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LABORATORY EVALUATION OF THE IMPACT OF ADDITIVES ON ASPHALT BINDERS USED IN COLD REGIONS

by

Neirouz Bouhrira

A Thesis

Submitted to the Department of Civil and Environmental Engineering College of Engineering In partial fulfillment of the requirement For the degree of Master of Science in Civil Engineering at Rowan University April 2020

Thesis Chair: Yusuf Mehta, Ph.D., P.E.



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Dedications

I dedicate my dissertation work to all my family members and friends. I am grateful to my loving parents Mohamed and Leila, my grandmother Fatma and my aunt Sonia for being always by my side. A special thanks to all my family members and friends, especially Ahmed Saidi, Ahmad Alfalah, and Basel Al-Badr, for their valuable support throughout the process.



Acknowledgements

I would like to express my gratitude for Dr. Yusuf Mehta, professor and graduate coordinator in the department of civil and environmental engineering, for offering me the opportunity to be part of his team and work in this project. His willingness to provide feedback and support made the completion of this research a valuable experience.

I had the pleasure to be part of Rowan University, and I am grateful to all the university faculty members and administrators that assisted me throughout the process. A special thanks to my thesis committee: Dr. Cheng Zhu and Dr. Gilson Lomboy from Rowan University Civil and Environmental Engineering Department for agreeing to be in my committee. A special thanks to Caitlin Purdy, CREATEs lab manager, for her dedication and support. Finally, I would like to thank the US Department of Defense (DoD) for the research grant, which made this research possible.



Abstract

Neirouz Bouhrira LABORATORY EVALUATION OF THE IMPACT OF ADDITIVES ON ASPHALT BINDERS USED IN COLD REGIONS 2019-2020 Yusuf Mehta, Ph.D., P.E. Master of Science in Civil Engineering

The objective of this research study is to evaluate the effect of additives (polymers, nanomaterials, and softening agents) on rutting, cracking, and fatigue performance of asphalt binders commonly used in cold regions (PG 52-34 and PG 64E-40). In this study, the first phase consisted of using polymers (Styrene-Butadiene-Styrene, Ground-Tire-Rubber) and nanomaterials (TiO2 and SiO2) to modify two asphalt binders used in cold regions (PG52-34 and PG64E-40). The second phase of the study consisted of adding a combination of softening agents (Corn oil or Sylvaroad) with polymers (SBR, Epoxy, and SBS) to PG52-34 asphalt binder. The performance evaluation was conducted using the Brookfield Viscometer (RV), Dynamic Shear Rheometer (DSR), standard Bending Beam Rheometer (BBR), BBR strength, and Linear Amplitude Sweep (LAS). The testing results showed that the polymers could improve the rutting, cracking and fatigue performance of asphalt binders. GTR improved high and low temperature performance grades, fatigue properties, and strain at failure. Nano-TiO₂ and SiO₂ did not show a considerable performance improvement compared to SBS and rubber in low temperature and fatigue properties. Results also showed that 7.5% SBS combined with corn oil is considered the best candidate asphalt binder modification to improve the resistance to rutting, fatigue, and thermal cracking.



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Chapter 1

Introduction

Background

Asphalt binder is a by-product of petroleum oil refinery typically used in pavement construction due to its low cost and ease of construction. Asphalt binder is a temperature-dependent material that tends to become a viscous fluid at high temperatures, a semi-solid material at intermediate temperatures, and a stiff, brittle viscoelastic material (glass-like elastic solid) at low temperatures. This variation in temperatures coupled with the increase in traffic wheel loads can cause performance failures in asphalt pavements such as thermal or low-temperature cracking, permanent deformation, and fatigue cracking. Thus, over the last few decades, researchers have focused on developing innovative asphalt binders using additives or modifiers, i.e., thermoplastic elastomer styrene butadiene styrene (SBS), to enhance pavements performances (Timm et al., 2012; Greene et al., 2014; Kim et al., 2010; Bahia et al., 2001; Xie et al., 2016; Shen et al., 2012).

For instance, the study of Tim et al. (2012) showed that adding 7.5% SBS leads to stiffer asphalt mixtures, improves the fatigue endurance limit, and decreases susceptibility to low temperature cracking. Similarly, Kuennen (2013) added 7.5% SBS to asphalt mixtures used as an intermediate course, which after 8 months, showed no sign of distresses. Other studies focused on developing highly elastic binders using rubber. For example, Sousa et al. (2013) found that Reacted and Activated Rubber (RAR) modified



mixes exhibited more strength, resiliency and improved recovery, fatigue, and rutting resistance compared to conventional asphalt rubber mixes. Camargo et al. (2019) investigated permanent deformation and fatigue behavior of neat, polymer, and rubber asphalt binders. It was found that modified asphalt binders showed better fatigue behavior compared with the neat binder and, in particular, rubber modified binders exhibited the best fatigue resistance.

Nanomaterials, including Nano-clay, Nano-Titanium dioxide (nano-TiO2), and Nano-Silicon Dioxide (SiO2), were used to improve the performance properties of asphalt binders. Shafabakhsh et al. (2015) showed that adding Nano-TiO2 led to great improvement in permanent deformation and fatigue life of asphalt mixtures, while 5% nano -TiO2 (by weight) as a modifier of asphalt was the optimal content in asphalt mixtures. Goh et al. (2011) reported that the addition of Nano-clay to asphalt mixtures would improve moisture susceptibility and decrease moisture damage potential through increasing the tensile strength of these mixtures. Amirkhanian et al. (2010) investigated the performance of asphalt binder modified with carbon Nano-particles. The authors found that the addition of Nano-particles increased the viscosity, failure temperature, complex modulus, and elastic modulus values as well as improved rutting resistance of asphalt binder. Other researchers studied the impact of rejuvenators, so-called softening agents, on the rheological, physical, and chemical performance of asphalt binders (Zaumanis et al., 2014; Zaumanis et al. 2013; Zargar et al., 2012). Some of these studies highlighted the ability of rejuvenators to enhance the low temperature cracking resistance of reclaimed asphalt pavement (RAP) binder and improve workability during construction since it reduces



viscosity and stiffness. Bonicelli et al. (2017) evaluated the mechanical and long-term performance of recycled asphalt mixes containing a combination of rejuvenators and polymers through a laboratory analysis of physical and rheological properties. Results showed that the combination of rejuvenators and polymers improved the overall durability of high recycled asphalt mixes.

In summary, the outcomes of the previous studies showed that high polymers and softening agents (rejuvenators) could potentially improve the performance of asphalt mixtures and high reclaimed asphalt pavement mixtures.

Problem Statement

During the last decades, researchers (Shafabakhsh et al., 2015; Soleymani et al., 2004., Timm et al., 2012) have conducted studies to construct sustainable, long-lasting, high-performing pavements/roadway and different binder modification procedures were developed. However, the majority of previous studies directed their researches towards assessing the impact of modifiers on stiffer binders (such as PG88-22, PG76-22) used for warm and hot regions, and very limited information is available pertaining to the performance of asphalt binders with high polymer and softening agent, especially properties at low temperatures. Therefore, there is a need to investigate the performance properties of the new asphalt binders.

Research Hypothesis

The introduction of additives such as softening agents, high polymers, and nanomaterials in soft asphalt binders used in cold regions improves the physical properties



of asphalt binders and leads to better performing asphalt in terms of rutting and low temperature cracking without sacrificing permanent fatigues properties.

Goal & Objectives

The aim of this research project is to develop an understanding of the way in which additives improve the properties of soft binder recommended in cold regions and evaluate the laboratory performance of asphalt binders with high polymers, nanomaterials, and softening agents. The objectives to accomplish the overall goal of this study:

Phase 1: Polymers and nanomaterials modified asphalt binders.

- Determine the impact of polymers and nanomaterials on the viscosity using Brookfield Rotational Viscometer.
- Determine the performance grade of the modified binders in accordance with AASHTO M320 on modified soft asphalt binders.
- Investigate the impact of additives on PG grade using Dynamic Shear Rheometer (DSR) and standard Bending Beam Rheometer (BBR) according to AASHTO T315 and AASHTO T313.
- Investigate the effect of additives on the creep stiffness parameter of asphalt binders from the standard Bending Beam Rheometer (BBR) results.
- Evaluate the rutting performance of modified asphalt binders using the Multiple Stress
 Creep and Recovery (MSCR) testing by looking into the J_{nr} at 3.2 kPa and percent
 recovery at 3.2 kPa.



• Evaluate the fatigue behavior of asphalt modified binders using the Modified Linear amplitude Sweep (LAS) testing on polymer and nanomaterials modified soft asphalt binders.

Phase 2: Polymers and softening agents modified asphalt binders.

- Determine the performance grade of the modified binders in accordance with AASHTO M320 on modified soft asphalt binders.
- Investigate the impact of additives on PG grade using Dynamic Shear Rheometer (DSR) and standard Bending Beam Rheometer (BBR) according to AASHTO T315 and AASHTO T313.
- Investigate the effect of additives on the creep stiffness parameter of asphalt binders from the standard Bending Beam Rheometer (BBR) results.
- Evaluate the rutting performance of modified asphalt binders using the Multiple Stress Creep and Recovery (MSCR) testing by looking into the J_{nr} at 3.2 kPa and percent recovery at 3.2 kPa.

Research Approach

The approach utilized to meet the overall goal of this study was divided into two phases. The first phase consisted of evaluating the laboratory performance of polymers and nanomaterials modified asphalt binders used in cold regions. In addition to that, the second phase consisted of evaluating the impact of the combination of polymers and softening agents on the performance of asphalt binders used in cold regions.

The research approach consisted of the following tasks:



Task 1: Conduct a comprehensive literature review. The objective of this task is to conduct a comprehensive literature review to synthesize information pertaining to the modification of asphalt binders and associated improvements in high temperature, low temperature, and fatigue properties by reviewing domestic and international previous binder modification-related studies. This task will present a general introduction about asphalt binder modification and the reasons behind adopting this technology followed by the history and present of asphalt binder grading systems. This chapter will include the types of additives used in asphalt binder modification and their effect on binder performance. In addition to that, the impact of additives on the rutting, cracking, and fatigue performance will be summarized by presenting the results of several numbers of asphalt binder testing already performed in various studies.

Phase 1: Polymers and nanomaterials modified asphalt binders. Regarding the first phase of the study, the research approach consisted of the following tasks:

Task 1: Select, modify, and prepare materials to be tested. In this task, the selection of material and their preparation will be performed. Two asphalt binders: neat PG 52-34, and polymer modified binder (PMB) PG 64E-40 will be used as base binders. These binders were selected because they are commonly used in cold regions and the northern United States. Types and contents of additives are selected based on previous studies. Several modified asphalt binders will be produced with various additives fractions, then subjected to various short term and long-term conditioning regimes prior to performance testing to evaluate improvement in asphalt binder properties. In this first phase, for PG64E-40 asphalt binder, several types of polymers were added, such as

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Ground Tire Rubber (GTR) and Nanomaterials (TiO₂ and SiO₂). Regarding PG52-34 asphalt binders, the same modifiers were used in addition to Styrene –Butadiene-Styrene (SBS).

Task 2: Conduct performance testing. In this task, several asphalt binder performance testing will be conducted. For the first modification procedure, the viscosity of binders will be determined by means of the Brookfield Viscometer (RV) in accordance with AASHTO T316. The performance grade of all binders will be determined according to AASHTO M320. Dynamic Shear Rheometer (DSR) and Bending Beam Rheometer (BBR) will be used to grade all the asphalt binders in accordance with AASHTO M320 and investigate the impact of additives on the performance grading. In addition to that, the Bending Beam Rheometer (BBR) will be a means to evaluate the creep stiffness in accordance with AASHTO T313. The rutting performance of asphalt binders will be characterized by means of the Multiple Stress Creep Recovery (MSCR) testing. This performance testing will be conducted according to AASHTO T350, and performance will be evaluated through the non-recoverable creep compliance and percent recovery parameters at 3.2KPa. Moreover, the rheological properties will be investigated using a frequency sweep test performed with the Dynamic Shear Rheometer (DSR). The modified BBR will be used to investigate the low temperature properties, while fatigue properties will be evaluated using modified Linear Amplitude Sweep (LAS). For the second modification procedure, previous performance testing will be conducted to evaluate the same properties except for the viscosity, the modified BBR, and LAS testing.



Task 3: Discussion and analysis of the performance testing results. This task is important to gain an understanding of the improvement in the properties of asphalt binder imparted by modifiers/additives. This task is important to gain an understanding of the improvement in the properties of asphalt binder imparted by modifiers/additives. The evaluation of asphalt binders' properties will be conducted through the analysis of the performance testing outputs. In this task, the ANOVA analysis will be performed for some asphalt binder testing.

Task 4: Summary, conclusion, and recommendations. In this section, based on the performance testing, results will be summarized, and conclusions and recommendations will be drawn concerning in order to select the best additive and exclude the additives that may not show good performance.

Phase 2: Polymers and softening agents modified asphalt binders. The following tasks will be adopted to fulfill the overall goal of the second phase of the study:

Task 1: Select, modify, and prepare materials to be tested. In this task, the selection of material and their preparation will be performed. Neat PG 52-34 will be used as the base binder. This binder was selected because it is commonly used in cold regions and the northern United States. Types and contents of additives are selected based on previous studies. Several modified asphalt binders will be produced with various additives fractions, then subjected to various short term and long-term conditioning regimes prior to performance testing to evaluate improvement in asphalt binder properties. In this second phase, two softening agents (Sylvaroad and Corn oil) will be



utilized, and three types of polymers will be selected; Epoxy, Styrene-Butadiene-Styrene (SBS) and Styrene-Butadiene-Rubber (SBR).

Task 2: Conduct performance testing. In this task, several asphalt binder performance testing will be conducted. The performance grade of all binders will be determined according to AASHTO M320. Dynamic Shear Rheometer (DSR) and Bending Beam Rheometer (BBR) will be used to grade all the asphalt binders in accordance with AASHTO M320 and investigate the impact of additives on the performance grading. In addition to that, the Bending Beam Rheometer (BBR) will be a means to evaluate the creep stiffness in accordance with AASHTO T313. The rutting performance of asphalt binders will be characterized by means of the Multiple Stress Creep Recovery (MSCR) testing. This performance testing will be conducted according to AASHTO T350, and performance will be evaluated through the non-recoverable creep compliance and percent recovery parameters at 3.2KPa. Moreover, the rheological properties will be investigated using a frequency sweep test performed with the Dynamic Shear Rheometer (DSR).

Task 3: Discussion and analysis of the performance testing results. This task is important to gain an understanding of the improvement in properties of asphalt binder imparted by softening agents combined with polymers. This task is important to gain an understanding of the improvement in the properties of asphalt binder imparted by these combinations. The evaluation of asphalt binders' properties will be conducted through the analysis of the performance testing outputs. In this task, the ANOVA analysis will be performed for some asphalt binder testing.

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Task 4: Summary, conclusion, and recommendations. In this section, based on the performance testing, results will be summarized, and conclusions and recommendations will be drawn concerning in order to select the best additive and exclude the additives that may not show good performance.

Significance of Study

This study is critical in developing innovative asphalt binder material using appropriate modifiers, evaluate and determine the effectiveness of softening agents and polymer additives in asphalt modification. The newly modified asphalt binders should resist cracking and rutting, especially in cold regions, without compromising fatigue performance. If such material is found to be successful, the field of civil engineering will benefit from the following advantages:

- Construct sustainable infrastructure through designing materials that improve the long-term performance of pavement systems.
- Minimize the cost of maintenance and rehabilitation of asphalt pavements.
- Achieve growth in the infrastructure industry.



Chapter 2

Literature Review

Introduction

Asphalt binder is a co-product of the petroleum-refining system and is considered an essential component of asphalt mixtures since it holds the aggregates together. Asphalt binder has proven to be a valuable material for flexible pavement construction for over 100 years. However, asphalt binders present an exceptional and complicated rheological behavior that varies from viscous to elastic depending on temperatures and loading times. This behavior can affect pavement performance and cause different distresses such as rutting (permanent deformation), thermal fatigue, stress fatigue, and aging. To overcome these challenges, in the beginning, the industry considered controlling the refining process of asphalt and selected an appropriate crude, but this was not enough. In fact, there exist few crudes that make good asphalt and the refining process was still unable to produce good quality. Thus, asphalt modification alternative has been taken in order to enhance binder quality over the last decades.

In this chapter, the results of a comprehensive literature review pertaining to asphalt modification are presented. The following subsections provide information relevant to the reasons behind asphalt modification, types of additives used in asphalt modification, methods utilized to modify asphalt binder, current grading systems and laboratory and field performance of modified asphalt binders.



History of Asphalt Binder Specifications

D'Angelo et al. (2009) reported that with increased traffic load on highways, pavement engineers had to work on developing mix design methods that considers selecting cost-effective materials capable of producing good performing asphalt mixtures, thus, pavements' service life would increase. As new materials have been increasingly used such as polymer modified asphalt binders, the empirical system that has worked relatively well in the past is no longer effective. In addition, there has been a tremendous focus on developing new binder testing procedures over the past years. Highway agencies shifted their developed relationships between asphalt material properties and performance from empirical based system to rheological based binder specifications. Consequently, these improvements in specifications led to an enhancement in pavements performance.

Chattaraj. (2011) indicated that Bowen penetration Machine invented in 1888 by B.C Bowen is the original version of today's penetrometer and the evolution of chewing's grading procedure. Originally, the grading temperature was +37°C. After several improvements of the penetration machine, the consistency of asphalt became measured and controlled at 25°C, which is the average ambient temperature in a year.

Early in 1960, a new grading system was developed and is based on measuring the viscosity at 60°C, which simulates the maximum pavement temperature in summer. This change in grading system was achieved to implement a rational scientific viscosity testing as an alternative to empirical penetration testing: the viscosity grading system allowed to measure the consistency at 60°C rather than 25°C, which simulates rutting occurrence.



In 1987, a new procedure was developed by Strategic Highway Research Program (SHRP), called Superpave performance grading system, which relies on engineering principals to address asphalt pavements distresses (Chattaraj, 2011). In fact, the Superpave asphalt design procedure focuses on evaluating the aged binder stiffness for a specific combination of climatic conditions and traffic loads. The designation of asphalt binders is based on environmental conditions (low or high temperatures) which are delineated by an increment of 6°C. These conditions are based on the average seven-day maximum pavement design temperature and minimum pavement design temperature (Kennedy et al. 1994).

The designation of PG X – Y (i.e., PG 64-22) represents the performance grades label or PGs as such:

Where,

PG stands for Performance Graded,

X: average 7-day high temperature, and

Y: the minimum pavement design temperature (Goliapour et al., 2011).

In the AASHTO M320, each test is presented in columns indicating the required engineering properties corresponding to a temperature and aging level for asphalt binders. The Superpave specification aimed to characterize rheological properties of asphalt binders by means of time-temperature superposition principles (TTS). For instance, at high



temperatures of testing, the phase angle (δ) and complex modulus G* measured at 10 radians are combined (the modulus divided by the sin of the phase angle G*/sin (δ)). The greater the G* value is, the stiffer the material and the more resistant to permanent deformation (D'Angelo, 2009). Concerning the low temperature characterization, this method grades the low temperature performance of binders using low temperature creep stiffness (S (t)) and rate of modulus relaxation (m-value) measured with the Bending Beam Rheometer (BBR). These parameters are obtained at relatively low stress strain levels within the linear viscoelastic range of asphalt binder. However, in-service pavements could be subjected to higher strain levels which represents a limitation for BBR testing method to characterize the low temperature properties (Johnson and Hesp,2014; Hesp and Shurvell 2012; Velasquez and Bahia, 2011).

On the other hand, AASHTO M320 specification provides criteria for selecting and specifying asphalt binders based on their laboratory performance. However, this specification was developed using asphalt binders that were commonly used in the late 1980s to early 1990s and didn't include polymer modified binders. The usage of asphalt modified binder has highlighted limitations in the AASHTO M320 parameters. Thus, to address these shortcomings, the multiple stress creep and recovery (MSCR) test (AASHTO T 350 and ASTM D7405) was developed to evaluate rutting susceptibility. Then, AASHTO M332 was developed to specify the performance graded asphalt binder (Salim et al 2019).



Asphalt Binder Polymer Modification

To further improve the properties of asphalt binders and ultimately the performance of flexible pavements, researchers have extensively evaluated various asphalt binder modifiers including, but not limited to: Styrene Butadiene Styrene (SBS) polymers, Ground Tire Rubber (GTR), Nanomaterials, warm mix additives.

Polymers are the most commonly used asphalt binder modifiers. Several research studies focused on evaluating the long-term performance of modified asphalt binders. For instance, Cardone et al (2014) investigated the influence of polymer modification on dynamic and steady flow viscosities of asphalt binders at high temperatures. Two polymers were considered: (a) Plastomer of Polyolefin, PO, and (b) SBS at three different dosages (2, 4, and 6%) by binder weight) to modify a Penetration Grade 70/100 binder. Cardone et al. (2014) reported that polymer nature and content significantly influence the rheological properties of modified binders. The use of polymers also led to increased stiffness, lowered phase angle, and decreased temperature susceptibility of modified binders (Cardone et al, 2014). In another study, Saboo and Kumar (2016) evaluated the rutting susceptibility of asphalt mixtures prepared using binders modified with SBS (at 3% by weight) or ethylene vinyl acetate (EVA at 5% by weight). Based on testing results, modified binders were found to result in more rutting resistant asphalt mixtures. Zhang et al. (2017) also assessed the high temperature properties of asphalt binders modified with 1% of SBS (by weight) mixed with bio-oil. The findings of the study of Zhang et al. (2017) showed that SBS-bio binder had more viscous characteristics and lower rutting susceptibility than neat asphalt binder.



In a recent study, Benhood et al. (2017) investigated the rheological properties of asphalt binders modified using SBS, ground tire rubber (GTR), or polyphosphoric acid (PPA). The authors found that all of the modifiers improved the high temperature properties of neat asphalt binders. Based on performance testing results, GTR significantly lowered the stiffness of binders at intermediate temperatures, in comparison to other modifiers. All three modifiers did not have a significant impact on low temperature grade of the neat binder (Benhood et al. 2017).

Sargand and Kim (2001) studied the fatigue and rutting resistance of PG 70–22 modified binders, one unmodified, one SBR modified, and one SBS modified. It was concluded that the incorporation of modifiers improved both fatigue and rutting performance compared to neat binder despite their same performance grade.

Styrene Butadiene Styrene (SBS). The SBS is the most common modifier due to its good dispersibility in bitumen, excellent properties and acceptable cost. (Lu and ISACSSON, 1997; Chen et al, 2002). Several studies (Valkering and Vonk, 1990; Krutz, et al 1991; Stock and Arand, 1993) concluded that the SBS modified asphalt binders showed improved performance in terms of cracking resistance at low temperatures, rutting resistance at high temperatures and elastic recovery.

Shukla et al (2003) investigated the use of SBS material in asphalt binder modification in India. The study results showed that in spite of the reduction of the asphalt layer of Delhi–Ambala expressway, its surface life would be almost doubled. Yet, when using polymer modified binders, the cost per km would be greater. Another study conducted by Greene et al (2014) showed that the asphalt mixture with high polymer



binder (PG 88-22) had greater fracture energy and less rut depth than PG 76-22 and PG 67-22 binders.

Andriescu et al. (2009) and Hesp et al. (2018) presented that a highly SBS polymer modified binder had significantly higher work of fracture at intermediate temperatures than traditional polymer binders based on Double-edge-notched tension (DENT) testing results indicating that highly modified binder could have higher fatigue properties.

Roque et al (2004) investigated the effect of SBS modifier on the performance of SuperpaveTM mixes. It was concluded that the SBS improved the cracking performance and healing characteristics due to its capability to reduce the rate of micro-damage accumulation.

Bowers et al (2018) investigated the cracking resistance of a 9.5-mm surface mixture with high polymer binder in Northern Virginia and found that high polymer mixture had a fatigue life approximately 40 to 50 times greater than that of the control mixture with PG 64-22 binder.

Airey et al. (2004) used six SBS modified asphalt binders originated from two crude sources at three different dosages to investigate the rheological properties of the asphalt binders. The dynamic shear rheometer (DSR) results indicated the source of asphalt binder, Polymer concentration and bitumen-polymer compatibility had an effect on the modification degree. It was found that the viscosity increased when the polymer concentration and binder-polymer compatibility allowed the establishment of a continuous polymer network. I addition to that, elastic response and stiffness increased



particularly at high service temperatures. However, the elastic response and molecular size of SBS copolymer decreased with aging.

Tim et al. (2012) concluded that that adding 7.5% SBS resulted in 45 times improvement in fatigue life compared to a control mixture with traditional SBS modified binder. Another study conducted by Farina et al. (2017) reported that, based on an internal industry review relating polymer modified binder in Europe that a typical SBS polymer content is around 3.5% by weight in the final product

Nanomaterials. Nanotechnology has been gradually incorporated into the field of asphalt modification. This technique offers the opportunity to develop new materials that have significant effects on improving asphalt binder properties. Several researchers have focused on assessing nano-modified asphalt binders in order to understand mechanisms of modification and the resulting performance enhancements (Zare-Shahabadi et al.2010; You et al.2011; Santagata et al.2012). For example, Al-Hdabi et al. (2019) studied the impact of nanomaterials added to asphalt mixtures utilized in road paving and investigated the feasibility of nanotechnology as a mechanism for improving asphalt mix characteristics. Results showed that Nano-carbon improves the properties of asphalt binders, which become more resistant to permanent deformation compared to regular asphalt mixtures prepared using unmodified asphalt binders. Another study conducted by De Melo et al. (2016) investigated the effect of various dosages of carbon nanotubes on the empirical and rheological properties of asphalt binders. This study also assessed the properties of asphalt binder mixtures prepared using the optimal binder content. The results showed that the optimum modifier content added is approximately 2%



by weight. This study also reported that carbon nanotubes presented a strong effect on the performance of asphalt mixtures in terms of resistance to permanent deformation. In a different study, Jahromi and Khodaii. (2009) found that the addition of Nano-clay had a significant effect on the rheological properties of asphalt binder. In fact, the stiffness of asphalt binders increased, while the phase angle decreased, which indicated that the aging properties of binders improved. Recently, Ashish et al. (2017) assessed the impact of organo-modified Nano-clay on rutting resistance, fatigue performance, and aging properties of asphalt binders. Results showed that with the addition of Nano-clay, the aging resistance of asphalt binders increased. Results showed that the rutting performance of the modified asphalt binders improved, indicated by the increase of the Superpave rutting parameter (G*/Sin δ) and the decrease in the non-recoverable creep compliance (J_{nr}). In addition, the fatigue resistance and high temperature performance grade of asphalt binders, modified with Nano-clay (CL-30B), seemed to be higher than that of the unmodified binder.

Other researchers studied the impact of nanosized asphalt binder modifiers on the engineering properties of asphalt binders. Goh et al. (2011) evaluated the impact on nanoclay and carbon microfiber modifiers on the indirect tensile strength (ITS) of asphalt mixtures. Goh et al. (2011) reported that the addition of Nano-clay would improve asphalt mixtures' resistance to moisture-induced damage. On the other hand, Amirkhanian et al. (2010) evaluated the high temperature rheological properties of asphalt binders modified with carbon nano-particles. Three base binders (PG 64-22, PG 64-16, and PG 52-28) were modified using carbon nano-particles at four different dosages (i.e., 0.0%, 0.5%, 1.0%, and



1.5% by binder weight). Based on rational viscometer and Dynamic Shear Rheometer (DSR) test results, Amirkhanian et al. (2010) reported that the addition of Nano-carbon particles increased viscosity, failure temperature, phase angle, and viscous and elastic modulus values; thus, potentially improving rutting in asphalt mixtures. Another study by Shafabakhsh et al. (2015) reported that adding Nano -Titanium Dioxide (TiO₂) and Nano-Silica (SiO₂) improved the rheological properties of base asphalt binders by 30% and 109%, respectively. Yao et al. (2012) also reported that using Nano-Silica enhanced antiaging properties of asphalt binders and improved asphalt mixtures' rutting and cracking resistance.

Styrene Butadiene Rubber (SBR). Usually used as a dispersion in water (Latex) has been widely used as a binder modifier. Bates and Worch. (1987) described the advantages of using SBR in bituminous concrete pavements and seal coat. This modifier has the ability to enhance low-temperature ductility, viscosity and elastic recovery. In addition to that it improves the adhesive and cohesive properties of the pavement.

Another study conducted by Becker et al. (2001) reported that the rubber particles, when exposed to asphalt during mixing had a rapid and uniform dispersion and form a reinforcing network structure. It was also found that SBR latex had a positive impact on asphalt pavement ductility.

Roque et al. (2004) concluded that the SBR modification enhanced the pavement flexibility and improved the low temperature cracking resistance. This modifier also helps solve hardening and aging issues thanks to its ability to increase elasticity, reduce the rate of oxidation and improve the adhesion and cohesion.



Kim et al (1999) found that using cement and SBR Latex for use in HMA to coat smooth rounded, siliceous gravel aggregates improved stability according to Hveem and Marshall Standards. In addition to that, the tensile strength, resilient modulus and resistance to moisture damage were greater. It was also found that coated aggregates had higher resistance to rutting and cracking. King et al (1999) reported that Elastomers such as SBR had a significant impact on the ductility which was higher for all temperatures compared to SBS modified asphalts.

Zhang et al. (2009) investigated the rheological, thermal and morphological properties of Natural Binder asphalt binder when modified by styrene butadiene rubber (SBR). The study results showed that the incorporation of 2% of NB resulted in high temperature properties improvement by increasing the softening point in SBR/NB modified bitumen. On the other hand, 3% of SBR in SBR/NB modified asphalt binders showed significant impact on the aging properties and the low temperature and resistance. Zhang et al (2009) also reported that compatibility and thermal properties were improved with a homogeneous and stable mix structure in modified bitumen. In addition to that FTIR analysis showed few new weak peaks for modified asphalt binders indicating that physical alteration is the main changes in the modified asphalt binder.

Rubber. Ground Tire Rubber (GTR) is a type of polymer originated from vehicle and small truck tires, has been commonly used in modifying asphalt binder for paving mixtures worldwide in the last decades. It has been proven that adding crumb rubber to asphalt mixtures improved the rutting performance, thermal cracking resistance and durability (Shu et al, 2014). The use of crumb rubber polymer with asphalt binders


seems to improve the fatigue resistance, as indicated in several studies (Raad and Saboundjian.(1998); McGennis(1995); Soleymani et al.(2004); Billiter et al. (1997). Several techniques are adopted to incorporate rubber into asphalt pavements; wet process, dry process, and terminal blend process. The dry process consists of adding larger size particles (4 to 18 mesh) directly into the asphalt mixture similar to reclaimed asphalt pavement (RAP) at the mixture production plant. Concerning the wet process, the percentage of Ground Tire Rubber is 15-22% by weight of asphalt binder and rubber mixture is typically field blended at 350 to 400°F for about 45 to 60 minutes. The terminal blend process consists of blending 5-10% smaller GTR particles (<0.6mm) and polymers to produce a rubberized asphalt binder comparable to standard polymer modified asphalt binder (Federal Highway Administration, 2014). Several researchers have used rubber to enhance asphalt pavements performance. For instance, George et al. (2009) reported that rubberized binders in Arizona decreased reflective cracking and enhanced rutting resistance and smoothness. In addition to that, it was also indicated that using rubber showed less average maintenance cost. Subhy et al. (2016) evaluated the potential of using pre-treated tire rubber for replacing SBS polymer modifiers. The researchers reported that using pre-treated tire rubber significantly reduced the high temperature viscosity of modified binders; indicating better handling, wetting of aggregates, and reduced mixing and compaction temperatures (Subhi et al, 2015). Another study conducted by Yildirim (2007) reported that modifying asphalt with tire rubber environmentally friendly. It was also indicated that tire rubber decreased rutting and reflective cracking. However, some issues can occur during special conditions such as



long absorption times high mixing temperatures. These conditions need to be maintained to prevent rubber separation from the asphalt binder. Turgeon 1989 reported that using 20% rubberized binder in wear courses and the rubberized pavements exhibited less cracking (Turgeon, 1989).

Hainin et al. (2015) reported that adding tire rubber to the asphalt binder improves the properties of modified binder. It was found with increasing the percentage of tire rubber, the rutting factor (G*/sin\delta) increases fatigue factor (G*sin\delta) decreases leading to better rutting and cracking performance. In this study, it was also indicated that tire rubber powder is a solution to improve environmental and financial sustainability of pavements. Another study conducted by Sousa et al. (2013) reported that Reacted and Activated Rubber (RAR) is used as asphalt modifier .In addition to that it was indicated that asphalt mixes with RAR were stronger, more resilient, and exhibited better recovery, rutting, and fatigue resistance compared to conventional asphalt rubber mixes.

Additionally, a principal advantage of RAR is that it can be added easily to any hotmix asphalt manufacturing facility with systems designed to feed particulate material into a batch plant (pugmill) or drum mix plant. Lehigh technologies reported that using 10% 40 mesh GTR and 1% Rheopave XP10 in base binder (PG 64-22) can produce highly elastic binder (PG 88-22). Rheopave XP10 is a blend of selective polymers and other additives designed specifically to enhance the performance of GTR in RMA binders. XP10 can improve MSCR performance (higher % recovery) and storage stability.

Wang et al. (2012) evaluated the viscosity properties and low temperature performance of a rubber modified PG64-22 asphalt binder. Two crumb rubber sized particles (fine and



coarse) were added at dosage rates of 10, 15, 20 and 25% crumb. It was reported that the crumb rubber significantly enhanced asphalt binder viscosity and low temperature performance. Furthermore, finer size crumb rubber had better performance in the modification. It was also indicated that 20% and 25% ratio CRM binders didn't show a significant performance difference.

Epoxy. In 1967, epoxy-asphalt mixture was used in the San Francisco Bay on San Mateo-Hayward Bridge and nowadays it is gradually used in the steel deck pavement (Herrington and Alabaster, 2008).

Epoxy modified asphalt binders have enhanced mechanical properties and high temperature stability than virgin ones (Herrington et al.2007; Huang et al 2003).

Peiliang et al. (2010) studied the effects of epoxy resin contents on rheological properties of asphalt binders. It was reported that the epoxy resin improves the heat resistance and strength of asphalt binders. Results showed that adding 20 % of epoxy resin by weight of asphalt binder lead to higher complex modulus value compared to the original binder. In addition to that, the epoxy modifier affected the phase angle, and the higher the epoxy dosages the higher the effect. Results also showed that epoxy resin also enhances the recovery from strain and reduces temperature sensitivity of asphalt.

Cubuk et al. (2009) investigated the effect of epoxy on 50/70 penetration grade asphalt binder. It was reported that adding 2% of epoxy by weight of asphalt binder yielded the greatest rheological and performance properties. In addition to that, the study results showed that epoxy addition could decrease rutting, bleeding, cracking and stripping. It was also indicated that epoxy modified asphalt binder can be recommended for hot regions and



humid climates. It can also be used when the traffic loading is heavy, at road curves and at bus stations.

Apostolidis et al. (2019) evaluated the epoxy modification in asphalt binder. It was reported that the temperature impacts on the development of mechanical and physicochemical characteristics during curing and aging of epoxy-modified asphalt binder. In addition to that, the degree of aging extent is related to the level of epoxy modification. In this study, it was indicated that adding epoxy to the modifier lead to enhanced mechanical characteristics such as higher tensile strength, longevity and flexibility.

Biomodified asphalt binders. In recent years, several studies focused on substituting or modifying the traditional asphalt binder. A potential alternative is the bio binder (Chailleux et al., 2012; Chaiya et al., 2011) and bio-oils have been used to modify petroleum asphalt used in flexible pavements. (You et al, 2011, Mills-Beale et al. 2012). Mogawer et al. (2016) assessed the effect of using a blend of polymer and rejuvenators in high reclaimed asphalt pavement mixtures. In this study, it was reported that combination of an asphalt rejuvenator and a PMA binder can produce a high RAP (50%) mixture with comparable or better performance than a similar conventional mixture. It was also reported that using PolyRejuvenated[™] can design a greater mixture which has much superior resistance to cracking without sacrificing rutting resistance.

Yang et al. (2013) investigated the performance of a PG52-28 asphalt binder partially substituted by a waste wood fast pyrolysis derived bio-oil. Three additives were introduced to the neat asphalt binder; the original bio-oil (OB), de-watered bio-oil (DWB) and polyethylene modified bio-oil (PMB) at 5% and 10% by weight. The study showed the



high temperature performance of asphalt binder was increased by the addition of bio-oil by increasing the $|G^*|/\sin\delta$ parameter. In addition to that, it reduced the mixing temperature. However, it had negative effect on the low and medium temperature performance. Results also indicated that the polyethylene modified bio oil had the highest stiffness followed by the DWB and OB modified binder. Another Study conducted by Sun et al. (2017) investigated the effect of bio-oil addition on asphalt binder performance. It was found that adding bio-oil decreased the deformation resistance and elastic recovery performance of control asphalt at medium and high temperatures. Meanwhile, at low temperatures, bio-oil improved stress relaxation property and thermal cracking performance of control asphalt.

Xiaoyang et al. (2014) evaluated the engineering properties of asphalt binder modified with waste engine oil residues. The study indicated that the addition of up to 5% of waste engine oil significantly transformed the infrared ranges and rheological properties of asphalt binder, which can lead to the enhancement of low temperature performance.

Laboratory Performance of Modified Asphalt Binders

Rotational viscometer. Hassanpour-Kasanagh et al. (2020) investigated the Time- and temperature-dependent properties of SBS and CM modified binders at high and intermediate temperature. The Rotational viscosity (RV) on unaged binders was carried out at 135°C and 16 °C according to AASHTO T-316. The RV values of the binder increases as the percentage of modifiers increases. This is also evident from the viscosity ratios of modified binders to base binder. The results show that although both modifiers significantly increase the viscosity of the binders; at the same percentage of modifier, the increase in the viscosity values of SBS-modified binders is more than that



of the binders modified with CM. For instance, at 135 C, the binder modified with 7% SBS shows 419% increase in RV value while the binder modified with 7% CM shows 306% increase in RV value.

Zhang et al. (2017) conducted a study aiming to enhance the high temperature performance of bio-asphalt by adding 1 % of Styrene-butadiene-Styrene (SBS) by weight of a total 50 penetration grade binder. The bio-oil dosages for the five types of binders were 0%, 5%, 10%, 15% and 20% of total binder by weight. In this research, the rotational Viscometer testing was conducted on SBS modified bio-asphalts by the Brookfield Rotational Viscometer following AASHTO Designation: T 316-13. The testing temperatures were 90 C, 135 C and 175 C, and the shear rates were 10 r/min, 20 r/min and 50 r/min, respectively. Results showed that the SBS modifier increased the viscosity of the bio-asphalt. However, when the bio-oil content increased, the viscosity of SBS modified bio-binder decreased. In addition to that, the mixing and compaction temperatures of SBS modified bio-asphalt were increased by the addition of SBS as expected. Yet, the increase in bio-oil decreased the temperatures which decreased with the addition in bio-oil content. The viscometer testing results also showed that, when the bio-oil content was more than 10%, the mixing and compaction temperatures remained the same compared to a 50penetration grade base binder. Wang et al. (2012) added crumb rubber to a Superpave PG64-22 asphalt binder at a dosage rate of 10, 15, 20 and 25% by weight of binder. The modified binders were produced by introducing crumb rubber progressively into the asphalt binder at 350 °F (177 °C) and mixed mechanically for about 45 minutes. In this study. In this study the viscosities of non- aged and RTFO aged CRM asphalt binders were



characterized by the Brookfield viscometer according to AASHTO T315 at 135, 140, 150, 160, 170, 177, and 190 °C. In this viscosity testing, #29 spindle was adopted since the CRM have high viscosities, the applied torque was 25% and the rotation speed was 100 rpm. Results indicated that crumb rubber can significantly increase asphalt binder viscosity. This means that crumb rubber improves the high temperature performance of asphalt binders and mixtures. The viscosity specification requirement of 3 Pa s is however not feasible for high percent CRM binder.

Bending Beam Rheometer. The Bending Beam Rheometer (BBR) test which was suggested by Superpave specification was widely applied to evaluate the lowtemperature properties of binder (Ghavibazoo, and Abdelrahman 2014). Two parameters which were creep stiffness and m-value could be obtained by BBR test. The creep stiffness was represented to resist constant loading of the binder and the m-value was represented to measure the rate change of asphalt stiffness as the loads were applied (Wang et al 2012).

The creep stiffness of the binder at any time (t) was calculated by the following equation (Liu et al 2010):

$$S(t) = \frac{PL^3}{4bh^3\delta(t)}$$
(1)

Where,

S(t) = creep stiffness (MPa) at any time t;

P = applied constant load (N);



L = distance between beam supports (102 mm);

b = beam width (12.5 mm);

h = beam thickness (6.25 mm);

And $\delta t = deflection (mm)$ at time t.

In order to avoid cracks in the pavement at very low service temperatures, the maximum value of creep stiffness should not exceed 300 MPa, while the minimum value of m-value was not less than 0.3. The decrease of creep stiffness made the tensile stress in the binders smaller, so as to reduce the probability of cracking at low temperatures (Kök, B. V et al 2013).

Wang et al. (2012) investigated the low temperature creep stiffness PG 64–22 asphalt binders modified with Crumb rubber with the following dosages (0%, 10%, 15%, 20% and 25% by weight of asphalt). In this study, two Crumb rubber types cryogenically manufactured from different sources in China were used. The low temperature stiffness of crumb modified asphalt binders was assessed at -12 and -18 °C using the Bending Beam Rheometer BBR equipment according to the AASHTO T 313 standard specification. Results showed that crumb rubber could decrease the creep stiffness of CRM asphalt binder at low temperature which means better cracking resistance. In addition to that, 10% crumb rubber could increase a low temperature grade from -22 °C to -28 °C. Another study conducted by Billiter et al. (1997) studied the several physical properties of crumb rubber modified asphalt binder including low temperature cracking resistance using the Bending



Beam Rheometer (BBR) test equipment according to the AASHTO T313 standard test specification. It this study, results indicated that crumb rubber had the capacity of keeping decent elasticity at low temperatures. Hence, crumb rubber improved the flexibility of asphalt by behaving as an elastic material in cold conditions, which improved the low temperature cracking resistance of asphalt binder. Shen et al. (2005) evaluated the low temperature properties of two CRM asphalt binders and one control binder of PG76-22. The three binders were used as recycled materials after an artificial aging by adding different rejuvenating agents, i.e. a rejuvenator and a softer binder. The low temperature properties of the two aged CRM asphalt binders and the aged control (PG76-22) were evaluated using the Bending Beam Rheometer (BBR) test equipment in accordance to the AASHTO T313-02 at -12°C and -18°C. The BBR findings indicated that adding a rejuvenator to a Crumb rubber modified asphalt binder resulted in lower creep stiffness and higher m-value compared with the control crumb rubber modified asphalt binder. This indicates that the CRMB had better low temperature cracking resistance when blended with a rejuvenator.

Dynamic Shear Rheometer . The Dynamic Shear Rheometer is commonly used to describe the viscous and elastic performance of asphalt binders at medium to high temperatures. It is also used in the Superpave PG asphalt binder specification. This testing uses a thin asphalt binder sample inserted between two circular plates. (Hefer, 2005; Hafez and Witzack, 1994; Yang et al 2003).

Zhang et al. (2017) assessed the visco-elastic properties and evaluated the antirutting performance of SBS modified bio-asphalt using the Dynamic Shear Rheometer



(DSR) according to AASHTO T315. The dynamic shear modulus and phase angle were obtained using DSR tests sweep temperatures and frequencies on unaged and RTFO-aged samples. Results showed that before the RTFO aging, the SBS modifier enhanced the rutting resistance of bio-asphalts. However, the increase in bio-oil content (more than 10%) decreased the ability of asphalt to resist rutting. It was also found that after RTFO aging and the same temperature, the resistance to rutting of SBS modified bio-asphalt was stronger than that of the base binder and grew with the increase in bio-oil content. In addition to that, the temperature sensitivity analysis showed that SBS modified bio-asphalt before RTFO aging is less temperature sensitive than the neat binder, and with the increase in bio-oil content, such sensitivity decreased. After RTFO aging, the temperature sensitivity of SBS modified bio-asphalt was still lower than that of the base binder when the bio-oil content was less than 20%, and it increased with the increase in bio-oil content.

Frequency sweep testing for rheological characterization. The Dynamic Shear Rheometer can be used to evaluate the rheological response of asphalt binders using the frequency sweep testing. During the testing, an oscillatory shear loading at constant amplitude over a range of loading frequencies is applied. For instance , in a study conducted by Moreno-Navarro and Rubio-Gamez (2015) ,the frequency sweep was carried out on four different asphalt binders types modified with SBS at various dosages (0, 2, 4, 6 % of total binder weight) .The amplitude of 0.1 % strain and a range of frequencies from 0,1 Hz to 20 Hz were applied at 10, 20, 30, 40, 45, 52, 58, 64, 70, 80 C. Black space diagram results indicated that using SBS as a modifier to asphalt binder improved the elasticity and consistency at high temperatures which means better



resistance to plastic deformation. In these diagrams higher phase angles and lower complex modulus are associated to more flexible materials (Viscous) whereas lower phase angle and higher complex modulus are associated to more rigid materials (elastic).

The master curve of the AC characteristic at a reference temperature T_{ref} is defined as the relation between the complex modulus and the reduced loading time or frequency (Walubita et al, 2011).This type of curves is constructed based on DSR measured data and requires a shift relative to the loading time or frequency and allows to summarize all the various curves representing the response at numerous temperatures to a single curve known as the master curve (Marasteanu et al 1996 ; Soleymani et al 1999).There exist seven various shifting methods, namely the numerical, non-functional shift approach, the Arrhenius equation, the modified Kaelble equation, Laboratoire Central des Ponts et Chaussées (LCPC) approach , the viscosity–temperature-susceptibility (VTS) equation ,the Williams–Landel–Ferry (WLF) equation, and a log–linear approach (Yusoff et al ,2011). The master curves were constructed using the Christensen-Anderson model (Christensen et al, 1999; Turner et al.2015).

$$G^{*}(\omega) = G_{g} [1 + (\omega_{c} / \omega_{r})^{(\log 2)/R}]^{-R/(\log 2)}$$
(2)

$$\delta(\omega) = 90/\left[1 + (\omega_{c}/\omega_{r}) \left(\frac{\log 2}{R}\right]\right]$$
(3)

 $G^*(\omega)$ = absolute value of complex modulus at frequency ω , (Pa). Gg = glassy modulus, (Pa)

 ω r = reduced frequency at the reference temperature, (rad/sec)

 ω_{c} = crossover frequency at the reference temperature, (rad/sec).



R = rheological index or shape parameter

and $\delta(\omega)$ = phase angle at frequency ω . (°)

Jahromi and Khodai. (2009) evaluated the effects of Nano-clay on rheological properties asphalt binder. In this study, DSR measurements were carried out over a wide range of temperatures (15 and 100 C) and loading frequencies (0.015–20 Hz). The thickness of the bitumen is selected 2 mm for the 10 mm diameter plate and 1 mm for the 30- and 40-mm plates. The master curve of the modulus/phase angle defines the frequency (time) dependency of the material. In this study the master curves were constructed the theory of Williams–Landel–Ferry (WLF) at a reference temperature 20 C. The complex modulus (G*) increases by decreasing temperature and/or increasing frequency. However, the phase angle increases as the temperature increases and/or the frequency decreases. Results showed that Nano-clay had an impact rheological properties of asphalt binders by increasing stiffness and decreasing the phase angle. It was also reported that this modifier improves ageing resistances, as well.

Another study conducted by Apostolidis et al. (2019) assessed the viscoelasticity behavior of epoxy modified asphalt binder which using the frequency-dependent material properties (i.e., complex shear modulus and phase angle). The isothermal frequency sweep measurements were conducted using a dynamic shear rheometer (DSR, Anton Paar, EC Twist 502) at a range of temperatures from 10 to 60 C Hz at temperature steps of 10 C and loading frequencies from of 0.1– 10 Hz. For temperature below 20, plates of 8-mm diameter with a 2-m gap were used whereas plates of 25-mm diameter with a 1-mm sample gap were used at temperature above 30 C. The properties were measured at frequencies of



0.1-10 from 10 to 60 C. was placed onto the bottom plate at the desired test temperature (±0.1 C). The master Curves were constructed using the time-temperature superposition model at a reference temperature of 30 C. Results showed that at relatively low frequencies, the additive had insignificant impact on the phase angle. At intermediate frequencies, the existence of phase angle plateaus specifies the epoxy molecular networks in asphalt binders. It was also indicated that epoxy improved the elasticity of asphalt binders. Moreover, when the hardening occurred, the material behaved more glassy, due to the dominance of modifier in EB50.Another finding was that the phase angle values are more sensitive to chemical changes than modulus.

Multiple Stress Creep and Recovery Test. Several Studies evaluated the rutting performance of modified binders using the Multiple Creep Recovery Testing. Arshad et al. (2017) assessed the impact of Nano-Silica (NS) concentration on the rutting performance asphalt binder using the multiple stress creep and recovery (MSCR) test. The dosages rates of Nano-Silica were between 1% to 5% (1% increment). The Nano-Silica modified asphalt binder (NSMB) were aged using rolling thin film oven (RTFO) before tested. The MSCR test was conducted at 64°C on RTFO Nano-Silica modified asphalt binders with two stress level (100 Pa and 3200 Pa). The results indicated that accumulative strain of NSMB decreased by adding the modifiers. In addition to that non-recoverable creep compliance (J_{nr}) decreased and recovery strain increased (R), which indicates an enhancement of rutting resistance and elasticity of the binder, respectively. In this study, The MSCR testing was also used to grade the asphalt binder and results showed an improvement from heavy (H) grade to extreme (E) grade. It was concluded



that 2% of Nano-Silica is the optimum dosage as it showed the best enhancement in terms of Jnr and %R. A study carried out by Moreno-Navarro and Rubio-Gamez (2015) used the Multiple Stress Creep Recovery (MSCR) in order to assess the mechanical properties of asphalt binders at high temperatures. The testing was carried out according to AASHTO TP70 at 45, 60 ,70 °C. In this testing the non-recoverable creep compliance measure is a parameter of permanent deformation characterization. It was reported that the use of polymers could enhance the rutting performance of asphalt binder.

Fatigue cracking laboratory performance testing. Current asphalt binder specification used to characterize the fatigue performance of asphalt binders lacks the ability to describe the damage resistance, and therefore, Viscoelastic Continuum Damage models cannot be applied directly to these specification test results. During the last decade, several researchers had been working on developing new methods to characterize fatigue cracking of asphalt binders such as Linear Amplitude (LAS) test. This testing had been used in numerous researches. However, Hintz and Bahia (2013) reported that the linear amplitude sweep (LAS) test is considered a temporary standard and is presently being considered for specification of asphalt binder fatigue resistance. Hintz et al. (2011) indicated that LAS testing protocol provides promising results. However, the time and the complex numerical procedures required for the analysis have raised concern. In addition, insufficient damage accumulation was observed when the strain amplitudes proposed in the LAS test were used for a set of polymer-modified binders

In the proposed procedure, strain amplitudes from 0.1% to 20% are used. However, some asphalt binders exhibit little damage under this procedure.



Wang et al. (2015) developed a modified Linear Amplitude Sweep Testing includes additional two amplitude sweep tests with standard strain range of 30% are performed within 600s and 900s, and the failure point is defined as the peak in stored pseudostrain energy (PSE). After testing, the test data are analyzed and fatigue lives at different strains are predicted using the viscoelastic continuum damage (VECD) mechanics approach.

Another study carried out by Hintz et al. adopted the Linear amplitude sweep testing (LAS) and conducted it on eight asphalt binders using an Anton Paar SmartPave DSR. All tests were performed at the intermediate-temperature PG of the asphalt binder after rolling thin film oven aging. In this study, the undamaged properties of the asphalt binders were obtained using the frequency sweep tests, which were conducted at 0.1%strain and a range of frequencies from 0.1 to 30 Hz. Afterward, an amplitude sweep testing was performed, and 100 cycles were initially applied at 0.1% strain. After this step, each successive load step consisted of 100 cycles at a rate of increase of 1% applied strain per step for 30 steps, starting at 1% and ending at 30% applied strain. Ameri et al. (2017) investigated the fatigue behavior of modified PG85-100 asphalt with styrene-butadienestyrene (SBS) and crumb rubber (CR). In this study the Linear amplitude sweep tests were performed to evaluate the fatigue properties of asphalt binders. The testing was carried out under different loads based on the concepts of viscoelastic continuum damage mechanics. It was found that the addition of Crumb Rubber and Styrene Butadiene Styrene could improve the fatigue life of modified asphalt binders based viscoelastic continuum damage analysis.



LAS is an accelerated test to evaluate the fatigue life of asphalt binders. Pressure ageing vessel (PAV)–aged samples were used in the LAS test using 8-mm-diameter spindle and 2-mm gap. The LAS test was performed in accordance to AASHTO TP101.

Shafabakhsh and Rajabi (2019) investigated the effect of SBS polymer, Nano-Silica, and SBS/ Nano-Silica, nano-composite on the fatigue properties of asphalt using the Linear amplitude sweep (LAS) test according to AASHTO TP 101-12. In this study, results were analyzed by means of the viscoelastic continuum damage model. It was reported that the fatigue resistance of asphalt binder was greater when adding SBS/ Nano-Silica nanocomposite. In addition to that SBS/ Nano-Silica nano-composite with 6% Nano-Silica and 5% SBS exhibited the best fatigue performance.

Safaei and Hintz (2014) assessed the impact of temperature on the fatigue performance PG64-22, PG70-22 and PG70-34 asphalt binder using a TA ARG2 DSR with an 8 mm parallel plate-plate set-up. The tests were in the strain control mode. Prior to testing, all asphalt binders were aged in the Rolling Thin Film Oven (RTFO) prior to testing. In order to determine the linear viscoelastic characteristics of, the frequency sweep was performed at a constant load amplitude of 1% strain and a range of frequencies from 0.1 to 30 Hz. Tests temperature selected were 50, 35, 20 and 5°C for all asphalt binders. The master curves were constructed from frequency sweep results and the Williams Landel Ferry (WLF) theory. In addition to that, the time sweep tests were performed to evaluate the fatigue behavior of asphalt binders. Tests were conducted at 5, 10, 15, 20, 25, and 35°C for all binders. Time sweep tests were all conducted in displacement-controlled model at 10Hz loading frequency and 3% initial strain amplitude. In this study Results were



examined using the Simplified Viscoelastic Continuum Damage (VECD) analysis. It was indicated that Results show that binder PG70-34 asphalt binder had greater fatigue life compared to other binders compared to other binders regardless of temperature, which is expected since it is a highly polymer modified asphalt binder. The study findings reported that fatigue behavior is related to the temperature and binder type.

Summary of the literature review. Numerous studies proved that the use of polymers and softening agents are a successful means to improving the asphalt binder performance in terms of rutting and cracking. In addition to that asphalt modification could prevent stripping, temperature susceptibility and increase viscosity and recovery. However, the compatibility between asphalt binder and additives should be taken into consideration, else the pavement will exhibit poor performance. It is also important to select the appropriate asphalt binder testing to characterize each distress properly since the Superpave specification seemed to be inapplicable for certain modified binders. Each additive impacts the performance of asphalt binder differently. Several researchers confirmed that the binder modification is a candidate approach to obtain highly elastic binder that exhibited good performance at high and intermediate temperatures. In addition to that, most researches focused asphalt binders utilized in hot and warm regions. However, very limited studies encountered the use of additives to improve the performance of asphalt binders used in cold regions.



Chapter 3

Materials and Experimental Methods

Overview of the Laboratory Experimental Program

The laboratory experimental program was established in order to evaluate rutting susceptibility and cracking resistance of modified asphalt binders. The goal of the performance testing was to determine the best performing additives and recommended dosages for asphalt binders used in cold regions. This section discusses the modification methodology and performance evaluation program.

Materials and Modification Methodology

In this study, two soft Performance Graded (PG) asphalt binders: neat PG 52-34, and polymer modified binder (PMB) PG 64E-40 commonly used in cold regions and northern United States were selected. In addition to that, various additives were used for binder modification; Nano- TiO₂ and SiO₂, SBS, GTR, Epoxy, SBR, Corn oil, and SYLVAROAD were selected to modify neat binder PG 52-34. On the other hand, GTR, Nano-SiO₂, Nano-TiO₂ were used to modify PG 64E-40. For PG64E-40 binder, no SBS or other softening agents were added because it already includes SBS. It is also noted that 0.1% of sulfur powder by weight of binders was added to both asphalt binder when modified with SBS in order to reduce the separation between base binder and additives. For GTR modified binders 1% crosslink by weight of GTR was added. Based on the supplier recommendation, the softening agent and SBS modified binders were developed



using a low shear mixer first to mix 7% of softening agent with the unaged PG52-34 asphalt binder.

The epoxy-based modifier is a product supplied by ChemCo Systems and includes two liquid constituents free from solvents; Part A and Part B. The first part is an epoxy resin including epichlorohydrin and bisphenol-A and the second part is a blend of fatty acid hardening agent and 70 pen bitumen). The epoxide groups of part A react with the monomers of part B in the bitumen. This reaction allows the production of covalent bonds and the polymerization process allows the molecular chains crosslinking.

According to the supplier recommendations, the softening agent and epoxy modified binders were developed by mixing part A and B at weight ratio of 20:80. The two component A and B were oven heated for 1 hour at to 185 °F and 230 °F respectively then mixed together for approximately 10–20 s. The last step consisted of mixing the epoxy modifier with an already pre-heated unaged PG52-34 asphalt binder at 295°F.For the other additives, the blending procedure consists of using a high speed shear mixer of 3000 rpm. In addition to that for all blending procedures except the epoxy and softening agent binders, a heating mantle was used to control the blending temperature. Table 1 shows the modification plan of the study and Table 2 shows blending temperature and time based on previous studies (Filonzi et al, 2018) and supplier's recommendations.



Table 1

Asphalt	Additives	Б		
binder		ID	Dosag	ge (%)
	No additives	-	-	
	Ground Tire Rubber	GTR	10	15
DC 52 24	Styrene Butadiene	CDC	2	7.5
PG52-34	Styrene	SBS	3	1.5
	Titanium dioxide	TiO ₂	3	5
	Silicon dioxide	SiO ₂	3	5
	No additives	-		-
PG64E-40	Ground Tire Rubber	GTR	5	10
	Titanium dioxide	TiO ₂	3	5
	Silicon dioxide	SiO ₂	3	5
Total number of combinations			1	6

Modification plan for polymer and nanomaterials modified asphalt binders



Table 2

Asphalt	Polymers	ID	Dosage	Softening	ID	Dosage
binder				agents		(%)
PG52-34	Epoxy	Е	25	Corn oil	С	
	Styrene Butadiene	SBS	7.5			
	Styrene		-	Sylvaroad	S	7
	Styrene Butadiene	SBR	3			
	Rubber					

Modification plan for softening agents and polymer modified PG52-34



Table 3

Blending temperatures and time of modified binders

Additives	Blending temperature (°F)	Blending time
GTR	350-360	1 hour
SBS	350-360	4 hours
	(340-350 when SA was used)	
SBR	340-350	1 hour
SiO ₂	330-340	2 hours
TiO ₂	330-340	2 hours
	Epoxy A heated at 185	Stirring the epoxy for 10-20 s
Epoxy	Epoxy B heated at 85	Stirring the epoxy and binder for
	Binder heated at 295	10-20 s

Binder Aging Procedures

The Superpave system uses two laboratory procedures for binder aging in order to measure their properties, namely the rolling thin film oven (RTFO) and the pressure aging vessel (PAV). The RTFO procedure was developed to simulate the aging that occurs during the construction while the PAV procedure is specific to simulate the aging process during service (Abbas et al, 2002). To investigate the binder properties at its least stiff condition, the binder should be unaged.



Rolling Thin Film Oven aging procedure or Short-Term aging. Rolling Thin Film Oven aging procedure or Short-Term aging. The RTFOT aging was developed in the 1960s in California and provides a means for conditioning asphalt to mimic the aging that occurs during the mixing and compaction of hot-mix asphalt concrete using a batch plant operating at approximately 150°C. The testing procedure consists of pouring 35 +-0.5 g of asphalt in a glass containers, which has a narrow top opening. Afterwards, the glass bottles are placed for 1 hour to 3 hours maximum in a cooling rack that must be constructed from aluminum or stainless steel .After cooling, the containers are placed in a carriage that must hold them firmly in a horizontal position and the container opening is facing a jet of air. The oven is kept at 163°C and the carriage is rotated in the oven at a rate of 15 +- 0.2 revolutions per minute for 85 min.

Pressurized Aging Vessel (PAV). Oxidation causes the hardening of asphalt binders during a long-term exposure in the field. This testing provides a tool for accelerating the in-service oxidative aging of asphalt by conditioning the binder at high pressure (2.10 MPa) and temperatures (90°C or 100°C or 110°C). Five to ten years of long-term field aging can be simulated in 20 hours using the PAV procedure. The testing provides a residue that can be tested with BBR, DSR and DTT in order to grade the asphalt binder in accordance to AASHTOM320 and AASHTO R29. The aging method consist of pouring 50+-0.5g of RTFOT aged asphalt binder in pans so that the layer reaches 3.2 mm thick. Then the pans are placed in the vessel which was preheated to the conditioning temperature for 20 hours. The pressure should be applied so that the conditioning time starts. After 20 hours of aging, the pressure is released over a period of



9+/-1 minute at a linear rate. Once the pressure is released, the vessel can be opened, and the binder can be tested.

Experimental Plan

In this study, several experiments were carried out in order to evaluate the modified asphalt binders' properties in terms of permanent deformation, cracking resistance and fatigue performance. Table 2 summarizes the laboratory performance testing conducted.

Table 4

Experimental plan

Properties	Testing	Specification	
High temperature	D-marker Classe D1		
PG grade (°C)	Dynamic Snear Kneometer	AASH10 1313	
	Bending Beam Rheometer		
Low Temperature	-24° and -30°C for PG 52-34	AASHTO T315	
PG grade (°C)	-30° and -36°C for PG 64E-40		
Complex modulus	Frequency sweep testing		
(MPa)/Phase angle	10°, 22°, 34° and 46 °C	N/A	
(°)			
Stress (MPa) /strain			
(Macrostrain) at	BBR Strength at -30 °C	De Oliveira et al. (2019)	
failure			



Table 4 (continued)

Properties	Testing	Specification	
Fatigue life	Modified Linear Amplitude Sweep	AASHTO TP101 and	
	(LAS) at 5 °C	Wang et al. (2015)	
	Brookfield Rotational Viscometer,		
Viscosity	110,120,130,140,150°C for PG 52-34	AASHTO T316-13	
	140,150,160,170,180 °C for PG 64E-40		
Percent Recovery			
(%) /J _{nr} at 3.2KPa	Multiple Stress Creep Recovery	AASHTO T350	
(1/kPa)	(MSCR), 64°C		

Standard method of test for viscosity determination of asphalt binder using rotational viscometer (AASHTO T316-13 (2017)). This method is used to determine binder viscosity at pumping and handling temperatures. It is also used to prepare viscosity-temperature charts for determining mixing and compaction temperatures. The binders were tested in accordance with the Superpave binder specifications (AASHTO T316-13 2017) using the Brookfield rotational viscometer, a spindle of #21 and approximately 7.5 to 8 g of binder. Test temperatures for PG52-34 varied from 110°C to



150°C and from 140°C to 180°C for PG64E-40 with 10°C interval to investigate the variability of all binders in this study.

Frequency sweep. A frequency sweep allows to determine the viscoelastic properties of the bitumen sample as a function of timescale. In this testing, the deformation amplitude or amplitude of shear stress is constant while the frequency is varied. Frequency sweep tests were performed on asphalt binders aged in accordance with the rolling thin-film oven test (RTFOT) test. The unaged binders were tested for grade verification only. The test was performed on 8-mm parallel plate with a 2mm gap. The sample was allowed to equilibrate for 10 min at each temperature before testing. The modified asphalt binders were heated in an oven for enough period of time to ensure fluidity. Afterwards, the binders were allowed to cool down until they became solid after pouring them into a silicone mold (8 mm in diameter). Each asphalt sample was tested at four temperatures (10°C, 22°C, 34°C, and 46°C) and sixteen frequencies ranging from 0.016 Hz to 15.92 Hz to evaluate the impact of binder modifiers on the rheological properties of each base binder.

The Dynamic Shear Rheometer (DSR) was used to assess the properties of 1 mm thick asphalt samples using a disposable plate with a diameter of 8 mm. Two asphalt base binders were used for this test: (1) PG 52-34 and (2) PG 64E-40. Each asphalt sample was tested at four temperatures (10°C, 22°C, 34°C, and 46°C) and sixteen frequencies ranging from 0.016 Hz to 15.92 Hz to evaluate the impact of binder modifiers on the rheological properties of each base binder.

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Standard test method for determining the rheological properties of asphalt binder using a Dynamic Shear Rheometer (DSR) (AASHTO T315-12). The DSR test is to measure the complex shear modulus and phase angle for aged and unaged binder at intermediate to high temperatures. It uses a 25 mm diameter parallel geometry plate with 1mm gap and at a frequency of 10 rad/s. The shear stress and shear strain were measured during each cycle and then were used to characterize both viscous and elastic behavior. This method is applicable to linear viscoelastic material and shows the asphalt binder's resistance to rutting and fatigue cracking and is used to grade asphalt binders according to AASHTO M320 and ASTM D6373. Since the asphalt binder is viscoelastic, the phase angle for a completely viscous material is 90 degree and zero degrees for a completely elastic material. This parameter increases with the increase of temperature. The test was conducted at a starting temperature and stops when it reaches a fail temperature, for unaged binder G* /Sinð value less than 1.0 kPa and 2.20 kPa for aged binder.

The DSR samples of modified asphalt binders were prepared using the following procedure. The modified asphalt binders were heated in an oven for enough period of time to ensure fluidity. Afterwards, the binders were allowed to cool down until they became solid after pouring them into a silicone mold (25 mm in diameter).

Bending Beam Rheometer (BBR) (AASHTO T313-12). Traditionally, standardized and/or modified standardized test methods, such as low-temperature penetration, Fraass breaking point and low-temperature ductility and low-temperature were used to evaluate low temperature properties of asphalt binders (Isacsson et al,



1995). However, these methods are not accurate in predicting the thermal cracking susceptibility (King et a, 1992) and especially when the binder is modified (Lu et Ekblad, 1998). The Strategic Highway Research Program (SHRP) developed the Bending Beam Rheometer (BBR) in order to measure the binder's rheological properties or susceptibility to thermal cracking at low temperatures. This testing is widely used with unaged or aged binders using aging procedures given by the ASTM and AASHTO.

The test method provides a means for determining the creep stiffness of asphalt binders versus loading time. The relationship between the low temperature and asphalt binders are shown and the stress relaxation is indicated when constant loading (0.98 N) is applied in the asphalt beam (Yao et al, 2012). In this study, the testing temperatures are -24°C and -30°C for polymer modified asphalt binders and -30 and -36 for softening agents plus polymer modified asphalt binders.

Modified BBR test for low temperature properties. In this study, low temperature properties of unmodified and modified binders were assessed using a BBR-Pro device in accordance with a pervious study (De Oliveira et al, 2019). This device is similar to the standard BBR device, but the only difference is that it has a high load cell capacity of 44N and ability to control loading rate. In addition to that the specimen dimensions are also similar to the standard BBR testing. The modified BBR test consists of applying a loading at a rate of 0.65N/s on a binder specimen until the specimen breaks and the load and deformation are then recorded. The stress and strain at failure (stress and strain at peak load) are calculated using Equations 1 and 2 based on the dimensions of binder specimen, peak load, and deflection at peak load. If the stresses and strains at



failure are higher, the binder is considered more resistant to low temperature cracking. In this study, only one temperature of -30 °C was used to compare the effect of additives on low temperature properties.

$$\sigma_N = \frac{3P_N L}{2bh^2} \tag{3}$$

$$\varepsilon_N = \frac{6\sigma_N h}{L^2} \tag{4}$$

Where:

 σ_N is stress at failure (MPa),

 ε_N is strain at failure,

P_N is maximum measured load (N),

L is the span length (mm),

b is the width of the beam (mm),

h is the thickness of the beam (mm) and

 σ_N is the deflection (mm) of the beam corresponding to the maximum load.

Multiple Stress Creep Recovery (MSCR) (AASHTO T350-14). This testing

evaluates the binder's susceptibility to permanent deformation using the creep and recovery test concept using the Dynamic Shear Rheometer (DSR). Creep load is applied to the binder sample for one-second then the sample is allowed to recover for 9 seconds. In the beginning of the test, a low stress (0.1 kPa) is applied for 10 creep/recovery cycles then 3.2 kPa is applied and repeated for an additional 10 cycles. The MSCR test was conducted according to the AASHTO T 350-14 standard procedure at 64°C using a dynamic shear (DSR) rheometer and 2 samples for each RTFOT aged investigated



dosage. This method provides a new high-temperature binder specification predicting more accurately the permanent deformation performance and the prevailing indicator of field rutting performance is the non-recoverable creep compliance (Jnr) .The current AASHTO M332 specification grades the asphalt binder using two Jnr-based values: Jnr3.2 for a given traffic level and loading rate, and the 75% max value for the Jnr diff for various groups of traffic loads. The specification mentions a maximum value for Jnr diff to avoid the use of binders with high sensitivity to stress. In case Jnr diff values are above 75%, the binder is considered rutting susceptible in unexpected situations of load and/or temperature.

Modified linear amplitude sweep . In the study, the fatigue properties of 20-hour PAV aged binders were assessed using the modified LAS test developed by Wang et al. (2015). In modified LAS test, three amplitude sweep tests with standard strain range of 30% are performed within 300s, 600s and 900s, and the peak in stored pseudostrain energy (PSE) is considered the failure point. The viscoelastic continuum damage (VECD) mechanics approach is used to predict the fatigue lives at different strains.

In this testing, three replicates were prepared according to AASHTO T 315 using the 8-mm parallel plate geometry with a 2-mm gap setting. The testing temperature of 5°C was selected based on a previous study (Safaei et al. 2014) to ensure initial complex shear modulus (G*) of binders within the range of 10 to 50 MPa. This control of initial G* or control testing temperature is conducted to avoid flow of binders at high temperature and an adhesive failure between the DSR plates and asphalt specimen at low temperature.



Chapter 4

Results and Discussion

In this chapter, the results of two modification phases are explained. The first phase consisted of evaluating the impact of polymers and nanomaterials on PG 52-34 and PG 64E-40 asphalt binders. In this part, viscosity plots, master curves, black space diagrams, continuous PG grade, and creep stiffness will be presented. In addition to that, modified BBR results and rutting parameters and fatigue life will be illustrated. Regarding the second phase, the impact of softening agents combined with polymers on the PG52-34 asphalt binder will be assessed. In this part, master curves, black space diagrams, continuous PG grade, creep stiffness, and rutting parameters will be presented.

Polymer and Nanomaterials Modified PG52-34 and PG64E-40 Asphalt Binders

Viscosity.The resistance of asphalt binders to flow or viscosity was investigated. Figures 2 and 3 show the viscosity plots for polymerized and nano-modified PG52-34 and PG64E-40 asphalt binders. The viscosity of neat asphalt binder decreases with the increase of the testing temperature. In addition to that, the viscosity of all modified asphalt binders at any concentration decreases with the decrease of temperatures as well. For instance, for PG52-34 modified with 15% rubber, the viscosity at 110 °C is 5.7 Pa.s and decreased to 0.6 Pa.s at 150°C. It can also be seen that additives can significantly increase the viscosity of asphalt binders, which leads to the increase of the binder film thickness to coat aggregates in the hot mixture. Eventually, the more viscous the binder, the more stable are the asphalt mixtures. For PG52-34 asphalt binder, it can be noticed



that 15% rubber leads to the highest increase in viscosity values compared to neat asphalt binder (850.8% at 110°C) compared to the neat asphalt binder followed by 10% rubber (433.5%), 7.5% SBS (390.7%) and 3% SBS (113.8%). On the other hand, nanomaterials exhibited the least increase in viscosity.

For PG64E-40, at 140°C, 10% rubber showed the highest increase in viscosity (106%) compared to neat asphalt binder followed by 5% rubber (73%) and nanomaterials. Results also showed that when the additives percentages increased, the asphalt binders' viscosities increased at each testing temperature. For instance, for PG52-34 asphalt binders, when the rubber percentages increased from 10% to 15%, the viscosity increases from 0.3 to 0.5 Pa.s at 110°C and from 0.3 to 0.6 Pa.s at 150°C. Concerning the SBS additives, when the percentage increases from 3% to 7.5 %, the viscosity increases from 0.13 to 0.29 Pa.s at 110°C and from 0.14.5 to 0.44 Pa.s at 150°C. The same trend is holding true for the PG64E-40 asphalt binders. It can be depicted that for mixing and compaction of asphalts with high viscosity, high temperatures are required. This can lead to the rise in heating costs of asphalt.





Figure 1. Viscosity of neat, polymer and nanomaterials modified PG52-34 asphalt binder





Figure 2. Viscosity of neat, polymer and nanomaterials modified PG64E-40 asphalt binder

Dynamic complex modulus and phase angle. The viscoelastic properties, i.e., phase angle and dynamic complex modulus, were used to evaluate asphalt sensitivity of stiffness to temperature and frequency.

Figures 3 and 5 illustrate the G^* master curves developed for neat and modified PG 52-34 and PG 64E-40 binders at a reference temperature of 21°C. As can be seen from Figures 4 and 6, as reduced frequency increased and temperature increased, $|G^*|$ of all tested binders decreased. On the other hand, based on $|G^*|$ master curves, the addition of asphalt modifiers seemed to have an impact on the stiffness of both PG 52-34 and PG 64E-40 binders. From Figure 4, all modified PG 52-34 presented higher $|G^*|$ values that those of the base binder (Neat PG 52-34) at high temperatures. This suggests that adding



asphalt modifiers may improve the rutting resistance of PG 52-34. In fact, rubbermodified PG 52-34 (at 15% per asphalt weight) and SBS-modified PG 52-34 (7.5%) presented the highest G* values at high temperatures. While at low temperatures, both base and modified PG 52-34 binders presented relatively similar G* values, with slightly higher stiffness measured for TiO₂ and SiO₂ modified PG 52-34, at 5% and 3% of asphalt weight, respectively. This suggests that modified binders have little to no impact on the cracking resistance of PG 52-34 asphalt binder. By looking at the phase angle master curve of the base and modified PG 52-34 asphalt binders, illustrated in Figure 5, 15% GTR and 7.5 % modified PG 52-34 binders are more elastic than the rest of the asphalt binders at both high and low temperatures.



Figure 3. Complex modulus master curves of neat, polymer and nanomaterials modified PG52-34 asphalt







Figure 4. Phase angle master curves of neat, polymer and nanomaterials PG52-34 asphalt binders

As can be seen in Figure 5, similar to PG 52-34, $|G^*|$ values of base and modified PG 64E-40 increased as temperature decreased, and reduced frequency increased. The addition of polymers to PG 64E-40 had an impact on asphalt binder stiffness at high and low temperatures. In fact, rubber modified PG64E-40 'p(at 10% and 5% of asphalt weight) showed lower stiffness at low temperatures compared to neat asphalt binder. Whereas, nanomaterials exhibited slightly higher stiffness at low temperature results, Rubber modified PG 64E-40 (at 10% and 5% of asphalt weight) presented higher $|G^*|$ values at high temperature than neat binder, which means better resistance to rutting. However, nanomaterials presented lower stiffness at high temperatures than neat binder stiffness at high temperatures than neat binder to rutting.


worse rutting resistance. Phase angle master curves were also developed for the base and modified PG 64E-40 asphalt binder and presented in Figure 6. Rubber at 10% and 5% improved the elasticity of PG 64E-40 asphalt binder at both high and low temperatures, which explains why rubber modified PG 64E-40 would exhibit better rutting resistance. However, nanomaterials had no to little impact on the elasticity at both high and low temperatures.



Figure 5. Complex modulus master curves of neat, polymer and nanomaterials modified PG64E-40 asphalt binders





Figure 6. Phase angle master curves of neat, polymerized and nanomaterials modified PG64E-40 asphalt binders

Black space diagrams. A Black Space diagram is a rheological plot that can define shear modulus $|G^*|$ vs. phase angle (δ). A Black space diagram for both PG 52-34 (with and without softening agents) and PG 64E-40 asphalt binders were developed to determine the change in phase angle with G* at different testing temperatures and reduced frequencies. Figures 7 and 8 illustrate the black space diagrams for PG 52-34, PG 64E-40, and PG 52-34 with softening agents, respectively. As can be seen in Figure 7, given the same phase angle (600), all SiO2 and TiO2 modified PG 52-34 asphalt binders presented higher G* modulus than the rest of the asphalt binders. This means that nanomodified asphalt binders are the most susceptible to cracking than SBS and rubber modified asphalt binders. Regarding PG 64E-40 (Figure 8), all modified PG 64E-40



showed higher stiffness and high phase angle than that of rubber modified PG 64E-40 binders. This indicates that rubber modified asphalt binders are the least susceptible to rutting and cracking compared to neat and other polymer modified asphalt binders.



Figure 7. Black space diagram for neat, polymer and nanomaterials modified PG 52-34





Figure 8. Black space diagram for neat, polymerized and nanomaterials modified PG 64E-40 asphalt binders

Continuous performance grade. Figures 9 presents the continuous performance grades for two base binders and modified variations. Additives used in PG 52-34, 7.5% SBS showed the highest increase in high temperature PG (25°C), followed by 15% GTR (22°C), 10% GTR (16°C), 3% SBS (11°C), and finally Nano-TiO2 and SiO₂ (7°C). The PG 64E-40 binder with 10% and 15% GTR produced the highest increase in high PG by 14°C and 23°C. However, nanomaterials resulted in the least increase of 5°C. These results show that high percentage of SBS and GTR led to the highest increase in high PG compared to nanomaterials ; thus, they could be considered as alternatives to significantly improve high temperature properties. The PG 64E-40, 10% and 5% GTR, increased the



high PG by 23°C and 14°C, respectively, and nanomaterials resulted in an increase of 5°C.

Additives used in PG 52-34, 15% GTR resulted in the highest decrease in true PGLT (3.6°C) followed by 3% SBS (0.4°C). However, 10% GTR, 7.5% SBS, and nanomaterials used in this study increased true PGLT. Regarding PG 64E-40, all the additives except for 10% GTR increased the true PGLT by 0.5 to 3.5°C, and 10% GTR reduced by 2.2°C of true PGLT.

These findings suggest that GTR could be an option to improve the low temperature properties of PG52-34 and PG64E-40 asphalt binders.



Figure 9. Continuous PG grade of polymerized and nanomaterials modified PG52-34 and PG64E-40 asphalt binders





Creep stiffness at low temperature. To reduce cracking at low temperatures, the binder should have low stiffness and high ability of stress relaxation at the lowest pavement temperature (Lu and Ekblad, 2003). The binder is expected to produce mixtures with high thermal stresses in case it is too stiff (Iliuta et al. 2004). In this study, creep stiffness at low temperatures was obtained from the standard BBR test. The creep stiffness of the binders and change due to additives are illustrated in Figures 10 and 11. As shown in Figure 10, 15% GTR showed the highest reduction in creep stiffness (44% at -24°C and 48% at -30°C). Results also indicate that 3%, 7.5% SBS, and 10% GTR decreased creep stiffness at -24 °C and -30°C even though they didn't reduce PGLT. However, Nano-TiO₂ and SiO₂ increased creep stiffness. For instance, at -24°C, 5%TiO₂ produced the highest increase of 22 %, followed by 3% SiO₂(16%). The increase of creep stiffness for 5% SiO₂ and 3%TiO₂ were 11% and 8%, respectively.





Figure 10. Creep stiffness and stiffness changes caused by polymers and nanomaterials for PG 52-34 asphalt binder

Figure 11 shows that for PG 64E-40, all additives didn't decrease the creep stiffness except 10% Rubber which reduced creep stiffness by 42% for -30°C and 29 % for -36°C. because Nanomodified asphalt binders exhibited higher stiffness at low temperatures. For instance, 5%TiO₂ increased the creep stiffness by 18% and 3% SiO₂ and 5% SiO₂ by 12% at -36° C.





Figure 11. Creep stiffness and creep stiffness changes caused by polymers and nanomaterials for PG 64E-40 asphalt binders

Stress and strain at failure at -30° C of polymer and nanomaterials modified asphalt binders. In this study, the stress and strain at failure of neat and polymer modified asphalt binders were determined by means of the modified Bending Beam Rheometer (Modified BBR). Figures 12 through 15 present the stress and strain at failures of polymer modified PG52-34 and PG64E-40 asphalt binders at -30 °C. For PG52-34 asphalt binders, 7.5% SBS resulted in the highest stress value (3.99 MPa) followed by 5% SiO₂ (2.92 MPa) and 5% TiO₂ (2.78 MPa). However, 10% GTR and 15% GTR showed the lowest stress values at -30°C.Regarding the strain at failure, among all binders 7.5% SBS exhibited the highest value (5140) followed by 15% GTR (2994) and 5% GTR (2403 µstrains). It can be noticed that the rubber modifier showed



the lowest stress at failure but not the lowest strain at failure which can be explained by the fact that rubber modified asphalt binders could resist higher deformation but not higher loading.

For PG64E-40 asphalt binders, it can be noticed that 10% GTR and 5% TiO₂ decreased the stress at failure. However, all other modifier dosages increased the stress at failure. It can be indicated that the GTR particles at 10% may deteriorate the existing polymer network in PG 64E-40. Regarding the strain at failure at -30°C, 10% GTR exhibited the lowest value (5588 µstrains) while 3% TiO₂ and 5% GTR showed the highest ones; 7735 and 6978 µstrains respectively.









Figure 13. Strain at failure of polymer and nanomaterials modified PG52-34 asphalt binder at -30°C



Figure 14. Stress at failure of polymer and nanomaterials modified PG64E-40 asphalt binder at -30°C





Figure 15. Strain at failure of polymer and nanomaterials modified PG64E-40 asphalt binder at -30°C.

Analysis of Variance and Post-Hoc were performed to statistically compare the asphalt binders' performances. For PG52-34 asphalt binders, the ANOVA analysis of stress results showed that there is a significant difference between the neat and at least one of the polymers modified asphalt binders(p-value=0.002). On the other hand, the Post-Hoc analysis showed that only 7.5 % SBS resulted in a stress performance significantly different from all other binders. (p-value=0.002).

For PG64E-40 asphalt binder, the ANOVA analysis of stress results indicates that there is no significant impact (p-value=0.132). Regarding the Post-Hoc analysis, results also shows no significance between the neat and modified asphalt binders.



Tables 5 through 8 illustrate the strain statistical analysis of polymer modified PG52-34 and PG64E-40 asphalt binders. From table 6 The ANOVA analysis for stress indicates that there is a significant impact between the neat PG52-34 and at least one of the modified PG52-34 asphalt binders (p-value=0.000). Regarding the Post-Hoc analysis, among all asphalt binders only 7.5% SBS modified binder showed a significant impact with a p-value=0.000.



Statistical analysis of stress at failure for polymer and nanomaterials modified PG52-34 asphalt binder at -30 $^\circ C$

		Modified I	BBR Stress			
Analysis of Variance (ANOVA)						
	p-value		Sig	gnificant?		
	0.002			Yes		
		Post	-Нос			
Neat v	s Modifiers	p-value	Significant?	Recommended		
PG52-	3% SiO2	0.987	No	X		
34 Neat vs	5% SiO2	0.424	No	×		
	3% TiO2	0.994	No	X		
	5% TiO2	0.635	No	X		
	10% GTR	1.000	No	X		
	15% GTR	1.000	No	×		
	3% SBS	0.992	No	X		
	7.5% SBS	0.002	Yes	~		



Statistical analysis of stress at failure for polymer and nanomaterials modified PG64E-40 asphalt binder at -30 $^{\circ}\mathrm{C}$

Modified BBR Stress							
	Analysis of Variance (ANOVA)						
	p-value		Signif	icant?			
0.132			N	0			
Post-Hoc							
Neat vs Moo	lified	p-value	Significant?	Recommended			
	3% SiO2	1.000	No	X			
	5% SiO2	0.972	No	X			
PG 64E-40	3% TiO2	0.996	No	X			
Neat vs	5% TiO2	0.998	No	X			
	5% GTR	1.000	No	X			
	10%GTR	0.429	No	X			



Statistical analysis of strains at failure for polymer and nanomaterials modified PG52-34 asphalt binder at -30 $^{\circ}\mathrm{C}$

Modified BBR Strain							
Analysis of Variance (ANOVA)							
	p-value	Sig	gnificant?				
	0.000		Yes				
Post-Hoc							
Neat vs Modifiers	5	p-value	Significant?	Recommended			
	3% SiO2	0.948	No	X			
	5% SiO2	0.955	No	X			
	3% TiO2	1.000	No	X			
PG52-34 Neat	5% TiO2	0.982	No	X			
VS	10% GTR	0.927	No	X			
	15% GTR	0.372	No	X			
	3% SBS	0.998	No	X			
	7.5% SBS	0.000	Yes	✓			



Statistical analysis of st	rain at failure f	or polymer an	nd nanomaterials	modified PG64E-
40 asphalt binder at -30) °C			

Modified BBR Strain							
Analysis of Variance (ANOVA)							
	p-value	S	Significant?				
0.580			No				
Post-Hoc							
Neat vs Modifiers		p-value	Significant?	Recommended			
PG 64E-40 Neat vs	3% SiO2	1.000	No	X			
	5% SiO2	1.000	No	X			
	3% TiO2	0.720	No	X			
	5% TiO2	1.000	No	X			
	5% GTR	0.964	No	X			
	10% GTR	1.000	No	X			

Rutting performance. The rutting performance of the binders was evaluated using the Multiple Stress Creep Recovery (MSCR) test. The MSCR is conducted using Dynamic Shear Rheometer (DSR) to measure the non–recoverable creep compliance ($J_{nr3.2}$) and percent recovery (R_{3.2}). The findings from the FHWA ALF study indicated that J_{nr} at 3.2



kPa (non-recoverable creep compliance at 3.2 kPa) can identify the rutting performance of the modified as well as the non-modified binders used at the ALF (FHWA-HIF-11-038, 2011). A lower J_{nr} means a higher rutting resistance and less rut depth. The permanent strain measured directly relates to rutting. The calculated Jnr is unrecoverable strain/ applied stress. The R_{3.2} which gives an idea about binder modification and tells us how willingly the asphalt binder will return to its original form after applying a stress.

Non–recoverable creep compliance (Jnr3.2). Figures 16 and 17 illustrate the Jnr at 3.2 kPa. As can be seen from figure 16, all modified binders showed a lower Jnr values at 3.2 kPa than the neat PG52-34 asphalt binder which is expected from the literature review. In addition to that, 7.5 % SBS modified PG52-34 asphalt binder exhibited the lowest Jnr at 3.2 kPa compared to all modified asphalt binders followed by 15% Rubber and 10% Rubber. These modifiers showed a decrease in Jnr at 3.2 kPa by more than 90% compared to the neat binder. On the other hand, for 3% SBS, 3% TiO2 and 5% TiO2 the decrease was by 77.63%, 83.56% and 53.28%. However, 3% SiO2 and 5 % SiO2 showed both a decrease by less than 50%. These results suggest that SBS and Rubber could be a solution to improve the rutting resistance of asphalt binders. In addition to that, as the dosage of SBS and rubber increases, the rutting performance increases. Yet, this is not valid for the nanomaterials.





Figure 16. Non-recoverable creep compliance (J_{nr}) at 64°C and 3.2 KPa for neat, polymer and nanomaterials modified PG52-34 asphalt binders

As shown in Figure 17, for PG64E-40, neat and modified binders exhibited negative values of Jnr at 3.2 KPa which indicates a full asphalt binder recovery after removing the applied stress. The values also can be considered tending to zero. These negative values are explained by the incapability of the rheometer to have the required response time is required to load again for the next cycle. Consequently, the binder recovers before the next loading cycle. Nanomaterials at 3% dosage rate exhibited an average enhancement of 97.47%. For SiO2, TiO2 and rubber (all 5% dosage rate) provided adequate enhanced performance by an average of 49.87%. Finally, 10% rubber showed minimal enhancement by 21.07%.





Figure 17. Non-recoverable creep compliance (J_{nr}) at 64°C and 3.2 kPa for polymerized and nanomaterials modified PG64E-40 asphalt binders

Statistical analyses were conducted to compare the statistical significance of differences in performance observed among the asphalt binders (neat versus polymer modified asphalt binders and neat versus softening agents/polymer modified asphalt binder. Accordingly, analysis of variance (ANOVA) was conducted at 95% confidence level (or p-value <= 0.05 for a significant impact). Moreover, the Tukey Kramer analysis, also called Tukey's Honest Significant Difference, was applied to investigate the significance between the neat asphalt binder and each modified asphalt binder separately. Furthermore, the statistical significance between modified asphalt binder was investigated. Table 9 and 10 present the ANOVA and Post-Hoc results for the Multiple Stress Creep Recovery (MSCR) test conducted for polymer modified PG52-34, polymer modified



PG64E-40, and softening agents/polymer modified PG52-34 asphalt binder respectively. As can be seen from Table 9, ANOVA results showed that all modifiers had a significant impact between the neat and at least one of the modified asphalt binders (p-value = 0.000). Regarding Post-Hoc analysis, results indicated that separately, all modified asphalt binders gave a sigmoid value of 0.000 for all dosage rates of modifiers. From these findings, it can be reminded to use the lowest dosages of additives (3% SBS, 3% SiO2, 10% GTR, and 3% TiO2) since there is no significant different between lower and higher dosage. This further supports the findings made previously regarding the impacts of modifiers on the rutting performance. Although the J_{nr} values at 3.2 kPa for PG64E-40 were different between the neat and the modified asphalt binders, ANOVA and Post-Hoc analysis presented in Table 10 showed that there is no true significance between neat and all modified binder.



Statistical analysis of J_{nr} at 3.2 kPa for polymer modified PG52-34 asphalt binders.

			J _{nr 3.2}		
		Analysis o	f Variance (AN	IOVA)	
p-value S					t?
	0.000			Yes	
			Post-Hoc		
Neat vs M	odifier	p-value	Significant?	Recommende	p-value
				d	between
					modifiers
PG52-34	3% SBS	0.000	Yes	×	0.000
Neat vs	7.5% SBS	0.000	Yes	~	
	3% SiO2	0.000	Yes	~	0.418
	5% SiO2	0.000	Yes	X	
	10%	0.000	Yes	X	0.018
	Rubber				
	15%	0.000	Yes	~	
	Rubber				
	3% TiO2	0.000	Yes	X	0.000
	5% TiO2	0.000	Yes	✓	





Statistical analysis of on al o.2 hi a joi polynici monifica i 6012 10 aspitati citae	Statistical	analysis	of J_{nr} at	t 3.2 kPa_	for polymer	modified P	PG64E-40 asphalt	binders
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J _{nr3.2}						
Analysis of Variance (ANOVA)						
	p-value		Signific	ant?		
	0.142		No			
		Post-Ho	oc			
Neat vs Modi	fiers	p-value	Significant?	Recommended		
PG 64E-40	3% SiO2	0.170	No	X		
Neat vs	5% SiO2	0.622	No	X		
-	3%TiO2	0.177	No	×		
-	5% TiO2	0.790	No	X		
-	5% Rubber	0.806	No	X		
-	10% Rubber	0.993	No	X		

Average percent recovery. Percent recovery gives an indication about the delayed elastic response of the asphalt binders. The lower recovery value obtained indicates that the binder has lower elastic component at the test temperatures. The percent recovery values at 3.2 KPa obtained from the multiple creep recovery (MSCR) testing at



64 C are presented in Figures 18 and 19. For PG52-34 modified with polymers, the neat binder and 3% SiO2, 5% SiO2 and 5%TiO2 modified asphalt binders showed considerably lower values of percent than the other modified binders. In addition to that these values are negative. This can be explained by the tertiary creep behavior of binders and delayed response of the Dynamic Shear Rheometer. It can be seen that 7.5% SBS resulted in the highest percent recovery value (83.35%) followed by 15% GTR (31.3) %, 10% GTR (18.15), 3% TiO2 (22.45) and 3% SBS (15.4) %.From these findings, it can be concluded that 7.5 % SBS and 15 %Rubber resulted in the best rutting performance since they are elastomeric having a dominant elastic network. This characteristic increases the flexibility of the binder and thus permanent deformation resistance. It can also be concluded that among all nanomaterials, TiO2 at a low dosage (3%) showed the best rutting performance.





Figure 18. Average percent recovery strain at 3.2 KPa for PG52-34with and without additives



Figure 19. Average percent recovery at 3.2 kPa for PG64E-40 asphalt binder with and without additives



Analysis of variance (ANOVA) and Post-Hoc analysis were also performed on the average percent recovery values. Tables 13,14 and 15 present the results for PG polymer modified PG52-34, polymer modified PG64E-40 and softening agents/polymer modified PG52-34 asphalt binder respectively. As shown in table 11, ANOVA analysis for PG52-34 and modified binders showed a p-value of 0.000 <0.05 which means a significant impact between the neat and at least one modified binder. More specifically, Post-Hoc presented sigmoid value of 0.000 for SBS modifier (3% and 7.5% dosage rates). Regarding SiO2, both dosage rates did not show a significant impact by having a sigmoid value of 1.000. Moreover, rubber resulted in a significant impact at 10 and 15 % because the sigmoid between 10% and 15% rubber is 0.000. Consequently, both are recommended. It can be seen that 3% TiO2 exhibited a significant impact compared to 5%TiO2.



Percent recovery statistical analysis for neat and polymer modified at PG52-34 asphalt binders at 64 $^{\circ}C$

	% I	Recovery					
	Analysis of V	ariance (AN	(OVA)				
р-у	alue	Significant?					
0.		Yes					
	Ро	ost-Hoc					
			<u>C'</u>	Recommended			
Neat vs wiodifier		p-value	Significant?	?			
	3% SBS	0.000	Yes	X			
-	7.5% SBS	0.000	Yes	~			
-	3% SiO2	1.000	No	X			
- PC52-34 Neat vs	5% SiO2	1.000	No	X			
1 (152-54 fical vs	10% Rubber	0.000	Yes	X			
	15% Rubber	0.000	Yes	~			
	3% TiO2	0.000	Yes	~			
-	5% TiO2	1.000	No	X			



As can be seen from Table 12, ANOVA results indicated a significant impact with a p-value of $0.046 \le 0.05$. On the other hand, Post-Hoc analysis showed that there is no significance between the neat and modified asphalt binders.

Table 12

Percent recovery statistical analysis for neat and polymer modified at PG64E-40 asphalt binders at 64 $^{\circ}\mathrm{C}$

	%	Recovery		
	Analysis of	Variance (A	ANOVA)	
p-v	alue		Signif	icant?
0.0	046		Ye	es
		Post-Hoc		
Neat vs Modifiers		p-value	Significant?	Recommended?
	3% SiO2	0.800	No	X
-	5% SiO2	0.996	No	X
PG 64E-40 Neat	3%TiO2	0.815	No	X
VS	5% TiO2	1.000	No	X
-	5% Rubber	1.000	No	X
-	10% Rubber	0.195	No	X
	10% Kubber	0.195	INO	~



Fatigue life. Figures 20 and 21 present the predicted fatigue lives for the neat and modified asphalt binders. Table 4 illustrates the fatigue life ratio of modified binder to base binder which is given using the following equation.

$$\left(\frac{N_f \text{ of modified binder}}{N_f \text{ of neat binder}}\right) \tag{4}$$

From Figures 20 and 21, it is shown that in the strain range of 1% to 5%, most of additives improved the fatigue life of PG52-34. However, when the strain is higher, a decrease in improvement was noticed. For instance, the fatigue life of 10% rubber modified asphalt binder was 81 times more than the neat asphalt binder when the strain is 1%. Yet, when the strain was 5%, the ratio was 7. Results also showed that 7.5% SBS modified asphalt binder had the highest fatigue life, followed by 10% rubber 10% which exhibited greater fatigue life than 15% rubber. On the other hand, TiO₂ modified asphalt binders resulted in higher increase in fatigue life was very high and this can be explained by the likely the little fatigue damage experienced during the test. To sum up, high polymer and 10% GTR could be a means to improve the fatigue properties of PG 52-34 neat asphalt binder compared to other additives used.

For PG64E-40 asphalt binders, nanomaterials improved the fatigue properties regardless of dosages and 5% rubber reduced the fatigue life at lower strain (1%). However, it enhanced the fatigue life at higher strain (5%). It was noticed that 10% rubber and 7.5 % SBS increased fatigue life of the asphalt binder and resulted in very high N_f. To sum up, the additional use of nanomaterials on polymer modified base binder, could result in



negative impact on the fatigue properties. However, rubber may enhance the fatigue properties.



Figure 20. Predicted fatigue life at testing temperature of 5°C for neat,polymer and nanomaterials modified PG64E-40 asphalt binder





Figure 21. Predicted fatigue life at testing temperature of 5° C for neat, polymer and nanomaterials modified PG 52-34 asphalt binder



Binder Type	Fatigue Life Ratio at Following Strain Amplitude				
	1%	2.5%	5%		
PG52-34+3% TiO ₂	33.9	8.1	2.7		
PG52-34+5% TiO ₂	33.5	6.0	1.7		
PG52-34+3% SiO ₂	5.7	1.9	0.8		
PG52-34+5% SiO ₂	2.2	1.3	0.9		
PG52-34+3% SBS	7.6	3.3	1.8		
PG52-34+7.5% SBS	1.1E+44	2.0E+29	1.5E+18		
PG52-34+10% GTR	81.2	19.5	6.6		
PG52-34+15% GTR	8.2	6.8	5.9		
PG64E-40+3% TiO2	0.04	0.13	0.30		
PG64E-40+5% TiO2	0.08	0.21	0.43		
PG64E-40+3% SiO2	0.02	0.08	0.25		
PG64E-40+5% SiO ₂	0.004	0.03	0.16		
PG64E-40+5% GTR	0.61	0.95	1.33		
PG64E-40+10% GTR	2.2E+22	2.6E+15	1.5E+10		

Fatigue life ratio of polymer modified PG52-34 and PG64E-40 asphalt binders



Summary of Findings for Polymer and Nanomaterials Modified Asphalt Binders

Regarding polymer and nanomaterials modified PG52-34 and PG64E-40 asphalt binders, the summary of findings is mentioned below:

- The viscosity of neat and modified asphalt binders at any concentration decreases with the decrease of temperatures.
- 2) Ground Tire Rubber (GTR) and Styrene-Butadiene-Styrene (SBS) contributed to the highest increase in viscosity among all additives. 15 % GTR, 10 % GTR and 7.5% SBS increased the viscosity by 850.8%, 433.5% and 390% respectively for polymer modified PG52-34 asphalt binder. Regarding PG64E-40, 10% GTR and 5%GTR increased the viscosity the most by (106%) and (73%).
- 3) At 110°C, when rubbers percentages increased from 10% to 15%, the viscosity increased from 3.3 Pa.s to 5.7 Pa.s and from 0.3 Pa.s to 0.6 Pa.s at 150°C.
- 4) Based on the stiffness master curves, regarding PG52-34 asphalt binder, 7.5% SBS and GTR modified binders exhibited higher stiffness at high temperatures compared to nanomodified asphalt binders. Regarding low temperatures, GTR modified PG64E-40 asphalt binder showed slightly lower stiffness in comparison with neat binder. However modified PG 52-34 binders presented similar G* values compared to neat asphalt binder. In addition to that nanomaterials presented a slightly higher stiffness than neat asphalt binder.
- 5) Based on fatigue life from modified LAS, GTR and SBS produced the highest increase in the fatigue life of binders at 95% confidence level compared to nanomaterials.



- 6) For PG52-34 asphalt binder, 7.5% SBS showed the lowest J_{nr} value at 3.2 KPa (less than 0.5 KPa⁻¹) followed by 15% GTR (0.58 KPa⁻¹). Statistical analysis showed that all modifiers had a significant impact. Furthermore, a statistical difference was observed between both additives 'dosages; regarding SBS and GTR and TiO₂ higher dosages were the most significant compared to lower dosages. However, for Nano-SiO₂, no significance was noticed between both dosages.
- 7) The highest percentage recovery at 3.2 kPa was noticed when adding 7.5% SBS compared to all binders (83.35%). In addition to that, ANOVA statistical analysis showed a significance for all modifiers. However Post-Hoc showed a significance for SBS, GTR and 3% TiO₂ and statistical differences were also observed between additives dosages.
- 8) Polymer modified PG64E-40 asphalt binders exhibited negative J_{nr} and high percent recovery values at 3.2KPa. Furthermore, no statistical significance was observed for J_{nr}. However, regarding the percent recovery, only ANOVA analysis showed a significance.
- 9) Based on phase angle master curves, GTR and SBS modified more elastic than the rest of the asphalt binders at both high and low temperatures. However, nanomaterials exhibited similar of slightly different elasticity as neat binder.
- 10) All additives improved the high temperature performance grade. However, the amount of improvement was variable from an additive to another. 7.5% SBS, 10% and 15% GTR showed more than two performance grade bumps. On the other hand,

nanomaterials and 3% SBS increased the high temperature performance grade by more than one PG grade but less than two PG grade temperature.

- 11) With respect to the low temperature performance grade, only 15% GTR reduced the grade among all polymers by 4°C for PG52-34 asphalt binder. Whereas all other polymers produced an increase in PGLT. Regarding PG64E-40, all polymers produced a reduction by less than 6°C except 10% GTR which reduced the PGLT by one grade.
- 12) Results from the BBR strength showed that 7.5% SBS had the highest stress and strain at failure among all the binders. In addition to that results were statistically significant than those for other polymers. 15% GTR exhibited the second highest strain at failure while the lowest stress at failure. 3% SBS, GTR and nanomaterials exhibited statistically equivalent stress and strain at failure to base PG52-34. On the other hand, all polymer modified PG64E-40 asphalt binders were also statistically equivalent to base binder.

Softening Agents and Polymer Modified PG52-34 Asphalt Binder

Dynamic complex modulus and phase angle. The impact of the addition of softening agents on the performance of PG 52-34 asphalt binders was also studied. Two softening agents selected for this study: (1) corn oil and (2) Sylvaroad. Each softening agent was added in constant dosages (7% by total asphalt weight) to PG 52-34 neat or modified with SBS (7.5%) or SBR (3%). It is also worthy to mention that both Sylvaroad and Corn oil were added to PG 52-34 asphalt binder with epoxy (25%) by total asphalt weight. Figures 22 and 23 illustrate both G^{*} and phase angle master curves for PG 52-34



asphalt binders for different testing temperatures and reduced frequencies. Based on Figure 22, all the modified asphalt binders presented lower stiffness at both high and low temperatures except 7C7.5SBS which showed similar performance to neat PG52-34. This suggests that adding softening agents to PG 52-34 asphalt binder may lead to an enhancement in fatigue cracking resistance. In addition to that, at low temperatures, asphalt binders are less sensitive to thermal cracking than neat binder. Figure 23 illustrates the viscoelastic properties of base and modified PG 52-34 asphalt binders containing agents. As can be depicted in Figure 23, all softening agents to increase the viscosity of PG 52-34 asphalt binder at all testing temperatures except 7C7.5SBS. This means that 7 Corn oil combined with 7.5 SBS is less susceptible to rutting than neat and all modified PG52-34 asphalt binder.





Figure 22. Complex modulus master curves for neat and modified PG 52-34 asphalt binders with softening agents and polymers



Figure 23. Phase angle master curves for neat and modified PG 52-34 asphalt binder with softening agents and polymers


For PG 52-34 containing softening agents, Figure 24 shows that given the same phase angle (60°), PG 52-34 containing 7% corn oil and 7.5 SBS showed lower stiffness than other modified PG 52-34 binders. It can be concluded that 7% Corn oil blended with 7.5% SBS are less susceptible to fatigue cracking distress than neat and other softening/polymer modified asphalt binders.



Figure 24. Black space diagram for neat and PG52-34 modified with softening agents and polymers

Continuous performance grade. Figure 25 illustrate the continuous PG grade of softening agents and polymer modified PG52-34 asphalt binder. It can be seen that all additives contributed to the decrease in true PGLT; 7C7.5SBS showed the highest decrease by (17°C) followed by 7C3SBR (16 °C), 7S3SBR (15 °C), 7S7.5SBS (14 °C),



7S25Epoxy (14 °C) and 7C25Epoxy (13 °C). The PG52-34 7S7.5SBS showed the highest increase in high temperature PG (13°C) followed by 7C7.5SBS (8 °C), 7S25E(1°C) and 7C25E (1°C). However, 7C3SBR and 7S3SBR showed a decrease in high temperature PG by 1°C and 5 °C respectively.



Figure 25.Continuous PG grade for neat and modified PG52-34 asphalt binder with softening agents and polymers

Creep stiffness. Figure 26 shows that all additives decreased creep stiffness by more than 42 % at -30°C and -36°C. At -30°C, when adding 7% Sylvaroad and 7.5%SBS to PG52-34 asphalt binder, the most decrease in creep stiffness is shown (75%) followed by 7C3SBR (72%), 7S3SBR (70%). On the other hand, at -36°C, 7C7.5SBS decreased the creep stiffness the most by 70 % followed by 7S7.5SBS and 7S3SBR by 62%.



However, at both temperatures, epoxy combined with softening agents exhibited the least decrease in creep stiffness. Results suggest that softening agents when combined with polymers could be a solution for improving low temperature cracking resistance.



Figure 26.Creep stiffness and stiffness changes caused by additives (Softening agents and Polymers) for PG52-34 Asphalt binder

One-way Analysis of Variance (ANOVA) (α = 0.05) was conducted to evaluate if there is a significance between the neat binder and at least one modified binder on the creep stiffness. Moreover, Post-Hoc statistical analysis was performed on the testing data to have a more specific statistical analysis on each modified binder in comparison with the neat binder. Table 14 and table 15 present the ANOVA and Post-Hoc analysis for creep stiffness at -30°C and -36°C respectively.



Creep Stiffness Statistical Analysis for Softening agents and polymer modified PG52-34 at -30 $^\circ\mathrm{C}$

Analysis of Variance (ANOVA)							
	p-value			Significant?			
	0.000			Yes			
Post-Hoc							
Neat	vs Modifier + Softening	n_valua	Significant?	Racommandad	p-value		
	Agent (SA)	p-vaiue	Significani:	Kecommenueu	between SAs		
	7% Corn Oil +25%	0.000	Yes	~			
Neat vs	Epoxy				0.740		
	7% Sylvaroad +25%	0.000	Yes	~			
	Epoxy						
	7% Corn Oil +3%SBR	0.000	Yes	✓			
	7%	0.000	Yes	~	0.457		
	Sylvaroad+3%SBR						
	7% Corn Oil	0.000	Yes	✓	0.001		
	+7.5%SBS						
	7% Sylvaroad	0.000	Yes	✓			
	+7.5%SBS						



Analysis of Variance (ANOVA)							
p-value			Significant?				
	0.000	Yes					
Post-Hoc							
Neat	vs Modifier + Softening Agent (SA)	p-value	Significant ?	Recommende d	p-value between SAs		
	7% Corn Oil +25% Epoxy	0.000	Yes	~	0.090		
	7% Sylvaroad +25% Epoxy	0.000	Yes	~	0.070		
	7% Corn Oil +3%SBR	0.000	Yes	~	0.612		
	7% Sylvaroad+3%SBR	0.000	Yes	✓	0.012		
Neat vs	7% Corn Oil +7.5%SBS	0.000	Yes	~	0.069		
	7% Sylvaroad +7.5%SBS	0.000	Yes	~	0.007		

Creep Stiffness Statistical Analysis for SA and Polymer Modified PG52-34 -36°C



As shown in Table 18, creep stiffness at -30°C ANOVA results showed that there is a significant impact between the Neat Binder and at least one additive plus softening agent with a p-value as 0.000.

Post-Hoc analysis indicated more specific statistical analysis between the modifiers plus the softening agent in comparison with the neat binder. As shown in table 18, all additives presented a p-value of 0.000, this indicates that all additives are showing improvement than neat binder. The p-value between 7C25E and 7% Sylvaroad is reported to be 0.740. This indicates that since there is no significance between the two combinations. Both softening agents are recommended. Similar recommendation is also given to 7C3SBR and 7 S3SBR since the p-value between them is 0.457. On the other hand, the p-value between 7C7.5SBS and 7S7.5SBS is 0.001 which means that even though both combinations did enhance the low temperature cracking performance, there is a significance in the level of enhancement. Results also showed that creep stiffness of 7% Sylvaroad is lower than there is for 7% Corn oil. (182 MPa for Sylvaroad and 248 MPa for Corn oil).

At -36°C, ANOVA results also showed that there is a significant impact between the Neat Binder and at least one additive plus softening agent (p-value is 0.000). Post -Hoc presented similar p-values to that of -30°C (all 0.000). However, since the p-value between softening agents is not showing a significant impact between corn oil and Sylvarod.

Rutting performance. Non–recoverable creep compliance (Jnr3.2). Figure 27 presents the asphalt binders Jnr values at 3.2 kPa. 7S7.5SBS and 7C7.5SBS showed lower Jnr values at 3.2 kPa compared to the neat PG52-34 (1.72 KPa-1 and 3.15 KPa-1



respectively compared to 15.10 kPa-1). However, 7C25E,7S25E, 7C3SBR and 7S3SBR exhibited higher Jnr values at 3.2 kPa compared to neat PG52-34 asphalt binder. The percentage decrease in Jnr values at 3.2 kPa for 7S7.5SBS and 7C7.5SBS were 79.13% and 88.58 % respectively. It appears that 7S7.5SBS and 7C7.5SBS are the best modification procedures to prevent permanent deformation.



Figure 27.Non-recoverable creep compliance (J_{nr}) at 64°C and 3.2 kPa for softening agents and polymer modified PG52-34 asphalt binders

ANOVA and Post-Hoc of PG52-34 neat versus softening agents/Polymer modified binders presented in table 16 showed that although there is a significant impact between the neat and 7C25E,7C3SBR,7S25E and 7S3SBR, the significance was going into the



direction of reduced performance because their Jnr values at 3.2 KPa were higher that the neat binder.

Table 16

Statistical analysis of Jnr at 3.2 kPa for PG52-34 asphalt binders modified with softening agents and polymers

	Analysis of	Variance (A	NOVA)		
	p-value	Significant?			
	0.000	Yes			
	l	Post-Hoc			
Neat vs M Agent (SA	Iodifier + Softening A)	p-value	Significant?	Recommended	
	7C25E	0.003	Yes*	X	
	7C7.5SBS	0.000	Yes	~	
 Neat	7C3SBR	0.000	Yes*	×	
vs	7825E	0.006	Yes*	×	
	7S7.5SBS	0.000	Yes	~	
	7S3SBR	0.000	Yes*	X	

*Reduced Performance



Average percent recovery. Figure 28 indicates that among all asphalt binders (neat PG52-34 and SA + polymer modified PG52-34), 7S7.5SBS exhibited the highest average percent recovery at 3.2 kPa (46.1%). The second highest value was noticed with 7C7.5SBS modified PG52-34 asphalt binder (31.4%). However, all other modified binders presented negative and slightly higher values compared to neat PG52-34. This suggests that 7% Sylvaroad combined with 7.5% SBS leads to the greatest recovery.



Figure 28.Average percent recovery at 3.2 kPa for PG52-34 asphalt binder modified with softening agents and polymers

ANOVA analysis shown in Table 17 indicates a significant impact (0.000)

between PG52-34 neat asphalt binder and at least one of the softening agents and

polymer modified binders. Post-Hoc analysis showed that both 7C25E and 7S25E did not



show a significant impact by presenting a sigmoid value of 0.993 and 0.974 respectively. 7C3SBR and 7S3SBR also did not show a significant impact than neat asphalt binder by presenting sigmoid values of 0.787 and 0.790 respectively. On the other hand, 7% of softening agent and 7.5% SBS showed 0.000 sigmoid value which means a significant impact.



Table 17

Percent Recovery Statistical Analysis for neat and PG52-34 modified with softening agents and polymers at 64 $^{\circ}C$

% Recovery						
Analysis of Variance (ANOVA)						
	p-value		Significant?	,		
	0.000	Yes				
		Post-Hoc				
Neat vs Mo Agent	difier + Softening	p-value	Significant?	Recommended		
	7C25E	0.993	No	X		
-	7825E	0.974	No	X		
PG 52-34	7C3SBR	0.787	No	X		
Neat vs	7S3SBR	0.790	No	X		
-	7C7.5SBS	0.000	Yes	X		
-	787.5SBS	0.000	Yes	~		



Summary of Findings for Polymer and Softening Agents Modified Soft Asphalt Binders

Regarding the polymer and softening agents modified PG52-34 asphalt binders, the summary of findings is mentioned below:

All combinations of softening agents and polymers improved the low temperature PG grade. However, among all of them only SBR modified asphalt binders showed a decrease in high temperature PG grade by 5°C. Results also showed that the combination of softening agents and SBS improved the most the high temperature grade by one full grade bump.

- From the BBR standard results analysis, all additives lead to the decrease in creep stiffness at -30°C and -36°C. In addition to that, the statistical analysis conducted supports the findings since a significance difference was observed for all modified PG52-34 asphalt binders. At -30°C, 7% Sylvaroad combined with 7.5% SBS exhibited the best performance compared to all binders. On the other hand, at -36°C,7% Corn oil combined with 7.5% SBS and 7% Sylvaroad combined with 7.5% SBS indicated the best performance enhancement.
- 2) The frequency sweep testing results indicated that 7% Corn oil combined with 7.5% SBS presented higher stiffness at high temperatures and lower stiffness at low temperatures. In addition to that, 7% Corn oil combined with 7.5% SBS exhibited the lowest phase angle which means better elasticity.
- The MSCR rutting testing indicated that the combination of both softening agents (Sylvaroad and corn oil) with SBS exhibited a significant impact among all



combination in terms of J_{nr} and percent recovery at 3.2 KPa. However, a significant difference was observed between both combinations in terms of percent recovery; Sylvaroad combined with SBS presented better recovery.



Chapter 5

Conclusions and Recommendations

This study assessed the impact of additives on the performance properties of two types of asphalt binders commonly used in cold regions. The first phase of the study consisted of selecting five additives (Nano TiO₂ and SiO₂, Styrene-butadiene-styrene (SBS), Ground tire rubber (GTR)) to produce modified PG64E-40 and PG52-34 asphalt binders. Performance evaluation of viscosity, rutting, low temperature cracking, and fatigue life of modified binders were carried out by the Brookfield viscometer (RV) Dynamic Shear Remoter (DSR), standard Bending Beam Rheometer (BBR), the Multiple Stress and Creep Recovery (MSCR), BBR strength and Linear Amplitude Sweep (LAS). The second phase consisted in modifying a PG52-34 asphalt binder by means of two softening agents (Sylvaroad and Corn oil) and three additives (Styrene-butadiene-styrene (SBS), Styrene-butadiene-Rubber (SBR) and Epoxy). The evaluation of performance was carried out using the Dynamic Shear Remoter (DSR), standard Bending Beam Rheometer (BBR), and the Multiple Stress and Creep Recovery (MSCR).

The conclusions and recommendation drawn from the results are presented below:

Conclusions for Polymers and Nanomaterial Modified Asphalt Binders

Regarding the first phase of the study, the conclusions and recommendations are presented below:



- Based on the viscosity analysis, it can be concluded that polymers improve the high temperature performance of asphalt binders. In addition to that, higher dosages of modifiers lead to higher viscosity, thus better high temperature performance.
- 2) GTR improved high and low temperature performance grades, fatigue properties, and strain at failure. It also produced stiffer and more elastic asphalt binders at low temperatures. However, it reduced stress at failure in some cases. Therefore, it is not concluded if GTR is able to improve resistance to low temperature cracking of soft binders. Further study is recommended to investigate the ability of GTR for improving low temperature cracking resistance.
- 3) Nano TiO₂ and SiO₂ did not show a considerable performance improvement compared to SBS and rubber in low temperature and fatigue properties. It also produced less stiff and less elastic asphalt binders compared to SBS and GTR, leading to poor rutting and cracking resistance. Therefore, nano TiO₂ and SiO₂ could not be considered as an option to produce highly performing asphalt binders in cold regions.
- 4) The standard BBR results indicated that 7.5% SBS could have a negative effect on low temperature properties. However, BBR strength results indicate that 7.5% SBS significantly improved strain and stress at failure. This suggests that the modified BBR is a better experimental procedure to determine low temperature properties.

Recommendations for Polymers and Nanomaterial Modified Asphalt Binders

The recommendations based on the conclusions above are:



- GTR and SBS could be a good option to modify soft asphalt binders in order to improve the viscosity and therefore obtain better coating asphalt binders and thus better mixtures.
- 2) High dosages of rubber and SBS are recommended for improving the low temperature cracking performance of soft asphalt binders. However, nanomaterial cannot be considered an option for enhancing the cracking resistance.
- 3) The Multiple Stress Creep and Recovery results may be conducted using different conditions (higher temperatures, different loads, and different machines) to better characterize the rutting performance of PG64E-40.
- 4) Modified BBR test could be a better option to evaluate low temperature properties of polymer modified asphalt binders as it is able to capture the benefits in low temperature cracking due to the use of polymer while the standard BBR test could not.
- 5) In this study, modified BBR tests were conducted only at -30°C. More testing temperatures could be used when conducting modified BBR tests to obtain a better understanding of the low temperature properties of asphalt binders.

Conclusions for Polymers and Softening Agents Modified Asphalt Binders

The conclusions and recommendation regarding the polymers and softening agents modified PG52-34 asphalt binders are presented below:

 From both high and low PG grade performance results, the combination of softening agents and SBS lead to the best rutting and cracking resistance.



- Based on creep stiffness results, the addition of softening agents and polymers enhanced the cracking performance of PG52-34 asphalt binder at low temperatures. It can be concluded that 7.5% SBS combined with softening agents lead to the best cracking resistance at low temperatures.
- Based on the rheological analysis results, it can be concluded that among softening agents and polymer modified PG52-34, the combination of 7% Corn oil and 7.5% SBS presented the best viscoelastic properties in terms of cracking resistance and rutting resistance.
- The percent recovery results indicated that 7% Sylvaroad combined with 7.5% SBS significantly improved the recovery of asphalt binder. Thus, it enhances the rutting properties of soft asphalt binders.

Recommendations for Polymers and Softening Agents Modified Asphalt Binders

From the conclusions above, the recommendations listed below were drawn in terms of polymers and softening agents modified PG52-34 asphalt binder:

- SBS modified PG52-34 using softening agents could be an option to improve asphalt binder resistance to rutting and cracking.
- From the creep stiffness analysis, 7.5% SBS combined with softening agents is recommended to enhance the low temperature cracking performance.
- From the rheological analysis, 7.5% SBS combined with corn oil is considered the best candidate asphalt binder modification to improve the resistance to rutting, fatigue, and thermal cracking.



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