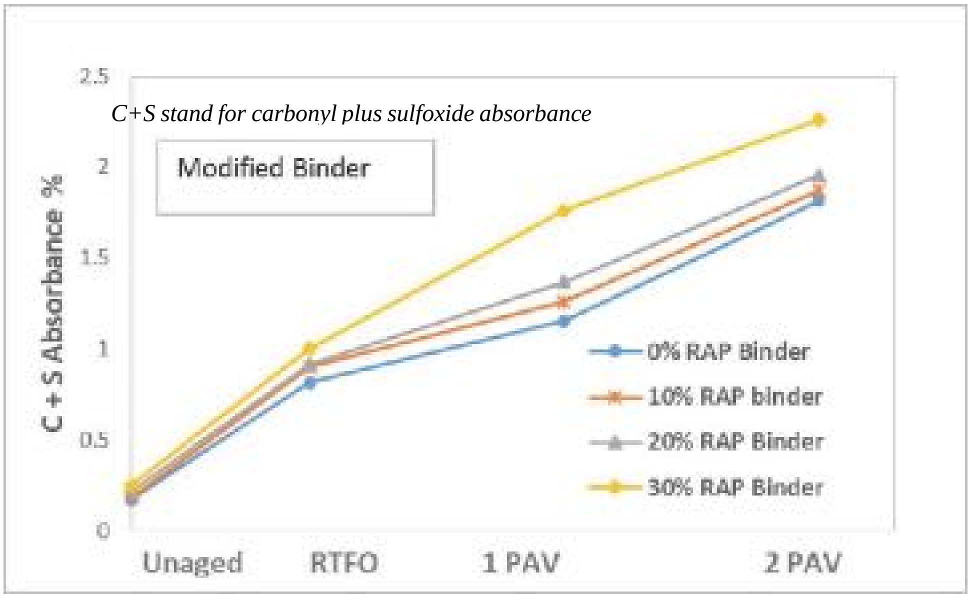


Figure 4-7: Changes in Carbonyl + Sulfoxide with aging

The correlation of the phase angle measured with DSR at 30 ˚C and 74 ˚C with chemical indexes (C+S absorbance) is shown in Figure 4-8. The measure phase angle at 74 ˚C is representative of binder rheological behavior at a high temperature (rutting), and the measure phase angle at 30 ˚C is representative of binder behavior at a medium temperature (fatigue). As shown, with aging and the increase of chemical indexes, the phase angle of binders decreased. Also, the rate of decrease in the measured phase angle at 30 ˚C is higher than the measured phase angle at 74 ˚C. More significantly, the rate of the phase angle decrease in the modified binders was higher than the unmodified binders. Also, the increase of the RAP binder in the recycled binders causes a higher reduction in the phase angle.

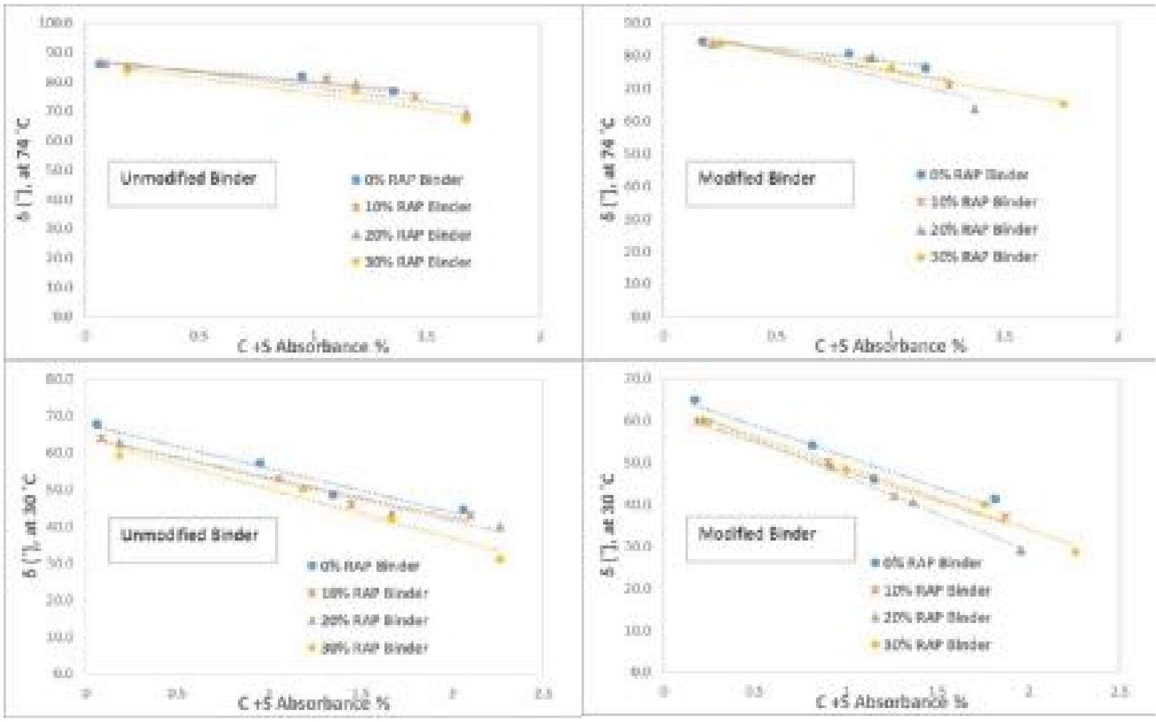


Figure 4-8: Correlation of chemical indexes with phase angle

The correlation of changes in measured G* at two different temperatures of 74 ˚C and 30 ˚C with changes in chemical indexes (C+S) and aging is shown in Figure 4-9. As shown, there is a strong correlation between chemical indexes and logG*. This correlation can be seen in recycled binders using unmodified and modified binders. Also, with the increase of the RAP binder in the recycled binders, the G* and C+S increased. More significantly, the measured G* for recycled binders with nanomodified binders was higher than the unmodified binders.

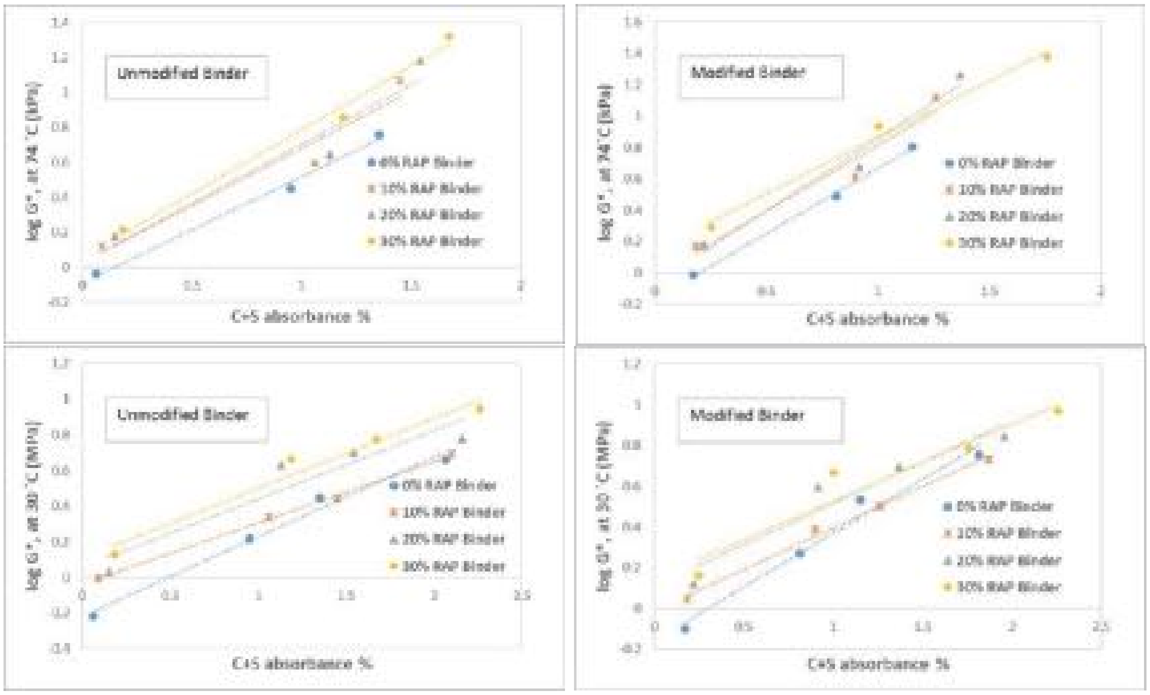


Figure 4-9: Correlation between changes in chemical indexes and LogG with aging*

4.5.5 Discussion

The nanoclay modified binder was used in the partial recycling of the RAP (aged) binder due to the unique properties of nanoclay. It was shown that nanoclay particles can modify asphalt binder and improve high-temperature (rutting), medium-temperature (fatigue), and low-temperature (thermal cracking) performance of asphalt binder. These improvements were due to the increase in the complex modulus and m-value, and the decrease in the phase angle and stiffness of the nanomodified asphalt binder compared to the unmodified asphalt binder. In this case, in order to investigate the incorporation of the nanomodified asphalt binder in the recycling of RAP material, a nanomodified and unmodified binder were used to recycle the RAP binder (2 PAV-cycle aged binder).

According to previous studies, the incorporation of RAP binder in the neat binder (unmodified binder) can improve the rutting performance because it can increase the stiffness and complex shear modulus of asphalt. This behavior was also shown in the results of this study. However, the incorporation of RAP cannot affect the phase angle of the asphalt binder, which can cause problems in fatigue and the low-temperature cracking resistance of asphalt binder. More significantly, the use of a nanomodified binder in the recycling of the RAP binder intensified the improvement in the rutting performance of recycled binders due to the increase in the complex shear modulus and the decrease in the phase angle of recycled asphalt binders.

In the medium-temperature performance of asphalt, the same behavior was shown as the high-temperature performance with the incorporation of the RAP binder. The incorporation of RAP in the recycled binders increased the complex modulus of the asphalt binder, which causes the restriction in the percentage of RAP binder incorporation in the recycled asphalt. As a result, the inclusion of RAP in recycled asphalt with the unmodified binder should be restricted to 10% based on the AASTO T315 standard specification to meet the medium-temperature performance criteria of recycled binders. The 10% RAP binder in the recycled binder is equivalent to the incorporation of 17% RAP material in the recycled asphalt mixture. However, when the nanomodified asphalt binder was used to recycle the asphalt binder, the maximum inclusion of a 20% RAP binder in the recycled binder was allowed to meet the recycled binder medium-temperature criteria. This indicated that the addition of nanoparticles in the binder caused a decrease in the phase angle and an increase in the complex shear modulus of the asphalt binder, leading to a decrease in the mid-temperature PG of the nanomodified binder, and less of an increase in the PG grade of the binder with aging.

The incorporation of the RAP binder in the recycled binders caused an increase in the creep stiffness and a decrease in the m-value of binders. This behavior limits the incorporation of RAP percentages in the recycled binder because it can result in less resistance to thermal cracking. Although the use of the nanomodified binder in the recycling of the RAP binder improved the thermal cracking resistance of the asphalt binder, it limited the incorporation of the RAP asphalt binder in the nanomodified binder to 20%. This indicates that use of a nanomodified binder in the recycling of asphalt can increase the incorporation of the RAP binder by 10%. However, in some locations where asphalt pavement does not experience severely cold weather, it is possible to consider a higher low-temperature grade for the recycled asphalt binder and increase the RAP percentage.

The strong correlation ($R^2 > 92\%$) of chemical indexes (carbonyl and sulfoxide) with rheological properties (complex shear modulus and phase angle) indicates that carbonyl and sulfoxide are good representatives of recycled asphalt aging using nanomodified and unmodified binders. It showed that the increase in the incorporation of RAP percentages in recycled binders could increase the carbonyl plus sulfoxide concentration at the unaged step. However, the concentration of carbonyl and sulfoxide in aged recycled binders with a nanomodified binder were slightly less than recycled binders using an unmodified binder. This trend indicated the anti-aging property of the nanomodified binder, which makes it suitable for use in the recycling of RAP material and construction of pavement for severe conditions. More significantly, the correlation of chemical indexes with measured rheological indexes at two different high and medium temperatures demonstrated that the chemical indexes of asphalt binders are a good representative of the rutting and fatigue performance of asphalt pavement.

It should be noted that in this study, the nanomodified binder was prepared with 3% of nanoparticles. According to literature, the percentage of nanoparticles in the modified binder and the type and PG of the virgin binder are factors that affect the properties of the nanomodified binder. Therefore, other studies can be conducted by considering different RAP binders, different percentages of nanoparticles in a nanomodified binder, and virgin binders with different PG types. Although this study has these limitations, and other studies need to be conducted to verify this study's results, it can pave the way toward using the nanomodified binder in the recycling and construction of new roadways.

Hossain et al. (2014) compared the cost of manufacturing a nanoclay modified binder with a polymer modified binder. It was concluded that the cost of the nanomodified binder production with 2% and 4% nanoparticles decreased to 22% and 33% compared to the polymer modified binders' manufacturing, respectively. Accordingly, it can be concluded that use of nanomodified binder in the recycling of RAP material not only can save the environment by increasing the incorporation of RAP content in the recycled pavement, but it is also economically more cost-effective than the use of the polymer-modified binder.

4.5.6 Proposed Conceptual Procedure to Define the Allowable RAP Content in Recycled Asphalt

As discussed in Section 4.5.5, defining the allowable amount of RAP content in the recycled nanomodified asphalt depends on the following factors.

- PG of virgin binder used to prepare nanomodified asphalt
- Percentage of nanoparticles that will be added to the virgin binder
- PG of produced nanomodified asphalt

- PG of RAP asphalt that will be recycled

To determine the amount of allowable RAP content in the recycled asphalt, the following steps should be followed. Steps 1 to 3 are the procedures used to prepare the nonmodified binder and its high-temperature PG. In cases where the nanomodified binder is available, these steps can be skipped and steps 4 and 5 should be followed.

Step 1: The high-temperature PG of the virgin binder should be measured.

Step 2: The amount of nanoparticles that need to be added to the virgin binder to produce the nanomodified binder should be defined.

Step 3: The high-temperature PG of the produced nanomodified binder should be measured. As shown in Figure 4-10, by having the virgin binder high-temperature PG and an amount of nanoparticles in the binder, the nanomodified binder high PG can be defined. It should be noted that Figure 4-10 is a schematic of the relationship between the high-temperature PG of the virgin binder and the percentage of nanoparticles in the binder with produced nanomodified PG. In this study, 3% of nanoclay was added to the virgin binder with a high-temperature PG of 72 ˚C. Then, the high-temperature PG of the nanomodified binder was measured, which was 74 ˚C.

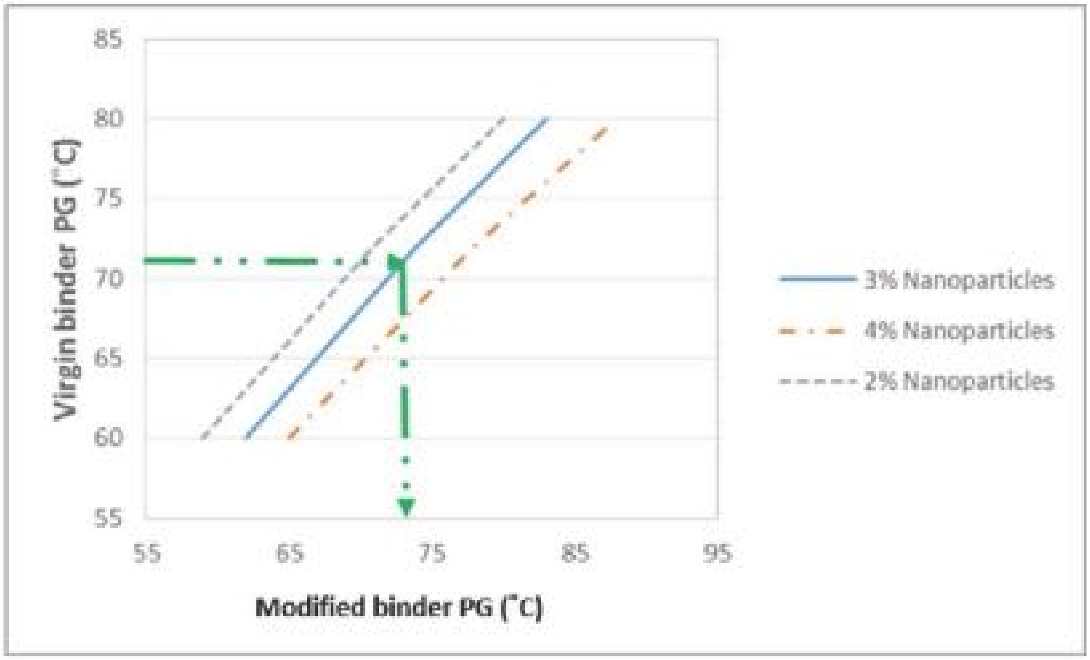


Figure 4-10: Defining the allowable amount of RAP in the recycled asphalt

Step 4: The high-temperature PG of RAP asphalt that will be recycled should be measured.

Step 5: By having the high-temperature PG of RAP from step 4 and the high-temperature PG of the nanomodified binder, it is possible to define the allowable amount of RAP in the nanomodified asphalt (Figure 4-11). Figure 4-11 is a schematic of the allowable amount of RAP in the recycled binder using a nanomodified binder based on the performed experimental study. As shown, by having a nanomodified binder with a high PG of 74 ˚C and a RAP with a PG of 94 ˚C, the allowable amount of RAP content is determined to be 30%.

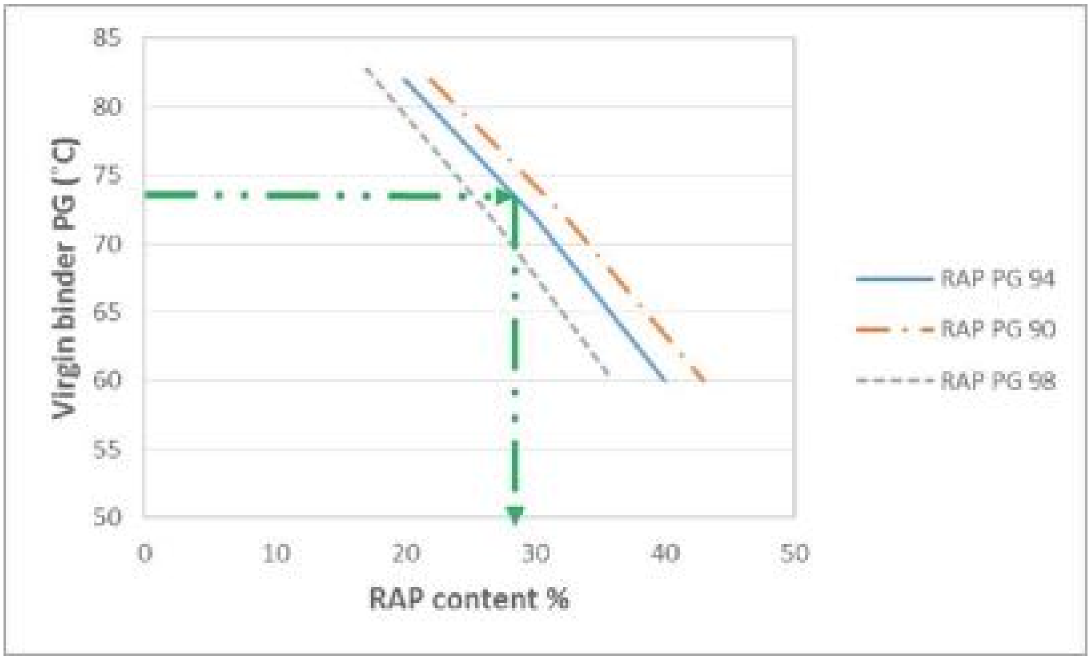


Figure 4-11: Defining the allowable amount of RAP in the recycled asphalt

5 CHAPTER 5: SUMMARY AND CONCLUSION

In this chapter, the results of the experimental study and analysis conducted are listed. The objective of this study was to evaluate the performance of recycled binder using the RAs and nanomodified binder. Also, the aging rate and relationship between rheological and chemical characteristics of recycled binders were investigated. The overall conclusion from this study and recommended future studies are described in this chapter.

5.1 Recycling of 100% RAP Binder using Recycling Agents

The short- and long-term performance of recycled binders were evaluated and compared with the virgin binder and it was concluded that the recycled binders' fatigue and low-temperature cracking performance were better than the virgin binders. The rutting performance of recycled binders were also compared to virgin binders, and the recycled binders rutting performance with RA2 and RA3 were better than that of the virgin binders.

From chemical perspective, the carbonyl and sulfoxide functional groups of asphalt binders were the representative of asphalt binder age. These functional groups concentration in the binder increased with asphalt age. Also, they had a very strong correlation with the rheological properties of asphalt. This correlation could be used to predict asphalt binder service life and performance. More importantly, it was shown that the recycled binder that had the lower concentration of carbonyl and sulfoxide in unaged step could provide a better short- and long-term performance. This result was used to select proper RAs in the recycling of asphalt pavement. Also, it was shown that the chemical indexes of asphalt binder could be used to define the required amount of RA in the recycling of asphalt pavement.

The results from the recycling of asphalt binder using different RAs and the strong correlation of chemical composition of asphalt binder with its rheological properties were used to develop a recycling process that was based on the chemical composition of asphalt binder. This method was highly based on performing FTIR test which is not time-consuming and costly in compared to the conventional recycling method which is based on performing rheological tests on the asphalt pavement.

5.2 Recycled Asphalt Binder using a Nanomodified Binder

This study was conducted to evaluate the use of a nanomodified binder in the recycling of the RAP binder. For this evaluation, rheological and chemical experimental tests were performed. The incorporation of RAP binder in recycled asphalt could improve the rutting performance of the pavement. The higher the percentage of RAP, the better the rutting performance of recycled asphalt. However, the increase of RAP binder in the recycled asphalt could cause a problem in fatigue (mid-temperature) and the low-temperature (thermal cracking) performance of asphalt pavement. It was shown that the nanomodified binder had rutting, fatigue, and low-temperature performance levels better than the unmodified (neat) asphalt binder. Therefore, it was concluded that the nanomodified asphalt can be used in the recycling of RAP material and can increase the percentage of RAP material in the recycled pavement compared to the use of an unmodified binder in the recycling process.

Based on the DSR, BBR, and FTIR tests that were conducted on the recycled binders using the nanomodified binder, it was possible to incorporate a 20% of RAP binder in the recycling of pavement. However, only 10% of RAP binder could be included in the recycled binders using the unmodified binder. This result indicated that the incorporation of RAP materials in the recycling of asphalt pavement using the nanomodified binder can be two times of using unmodified binder in the recycling process.

Also, the lower concentration of chemical indexes with aging in nanomodified recycled binders demonstrated the antiaging properties of nanomodified asphalt. This property can justify the use of a nanomodified binder in severe environmental and traffic conditions.

5.3 Recommended for Future Studies

1- In this research, different RAs were selected to be used in the recycling of asphalt. One study can be conducted to categorize the RAs based on their chemical properties.

2- To prepare the nanomodified binder, 3% of nanoparticles were used. It is recommended to develop a procedure to identify the optimum amount of nanoparticles in the nanomodified binder based on the properties of the nanomodified binder.

3- In this research, the nanoparticles were mixed with a binder to prepare the nanomodified binder, and it was not possible to add nanoparticles to the asphalt mixture. It is recommended to overcome drying by oversoftening, in which more rejuvenations are added, followed by adding some nanomaterial.

4- This study’s tests were conducted on the asphalt binder. It is recommended to perform these tests on the asphalt mixture to compare its results with this study’s results.

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Education and Honors

2011	Yazd University (Iran)	Civil Engineering	B.S.
2014	Isfahan University of Technology (Iran)	Civil Engineering-Geotechnical Engineering	M.S.
Ph.D. Candidate, Present	Florida International University	Civil Engineering	

Outstanding Student of the Year in Transportation Research and Education award, Accelerated Bridge Construction University Transportation Center, The United States Department of Transportation, 2019.

Dissertation Year Fellowship (DYF) Award, Florida International University, University Graduate School, Spring and Summer 2019.

American Concrete Institute (ACI) Award for University Student Activities, ACI Outstanding University, 2017

Journal Publications

1. Baqersad, M., M. Mohammadafzali, B. Choubane, C. Holzschuher, A. Hamedi and H. Ali (2018). "Precision Assessment of the Florida Texture Meter in Hot Mix Asphalt." ASCE Journal of Transportation Engineering, Part B: Pavements 144(2): 04018003, ASCE Publications.
2. Baqersad, M., Mohammadafzali, M., Choubane, B., Holzschuher, C., Hamedi, A., & Ali, H. (2018). "Application of Laser Macrotexture Measurement for Detection of Segregation in Asphalt Pavements." ASCE Journal of Transportation Engineering, Part B: Pavements 144(3): 04018032, ASCE Publications.

3. Baqersad, M., Hamedi, A., Mohammadafzali, M., & Ali, H. (2017). "Asphalt Mixture Segregation Detection: Digital Image Processing Approach." Advances in Materials Science and Engineering, 2017, Hindawi Publications.

4. Massahi, A., Ali, H., Koohifar, F., Baqersad, M., & Mohammadafzali, M. (2017). "Investigation of pavement raveling performance using a smartphone." International Journal of Pavement Research and Technology, Elsevier Publications.

5. Baqersad, M., Haghighat, A. E., Rowshanzamir, M., & Bak, H. M. (2016). "Comparison of coupled and uncoupled consolidation equations using finite element method in plane-strain condition." Civil Engineering Journal, 2(8), 375-388, K.N. Toosi University of Technology Publications.

6. Massahi, A., Ali, H., Koohifar, F., Baqersad, M., & Mohammadafzali, M. (2018). "Implementing a Smartphone Application for Assessing the Raveling Performance of Asphalt Pavements". IJASRE, 4, Science Publications.

7. Mohammadafzali, M., H. Ali, G. Sholar, W. Rilko, and Baqersad, M., (2018). "Effects of Rejuvenators and Aging on Binder Homogeneity of Recycled Asphalt Mixtures." ASCE Journal of Transportation Engineering, Part B: Pavements, 145(1), 04018066, ASCE Publications.

8. Ali, H. and Baqersad, M., (2018). "Pavement Manager at Risk in Construction of Hot Mix Asphalt" Advances in Civil Engineering, 2018, Hindawi Publications.

9. Baqersad, M., Ali, H., Haddadi, F., & Khakpour, I. (2019). Short-and long-term rheological and chemical characteristics of nanomodified asphalt binder. Petroleum Science and Technology, 1-12.

10. Massahi, A, Hadi, M., Shams, K., & Baqersad, M. (2019). "Evaluating Incident Responsive Signal Control Plans Using Multi-Resolution Modeling." Transportation Research Record Journal, 2019, In Press.

11. Baqersad, M., & Ali, H. (2019). Rheological and chemical characteristics of asphalt binders recycled using different recycling agents. Construction and Building Materials, 228, 116738.